RADFORD ARMY AMMUNITION PLANT
RADFORD, VIRGINIA

Performance Based Acquisition
Solid Waste Management Unit 57 (RAAP-022)
Pond by Buildings 4931 & 4928
Interim Measures Work Plan

DRAFT FINAL
NOVEMBER 2010

PREPARED BY:

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Delivery Order Number: DA01

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Sent: Thursday, January 06, 2011 3:33 PM
To: McKenna, Jim J Mr CIV USA AMC
Cc: Richard Mendoza; Cutler,Jim; jeremy.flint@atk.com; jerome.redder@atk.com; Mary Lou Rochotte; Richard Mendoza (External); Meyer, Tom NAB02
Subject: Re: FW: SWMU 57 groundwater data status update (UNCLASSIFIED)

Jim, EPA/VDEQ approve of the SWMU 57 IMWP. Below are several comments from Mike Cramer. While they do not affect this approval, I've included them for future reference. Please call or email me with any questions. Thanks

Ground water samples described in Subsection 5.7 of the IMWP include a purge rate of 500 ug/min for low flow sampling.

EPA region III guidance recommends a low flow purge rate of 400 ug/min when sampling for VOC, and to keep turbidity low for metals sampling.

The field water quality analytical parameters for ground water samples is not listed on the field forms. Generally, field personnel purge a certain volume and just record the parameter results without reference to whether the parameters meet the standard. The standards should be available in the field and the forms should be annotated for compliance/non-compliance(with justification) for each ground water sample.

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November 22, 2010

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Mr. James L. Cutler, Jr.
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Subject: With Certification, Solid Waste Management Unit 57 (RAAP-022) Pond by Buildings 4931 & 4928, Interim Measures Work Plan, Draft Final, November 2010
EPA ID# VA1 210020730

Dear Mr. Geiger and Mr. Cutler:

Enclosed is the certification for the subject document that was sent to you on November 17, 2010. Also enclosed is the 17 November 2010 transmittal email.

Please coordinate with and provide any questions or comments to myself at (540) 639-8658, Jerry Redder ATK staff (540) 639-7536 or Jim McKenna, ACO Staff (540) 731-5782.

Sincerely,

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10-815-165
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Concerning the following:

Radford Army Ammunition Plant
Solid Waste Management Unit 57 (RAAP-022)
Pond by Buildings 4931 & 4928
Interim Measures Work Plan,
Draft Final, November 2010

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowing violations.

SIGNATURE:
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TITLE: LTC, CM
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SIGNATURE:
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PERFORMANCE BASED ACQUISITION
INTERIM MEASURES WORK PLAN
TABLE OF CONTENTS

Section ***Page***

PERFORMANCE BASED ACQUISITION
INTERIM MEASURES WORK PLAN
TABLE OF CONTENTS
(CONTINUED)

LIST OF FIGURES

PERFORMANCE BASED ACQUISITION
INTERIM MEASURES WORK PLAN
TABLE OF CONTENTS
(CONTINUED)

LIST OF TABLES

PERFORMANCE BASED ACQUISITION
INTERIM MEASURES WORK PLAN
TABLE OF CONTENTS
(CONTINUED)

LIST OF APPENDICES

PERFORMANCE BASED ACQUISITION
INTERIM MEASURES WORK PLAN
TABLE OF CONTENTS
(CONTINUED)

LIST OF ABBREVIATIONS AND ACRONYMS

APP	Accident Prevention Plan
BGS	Below Ground Surface
BMP	Best Management Practices
BTAG	Biological Technical Assistance Group
CFR	Code of Federal Regulations
CMOs	Corrective Measures Objectives
CMS	Corrective Measures Study
COCs	Constituents of Concern
COD	Chemical Oxygen Demand
DERP	Defense Environmental Restoration Program
DFW	Definable Features of Work
DoD	Department of Defense
DOT	Department of Transportation
DQO	Data Quality Objectives
ESCP	Erosion and Sedimentation Control Plan
EPP	Environmental Protection Plan
FARs	Federal Acquisitions Regulations
FS	Field Supervisor
FSP	Field Sampling Plan
FT MSL	Feet Above Mean Sea Level
HI	Hazard Index
IM	Interim Measures
IMWP	Interim Measures Work Plan
IDM	Investigative Derived Material
I-RBC	Industrial Risk Based Concentration
IRP	Installation Restoration Program
LTM	Long Term Monitoring
MMA	Main Manufacturing Area
MWP	Master Work Plan
MSDS	Material Safety Data Sheet
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MSP	Master Site Plan
NCR	Notification Control Report
NRU	New River Unit
NTP	Notice to Proceed
OSHA	Occupational Safety and Health Administration
PBA TO	Performance Based Acquisition Firm Fixed Price Task Order
PCBs	Polychlorinated Biphenyls
PETN	Pentaerythritol Tetranitrate
PM	Project Manager
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RCRA	Resource Conservation and Recovery Act

RFAAP	Radford Army Ammunition Plant
RFI	RCRA Facility Investigation
RG	Remedial Goal
SM	Site Manager
SOP	Standard Operating Procedure
SSHO	Site Safety and Health Officer
SSHSP	Site Specific Health and Safety Plan
SSP	Site Screening Process
SVOCs	Semi-Volatile Organic Compounds
SWMU	Solid Waste Management Unit
TAL	Target Analyte List
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TOC	Total Organic Carbon
TSDF	Treatment, Storage, and Disposal Facility
URS	URS Corporation
USACE	United States Army Corp of Engineers
USEPA	United State Environmental Protection Agency
UXB-KEMRON	UXB-KEMRON Remediation Services, LLC
VAC	Virginia Administrative Code
VDEQ	Virginia Department of Environmental Quality
VESCLR&C	Virginia Erosion and Sediment Control Law, Regulations, and Certifications
VOCs	Volatile Organic Compounds
WTDP	Waste Transportation and Disposal Plan
WWPT	Waste Water Treatment Plant

1.0 INTRODUCTION

UXB-KEMRON Remediation Services, LLC (UXB-KEMRON) has been contracted by the U.S. Army Corps of Engineers (USACE) to perform an Interim Measures (IM) at the Pond by Buildings 4931 and 4928, SWMU 57 (RAAP-022), at Radford Army Ammunition Plant (RFAAP), Radford, VA.

The following Interim Measures Work Plan (IMWP) includes details of mobilization, sample collection and analysis, site preparation, erosion control, excavation, disposal, backfill and site restoration at SWMU 57 (the Site). Appendix A contains the proposed Project Schedule, Appendix B contains the Draft Quality Assurance Project Plan (QAPP), and Appendix C contains the DEQ Memorandum of January 8, 2010.

This IMWP is presented as an addendum to the *RFAAP Master Work Plan (MWP)* [URS Corporation (URS), 2003], and incorporates by reference the elements of the MWP, including Section 8, which discusses entry to the Installation and security concerns and requirements. The IMWP also incorporates details specified in the *Solid Waste Management Unit (SWMU) 57 (RAAP-022) Final Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI)/Corrective Measures Study (CMS) Report* (URS, 2009).

This IMWP details site-specific procedures for the IM at SWMU 57. Specifically, this IMWP addresses the excavation and off-site disposal of soil with concentrations of specific metals to below the residential remedial goals (RGs) in order to facilitate clean closeout in accordance with Part II(D)(16) of the RFAAP Corrective Action Permit (USEPA, 2000a). The Interim Measures work is being performed under a Performance Based Acquisition Firm Fixed Price Task Order (PBA TO) for environmental remediation services for three (3) sites at the Radford Army Ammunition Plant (RFAAP), including SWMU 57. All three (3) sites are being addressed under the Installation Restoration Program (IRP) and the RFAAP RCRA Corrective Action Permit. The Department of Defense (DoD) established the Defense Environmental Restoration Program (DERP) to address environmental contamination located on current and former military installations. The contract was issued by USACE – Baltimore located at 10 S. Howard Street, Box 1715, Room 7000 in Baltimore, Maryland. This TO # DA01 was issued under UXB-KEMRON's Worldwide Environmental Remediation Services contract, number W912DY-10-D-0027, with an award date of 30 June 2010 and a Notice to Proceed (NTP) date of 15 July 2010.

1.1 Background

1.1.1 Site Description

RFAAP is a government owned; contractor operated manufacturing facility located in southwestern Virginia approximately 8 miles southwest of Blacksburg (Figure 1). ATK Energetics Systems is the current operator along with a variety of other tenants. RFAAP consists of two noncontiguous areas, the Main Manufacturing Area (MMA) and the New River Unit (NRU). The three sites included in this RFP are all located in the MMA. RFAAP is operating under a 2000 RCRA Corrective Action permit, with a new permit currently being negotiated.

SWMU 57 consists of a 0.06 acre area (2,600 ft^2) inactive, fabricated, asphalt lined pond, an associated terra cotta drainage pipe that leads from Building 4931 to the pond, associated terra cotta piping, and an adjacent drainage swale. SWMU 57, RAAP-022, is located in the MMA, adjacent to the Rocket Area Office at RFAAP (URS, 2009). To the east of the pond are an asphalt-paved road and a system of aboveground and overhead steam lines (Figure 2). A Final RCRA Facility Investigation and Corrective Measures Study (Final RFI/CMS) (URS, 2009) for SWMU 57 was approved by the US Environmental Protection Agency (USEPA) and the Virginia Department of Environmental Quality (VDEQ) in

September 2009. This IMWP is based upon the approved Final RFI/CMS, with the selected Corrective Measures being implemented as Interim Measures as requested by USEPA.

The area adjacent to the pond slopes downward to the northwest toward the New River. Surface elevations range from approximately 1,810 feet above mean sea level (ft msl) to approximately 1,802 ft msl. A soil berm surrounds the pond. A chain link fence surrounds the pond, providing a four foot high barrier. An asphalt liner is present in the pond. The Final RFI/CMS indicates that the asphalt liner of the pond is approximately one inch thick.

The Final RFI/CMS includes an approved Screening Level Ecological Risk Assessment. The July 29, 2005 ecological reconnaissance memorandum, prepared from a URS July 21, 2005 site visit, indicates that upland species dominate the vegetation outside the pond fence, and the drainage feature (e.g., swale) did not convey water and did not support hydrophytic vegetation. The pond is dry at times; SWMU 57 was observed to be absent of standing water during a 17 August 2010 UXB-KEMRON site visit.

The historic discharges from Building 4931 to the pond have resulted in environmental impact within the pond and in the adjacent drainage swale. The Final RFI/CMS concludes that groundwater has not been adversely impacted by the historic site activities. However, the selected remedy for the site requires that groundwater be sampled and analyzed prior to remedy implementation to provide final verification that clean closure of the site is achievable. The Final RFI/CMS states that groundwater corrective measures are not required.

1.1.2 Site History

The URS 2009 Final RFI/CMS states that as-built drawings from 1954 and 1967 illustrate the pond as the "Acid Settling Pool", with a diameter of approximately 50 feet and a capacity of 30,000 gallons. The Final RFI/CMS states that a six-inch diameter terra cotta drainage pipe originated at a four-inch floor drain in Building 4931, located south of SWMU 57. The terra cotta pipe is still present, and leads through the subsurface, emanating at the ground surface and into the pond. The Building 4931 floor drain is represented in the RFI/CMS as having been located near a chromic acid tank and Oakite-33 wash stations. The Final RFI/CMS indicates that chromic acid, hydraulic oil, Oakite-33 and zinc phosphate were reportedly discharged through the floor drain to the pond. According to the Final RFI/CMS, Oakite-33 is a mixture of phosphoric acid and butyl Cellosolve® which replaced chromic acid use after 1974 for purposes of rust stripping, conducted to clean rocket encasements. The Final RFI/CMS indicates that no liquids were visible in the terra cotta pipe at the time of an August 2005 site visit. Likewise, during the May 19, 2010 site walk, UXB-KEMRON did not observe any liquids in the drain pipe. Use of Building 4931 has changed and liquids are no longer managed in the wash station area at Building 4931, nor does discharge from the terra cotta pipe to the pond occur.

1.1.3 Physiography

The Site is located in the western section of the RFAAP Horseshoe Area on a small plateau above a hillside, which slopes downward to the northwest toward the New River (Figure 2-1). Land surface elevations in the vicinity of the SWMU range from approximately 1,810 feet above mean sea level (ft msl) to approximately 1,802 ft msl, as illustrated in Figure 1 (URS, 2009).

1.1.4 Surface Water

The berm that surrounds the pond produces interior drainage within the pond and prevents run-on and runoff of storm water. According to the Final RFI/CMS the pond contained several inches of standing water and heavy vegetation during the field investigation. Additionally, the pond was observed to contain

several inches of standing water and aquatic vegetation during September-October 2003 and a site reconnaissance in July of 2005. However, the pond was observed to be absent of any standing water during a UXB-KEMRON 17 August 2010 site visit.

A shallow drainage depression has been excavated that surrounds the pond and diverts storm water around the pond. The drainage ditch connects with a drainage swale to the northwest (Figure 2) and leads to an intermittent stream located approximately 500 ft northwest of the site. The drainage swale periodically flows northwestward approximately 1,500 ft to the New River, which is the nearest naturally occurring perennial water body. Other manholes, catch basins, storm drains, or drainage ways have not been associated with the site. An approximately eight inch culvert underlies the RFAAP road to the west of SWMU 57, draining the upslope area on the western side of the road. Storm water drains under the road and toward the swale adjacent to SWMU 57.

1.1.5 Site Soil

SWMU 57 is underlain by Braddock loam soil. The Braddock loam soil has been described as being acidic-to strongly acidic, moderately organic, and moderately permeable (URS, 2009). A typical profile of the Braddock soil has been described as consisting of a seven-inch thick surface layer of dark yellowish brown loam, which is underlain by a 60-inch (minimum) thick subsoil of yellowish-red clay and red clay. The depth to bedrock has been determined to be greater than 60 inches (URS, 2003). Soil borings completed at the site indicate that the primary soil type at the site is lean to fat clay (CL or CH) (URS, 2009), which extends to depths of 35 to 40 ft below ground surface (bgs). A cross-section is presented at Figure 2-5 in the Final RFI/CMS (URS, 2009).

1.1.6 Site Geology

The profile of the soil consists of approximately 35 to 40 ft of alluvial terrace deposits, consisting primarily of lean to fat, brown to reddish yellow clay (CH), immediately underlying the site. A transitional zone consisting of saprolite (approximately 4 to 8 ft thick) is below the alluvial deposits and above competent carbonate bedrock. Bedrock at the site consists of Cambrian Elbrook Formation, which is described as dolomite and limestone with lesser shale and siltstone (URS, 2009). The Final RFI/CMS states that the depth to competent bedrock ranges from 57 ft bgs at 57MW2 (1,748 ft msl) to 40 ft bgs at 57MW3 (1,764 ft msl) in the vicinity of the pond.

1.1.7 Site Hydrogeology

Regional hydrogeology and general hydrogeology information for RFAAP is included in Section 3.8 of the MWP. Site-specific hydrogeologic conditions are discussed below.

Groundwater conditions at the site were characterized in the Final RFI/CMS by three monitoring wells (57MW1 through 54MW3), obtaining water level measurements at well locations, and conducting and analyzing slug tests to provide estimates of the hydraulic conductivity of the uppermost aquifer. Slug testing was performed within the saprolite/competent bedrock (57MW2) and bedrock (57MW3) (URS, 2009).

1.1.8 Previous Investigations

In 1992, Dames & Moore collected one sediment/water sample pair (57SE1/57SW1) from material present within the inactive pond for analysis of target analyte list (TAL) metals, volatile organic compounds (VOCs), and semi-volatile organic compounds (SVOCs). The sediment sample was also analyzed for Total Organic Carbon (TOC), total organic halogens, and pH.

Analytical results indicated VOCs and SVOCs were not detected in the sediment sample or surface water sample. Aluminum, arsenic, chromium, iron, and vanadium were reported in sediment at concentrations above their adjusted residential risk-based concentrations.

Additionally, arsenic concentration in sediment was above its adjusted industrial risk-based concentration (I-RBC). The iron concentration in sediment was above its Biological Technical Assistance Group (BTAG) sediment screening level (EPA, 2006c). The water sample reported arsenic, chromium, iron, and manganese concentrations above their adjusted tap water risk-based concentrations, and concentrations of aluminum, arsenic, barium, copper, iron, lead, manganese, and zinc were above their respective BTAG freshwater screening levels (USEPA, 2006b).

In 1992, photogeologic analysis of aerial photographs from 1937 to 1986 was performed. The presence of the pond (SWMU 57) was first noted in 1962, and the drainage channel from the pond to the New River was developed at a later date.

A Site Screening Process (SSP) Investigation was performed in 2007. As part of the SSP, four borings were advanced in and around SWMU 57 to evaluate potential impacts to the soil from previous use as a settling pond. Soil samples were collected and analyzed for target compound list (TCL) VOCs, TCL SVOCs, polycyclic aromatic hydrocarbons (PAHs), explosives (including nitroglycerin and pentaerythritol tetranitrate [PETN]), and Total Analyte List (TAL) metals. One sample was also analyzed for pesticides, polychlorinated biphenyls (PCBs), and herbicides. The human health risk assessment performed indicated that additional investigation for VOCs (Soil Screening Level only) and metals was required. Furthermore, the ecological risk screening portion of the SSP required additional investigation of metals. The SSP resulted in the recommendation of a focused RFI for soil and groundwater media at the site (metals and VOCs).

In order to complete the characterization of soil, and investigation of groundwater at SWMU 57, URS conducted a RFI/CMS in 2008-2009. Five soil borings (57SB4-57SB8) were advanced within the pond areas to refusal. Soil samples collected from borings within the pond area were analyzed for TAL Metals, TCL VOCs, TCL SVOCs, PCBs, pesticides, and explosives. Additionally, a sample from 57SB5 was collected for physical testing of pH, TOC, grain size, Atterberg Limits, moisture content, and cation exchange capacity. A sample from 57SB4 was also collected for physical testing of TOC and cation exchange capacity.

A total of thirteen soil borings were advanced outside of the fenced pond area. Three soil borings were advanced outside the fenced area adjacent to the terracotta pipe. Two samples were collected for chemical analysis (TCL VOCs, TAL metals). Five borings were completed in the lower areas surrounding the pond and drainage swale. Five additional soil borings were advanced further from the perimeter fence (20 and 25 feet away). Three samples were collected from each boring of the ten borings and soil was analyzed for TCL VOCs and TAL metals.

As part of the RFI/CMS, three monitoring wells (57MW1, 57MW2, and 57MW3) were installed at the site. Groundwater samples were collected from newly installed monitoring wells 57MW2 and 57MW3 but could not be collected from 57MW1 due to the lack of sufficient water in the well for sample collection. Along with field parameters, groundwater samples were analyzed for TAL metals (filtered and total), TCL VOCs, TCL SVOCs, pesticides, PCBs, explosives, and perchlorate.

The Final RFI/CMS presents remediation goals (RGs) calculated for constituents of concern (COCs) at the site and are presented in Section 1.2. Groundwater sampling and analysis in site monitoring wells indicates that the maximum detected concentrations reported in groundwater are below RGs for each

groundwater constituent of concern, and therefore development of corrective measures specifically to address COC concentrations in groundwater is not required (URS, 2009).

1.2 Corrective Measures Objectives

CMOs were developed in the 2009 Final RFI/CMS for the COCs in soil at SWMU 57. Section 8.4 of the Final RFI/CMS presents RGs for soil and groundwater COCs at SWMU 57. Maximum detected concentrations of COCs in groundwater were below the established RGs, and therefore corrective measures for groundwater were not developed. CMOs for soil were developed with consideration of the following:

- Current land use of the site is industrial consisting of a 0.06 acre former chromic acid pond, a terracotta pipe, and a drainage swale located adjacent to the pond for a total site area of 0.16 acre;
- Land use of the site is unlikely to change in the future (e.g., industrial with no development) due to the small size of the site and that the site is surrounded by unassociated aboveground piping precluding development and access for development;
- Groundwater is not used as a water supply in the site area;
- The pond area is currently inhabited by amphibians.

The following CMO was developed for soil at SWMU 57 based on the results of the site, risk, and fate and transport assessments and the most likely future land use at the site (industrial):

- Mitigate the potential risks/hazards that have been identified for evaluated future hypothetical industrial receptors for exposure to soil (construction workers) at the site.

The residential exposure pathway also was evaluated in the CMS to assess the remedial effort that would be required to achieve clean closure at SWMU 57 with unrestricted future land use without controls or long-term monitoring (LTM) requirements.

Remedial Goals (RGs) for COCs in soil were calculated for future industrial and residential scenarios in the Final RFI/CMS.

A summary of the RGs for soil:

Soil:

- Aluminum
  - Industrial = 40, 041 mg/kg
  - Residential = 40, 041 mg/kg
- Antimony
  - Industrial = 49.1 mg/kg
  - Residential = 13.2 mg/kg
- Manganese
  - Industrial = 2, 543 mg/kg
  - Residential = 2,543 mg/kg
- Cadmium
  - Industrial = Not Applicable (NA)
  - Residential = 23.2 mg/kg
- Chromium
  - Industrial = NA
  - Residential = 65.3 mg/kg
- Iron
  - Industrial = NA
  - Residential = 50,962 mg/kg

As stated above, RGs for groundwater were calculated for antimony, arsenic, chloroform, and manganese in Section 8.4 of the Final RFI/CMS, however, the maximum concentrations in groundwater did not exceed the RGs. Therefore, groundwater analytical results from groundwater monitoring as part of the IM will be evaluated as discussed in Section 10.4.2 of the Final RFI/CMS.

1.3 Interim Measures Scope

Based on the *SWMU 57 Final RFI/CMS Report,* September, 2009, Alternative Four: Excavation of Soil and Offsite Disposal for Clean Closure and Unrestricted Land Use was selected as the final alternative for SWMU 57.

The objective of the IM action is to reduce the concentrations of aluminum, antimony, cadmium, chromium, iron and manganese such that they do not exceed the residential RGs (as presented in Section 1.2) and facilitate clean closeout in accordance with Part II (D) (11-21) IM of the *RFAAP Corrective Action Permit* (USEPA, 2000a).

Based on the *SWMU 57 Final RFI/CMS Report,* September, 2009, IMs are to be performed at SWMU 57. The IMs are being conducted to mitigate the threat of a contaminant release, migration, and/or exposure to the public and the environment, as well as facilitate clean closeout in accordance with Part II(D)(11-21) IMs of the *RFAAP Corrective Action Permit* (USEPA, 2000a).

The IMs include:

1. **Confirmation Groundwater Sampling for Closure:** Confirmation groundwater sampling will be conducted prior to remedy implementation to verify findings in the Final RFI/CMS that COC concentrations in groundwater are below the applicable MCLs and risk-based criteria and to confirm that clean closure is achievable. Groundwater will be analyzed for COCs as presented in Section 10.4.2, page 10-8, of the Final RFI/CMS.
2. **Surface Water and Soil Waste Characterization:** Surface water from the pond will be sampled to determine disposal options for the water. Four soil samples also will be collected from 2 locations prior to excavation for waste characterization. The soil sampled will be collected from within the footprint of the pond and the immediately surrounding area. Hand augering will be performed to facilitate soil sampling around the supports of the steam lines to determine concentrations of soil COCs immediately surrounding the overhead steam line supports. These sample data will be used to determine appropriate engineering design for the excavation in the vicinity of the overhead steam line supports.
3. **Site Preparation.** Prior to commencement of work, a utility survey will be performed in accordance with established RFAAP procedures and appropriate permits will be obtained from RFAAP. In addition, erosion/sediment control measures will be implemented prior to excavation at the site.
4. **Pond Water Removal and Soil Excavation.** Any standing water in the pond will be removed with a vacuum truck and, based upon the previously collected waste characterization samples, will be disposed of off-site at an appropriately licensed disposal facility. If determined to be acceptable, wastewater maybe disposed of at the RFAAP Wastewater Treatment Plant (WWTP). Soil excavation will be performed within the pond (maximum depth of 15 feet), the drainage swale and exposed portion of the terra cotta pipe (see Figure 3), such that remaining soil is below the residential RG for aluminum, antimony, cadmium, chromium, iron and manganese. The approximately 80 foot long section of the drainage swale closest to the pond will be excavated to progressively shallower depths at increased distance from the pond (Figure 3). Portions of the terra cotta pipe that are not excavated will be sealed with a grout plug; excavation of the terra cotta piping will be limited by the soil berm, and steam line and associated supports.

5. **Confirmation Sampling.** Confirmation sampling will be performed from the bottom and sidewalls of the excavation to confirm the vertical and lateral extent of the soil containing COCs exceeding residential RGs have been removed.
6. **Land Disposal.** Pre-excavation soil characterization will allow for direct loading and disposal to occur during implementation of the IMWP. While no hazardous waste is anticipated at the site, should hazardous waste be encountered, it will be appropriately segregated, containerized and either treated to render it nonhazardous or disposed in a RCRA Subtitle C Landfill. The excavated soil that is classified as non-hazardous waste will be disposed in a RCRA Subtitle D Landfill.
7. **Site Restoration.** The area requiring excavation will be backfilled with pre-sampled clean soil, once the excavation is complete. The area will be backfilled to a grade slightly lower than the base of the storm water culvert that conveys storm water under the adjacent road toward the drainage swale (Figure 5). From the elevation of the storm water culvert, backfill soil will be graded consistent with the surrounding terrain and sloping towards the drainage swale. Limited rip rap/rock may be placed at the mouth of the culvert discharge to prevent erosion during high flow storm water events. During implementation of the IM, the culvert depicted in Figure 5 will be blocked to prevent stormwater discharge into the excavation. Section 7.3 provides details regarding the stormwater diversion. Following the removal of contaminated soil to the established residential RGs and placement of clean backfill soil, the site will be restored, seeded, and all equipment will be demobilized.

Specific details on the contractor organization and technical approach for the IM listed above are provided in the Technical Approach Plan, *Section 3.0*.

1.4 Work Plan Content

This IMWP is composed of an Introduction (*Section 1.0*), eight sub-plans (*Sections 2.0 through 9.0*), and references (*Section 10.0*). The eight sub-plans are as follows:

Section 2 – Organization
: Identifies the UXB-KEMRON project staff and subcontractors, their roles and responsibilities.

Section 3 – Technical Approach
: Provides the details regarding the technical approach to the followed for the IM.

Section 4 – Project Schedule
: Presents the milestone events for the IM.

Section 5 –Field Sampling Plan
: Describes the sampling rationale and field sampling procedures that will be used to collect field samples

Section 6 – Environmental Protection Plan
: Details the procedures that will be taken to minimize and/or eliminate introduction of chemicals to the environment during IM work.

Section 7 – Erosion and Sediment Control Plan
: Defines the steps that will be taken to minimize and/or eliminate erosion and sedimentation during IM work.

Section 8 – Waste Transportation and Disposal Plan
: Identifies safe handling, transportation, and disposal procedures for waste material resulting from IM.

1.5 Work Plan Changes

Work outside the scope of this work plan will not to be performed without the approval of the USACE, Baltimore District. Amendments or supplements to this work plan will be submitted in writing to the USACE for approval prior to being implemented by project personnel.

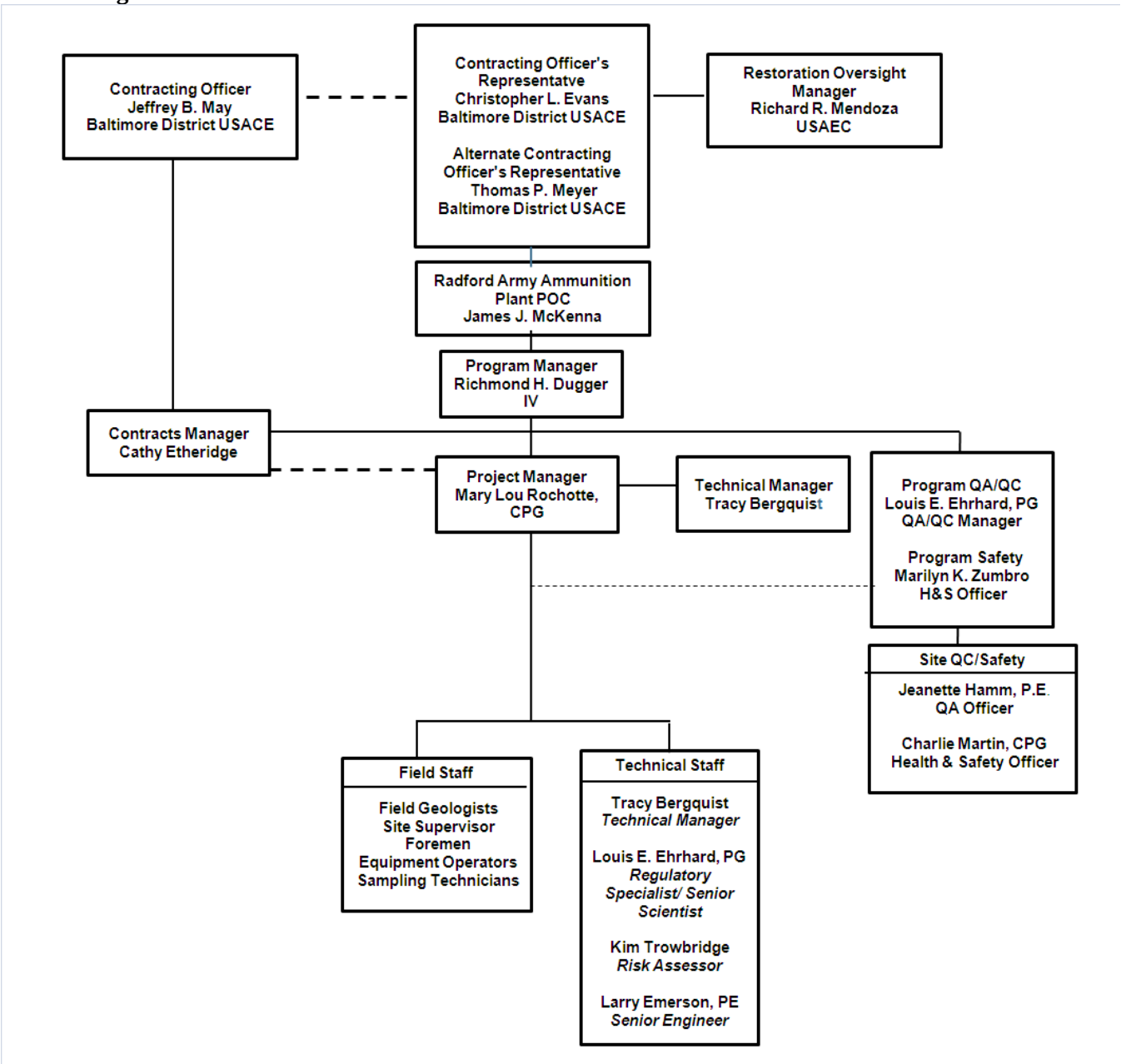
2.0 ORGANIZATION

This section describes the organization and activities to be conducted to accomplish the IM at SWMU 57. Specifically, this section outlines the organization and responsibilities for project personnel as well as presents the step by step approach to be performed for each of the IM tasks.

The duties and responsibilities of the key members of this organization are described below, and in Appendix B.

2.1 Organization and Responsibilities

RFAAP Organizational Chart



The management of this contract involves multiple authorities, including the United States Army Environmental Command (USAEC), Baltimore District of the USACE, and RFAAP Army and ATK personnel. The Baltimore District, USACE is responsible for contract execution and oversight and provides technical reviews of contractor deliverables. RFAAP personnel, including both the Army and ATK, are the primary points of contact regarding coordination with regulatory personnel; USAEC and the Baltimore District support regulatory coordination. UXB-KEMRON is responsible for all technical issues, including development and negotiation of remediation and long term monitoring project standards and requirements.

The Baltimore District has made the following assignments for this contract: Jeffrey B. May, Contracting Officer; Christopher L. Evans, Contracting Officer's Representative; Thomas P. Meyer, Alternate Contracting Officer's Representative. UXB-KEMRON has been instructed that Mr. Meyer is our primary point of contact for day to day contracting matters.

Richard Mendoza has been assigned as the USAEC Environmental Restoration Manager (ERM). James McKenna, RFAAP Installation Restoration Program Manager, is the key point of contact (POC) for UXB-KEMRON site activities. Jerome Redder is the lead POC for the site operations contractor, Alliant Techsystems (ATK). Matthew Alberts is the ATK POC regarding field access and site work activities.

Mary Lou Rochotte will serve as the Project Manager. Ms. Rochotte, a Certified Professional Geologist, has been successfully managing Performance Based Contracts (PBCs) since 2002 and has managed a $7.6 million project located in Region III. She will be supported by a team of qualified environmental professionals including Louis Ehrhard as the Senior Scientist and Risk Assessor and Kim Trowbridge as the Risk Assessor. Mr. Ehrhard and Ms. Trowbridge also bring relevant and recent experience on PBCs in USEPA Region III. Additional scientists, chemists, engineers and technicians will support the project.

As the Project Manager, Ms. Rochotte is responsible for all day to day activities and coordination of such activities with Mr. Mendoza, Mr. Meyer and Mr. McKenna, as well as ATK representatives including Mr. Redder and Mr. Alberts. She will have work stoppage authority and/or will authorize field supervisors to have work stoppage authority in her absence from the field. She will coordinate closely with the project Quality Assurance Officer, and will oversee project quality control reviews of project deliverables.

The Project Manager and Quality Assurance Officer will maintain contact with the project chemists/project managers at all subcontracted analytical laboratories, and will also coordinate review and validation of all decision making data generated for the project.

UXB-KEMRON will select subcontractors as the project proceeds for field activities such as drilling, waste transport and disposal, surveying, and other specialty subcontracting project needs. The subcontractors will be selected through a competitive bidding process after safety prequalification and based upon task specific requirements such as required licensure or certification.

3.0 TECHNICAL APPROACH

The following sections describe the background and technical approach to the SWMU 57 IM. Implementation of the IM will occur following receipt of written regulatory approval of the IMWP. The field activities to be performed include: confirmation groundwater sampling, waste characterization, hand augering around steam line supports, site preparation, excavation of the pond area and drainage swale such that the remaining soil is below the residential RG; waste characterization and off-site disposal of soil, waste characterization and on-site or off-site treatment/disposal of surface water from pond, well abandonment, excavation and grouting of the terra cotta pipe, and site restoration. Detailed safety and health requirements for this scope of work are presented in the UXB-KEMRON Accident Prevention Plan (APP) prepared for this project. All UXB-KEMRON employees, subcontractors and visitors to the site during Interim Measures implementation will be required to review and abide by the APP.

A preliminary schedule for conduct of the Interim Measures is included in Appendix A. The schedule will be refined in any revisions to this document, and following regulatory approval. Upon establishment of a firm mobilization date, the UXB-KEMRON will coordinate with RFAAP to provide advance notification of mobilization to USEPA and VDEQ.

Interim measures implementation activities will be documented in multiple ways and in conformance to quality assurance standards established in site plans. At a minimum, notes of activities will be documented in field books, sampling logs/forms will be completed, and activities and site progress will be photodocumented.

3.1 Definable Features of Work

The field work for the RFAAP SWMU 57 pond by buildings 4931 & 4928 involves the following definable features of work (DFWs):

1. Pre-Excavation and Confirmation Sampling;
2. Site Preparation/Stabilization/Fence Removal;
3. Excavation;
4. Monitoring Well Abandonment;
5. Waste Characterization and Disposal;
6. Backfill and Site Restoration; and
7. Preparation of Interim Measures Completion Report.

3.2 Three Phases of Control Procedures

The project manager (PM) and field supervisor (FS) will ensure that a three-phase control process is implemented for each definable feature of work. Each control phase is important for obtaining a quality product. However, the preparatory and initial inspections will be particularly valuable in preventing problems. Production work will not be performed on a DFW until a successful preparatory and initial phase inspection has been completed.

3.2.1 Preparatory Phase Inspection

A Preparatory Phase Inspection will be performed prior to beginning each DFW. The purpose of this inspection will be to review applicable specifications and verify the necessary resources, conditions, and controls are in place before the start of work activities.

The PM will verify with RFAAP representatives that all pre-construction submittals have been received and approved, and that lessons learned during previous projects have been incorporated, as appropriate, into the project procedures to prevent recurrence. The FS will meet with the PM, and the staff responsible

for a DFW. Work plans and operating procedures will be reviewed by the PM to ensure they describe pre-qualifying requirements or conditions, equipment and materials, appropriate sequence, methodology, and QC requirements.

The PM and/or FS will verify the following:

- All plans have been prepared, reviewed, and approved, and are available to field personnel;
- All associated materials have been submitted and approved, have been properly stored, and are available on site;
- Appropriate field equipment is available, functional, and properly calibrated;
- Responsibilities have been assigned and communicated;
- The job hazards in the APP and SSHSP have been communicated and the necessary safety measures are in place and ready for use;
- Field personnel have the necessary knowledge, qualifications/expertise, and information to perform their duties; and
- Arrangements for support services have been made and the prerequisite site work has been completed.

Discrepancies between existing conditions and approved plans and/or procedures will be resolved by the FS prior to the PM granting approval for work to begin.

3.2.2 Initial Phase Inspection

An Initial Phase Inspection will be performed by the FS or his/her designee the first time a DFW is performed. The purpose of the inspection is to:

- Check the preliminary work for conformance to procedures and contract specifications, as identified in the Preparatory Phase;
- Verify inspection and testing, as applicable;
- Discuss the acceptable level of workmanship with assigned personnel;
- Ensure safety compliance; and
- Check for omissions, deficiencies; and resolve differences of interpretation.

The PM ensures that discrepancies between site practices and the drawings and specifications are identified and resolved before granting approval to proceed.

3.2.3 Follow-up Phase Inspections

Follow-up Phase Inspections will be performed routinely during each DFW. The purpose of these inspections is to ensure continued compliance and quality workmanship and materials. The FS will monitor the practices and operations and verify continued compliance with the approved interim measures work plans. A Stop Work Order will be issued by the PM if a work stoppage is required to correct a deficient procedure or practice.

The FS and/or PM will also verify that a daily safety and health inspection is performed and documented as prescribed in the SSHSP. The PM will oversee and observe the same activities as under the initial inspection. Discrepancies between site practices and the approved plans and/or procedures will be resolved by the PM prior to granting approval to continue work.

At the discretion of the COR, RFAAP representative or PM, additional inspections may be required on the same DFW. Such instances may be:

- Unsatisfactory work, as determined by UXB-KEMRON or RFAAP representatives;

- Change in key personnel, or resumption of work after a substantial (2 weeks or more) period of inactivity ; and
- Changes to the project SOW and/or specifications.

3.2.4 Completion Inspections

Completion Inspections will be performed at the conclusion of a work feature or group of features to verify that project requirements are satisfied. A list of any deficient items will be prepared with due dates and space to document corrections made by the field team; documentation of completed responses will be submitted to the Army, if such responses are necessary.

3.3 Pre-Excavation and Post-Excavation Confirmation Sampling

Pre-excavation and post-excavation sampling will be conducted during the IM. Pre-excavation activities will include confirmation groundwater sampling, surface water sampling and soil sampling for waste characterization, and soil sampling around the supports of the above ground steam lines. Post-excavation confirmation soil sampling will be performed to determine the limits of the excavation.

Confirmation groundwater sampling will be conducted prior to remedy implementation to verify RFI findings that COC concentrations in groundwater are below applicable MCLS as listed in the Final RFI/CMS, and confirm that clean closure is achievable at the site.

As specified in the Final RFI/CMS, the selected action for SWMU 57 includes sampling of two monitoring wells at the site, 57MW2 and 57MW3, for analysis of the groundwater COCs, including antimony, arsenic, chromium, manganese, and TCL VOCs. Metals sampling and analysis will include dissolved metals sampling and analysis for these four groundwater COCs, as stated on page 10-8 of the Final RFI/CMS. Monitoring wells are show on Figure 3.

Sampling will be conducted in conformance with approved standard operating procedure SOP 30.2 and as described in the Section 5.2.10 of the MWP (URS, 2003). Groundwater sampling will be conducted using low flow purge and sampling, consistent with past sampling events. Section 5.7 presents the sampling plan for groundwater sampling as part of the IMWP.

Hand auger borings will be advanced around the supports for the steam pipes that are within the proposed excavation area (Figure 4). A minimum of 4, and up to a maximum of 9 hand augers will be advanced to an estimated depth of 5 feet. The presence of footers around the supports will be determined, and soil samples will be collected at the estimated center point depth (2.5 ft bgs) of the excavation under the steam pipes and bottom of the excavation at that location (5 feet bgs). Hand auger borings will be performed in accordance with the procedures for drilling presented in Section 5.2 of the MWP. The results of this will allow UXB-KEMRON to determine if the excavation must be performed directly under the supports, or if the soil under the supports meets RGs, and therefore a 1:1 grade slope can be performed around the supports during excavation of the soil exceeding RGs in the drainage swale. If analytical results indicate that COCs exceed RGs immediately adjacent to the supports, then temporary supports will be placed outside of the excavation area and the supports will be removed during excavation if and as approved by RFAAP. Section 5.6 presents the details regarding sampling plan for hand auger borings and soil sampling at the site.

UXB-KEMRON will ensure all analytical data will meet the data quality objectives specified in the QAPP for Radford (Appendix B). Following Army authorization for data release, the groundwater data will be shared with USEPA and VDEQ. Data will be presented in both map and tabular form with comparison to analytical results presented in the Final RFI/CMS, as well as the groundwater RGs. If none of the analytes in samples from MW572 and MW573 exceeds a groundwater RG, the data will

verify that clean closure is achievable at the site. If groundwater RGs are exceeded, the Army will discuss appropriateness of proceeding with the selected alternative with USEPA and VDEQ. It is anticipated that the groundwater analytical results will be discussed with regulatory stakeholders via teleconference or online meeting to expedite agreement regarding the appropriateness of proceeding with the excavation of soil in accordance with the approved CMS. Regulatory discussion and concurrence will be documented in teleconference/meeting minutes and, upon Army approval, issued for regulatory concurrence to document this critical step in the project record.

Concurrent with or subsequent to the groundwater sampling, UXB-KEMRON will conduct sampling of the surface water in the pond, if present, and soil as necessary. These media will be characterized for purposes of IDM management and disposal n. Surface water sampling will be conducted in accordance with Section 5.3.1 and SOP 30.3 of the MWP. UXB-KEMRON will identify a permitted off-site disposal facility for soil and water disposal, and also will coordinate with RFAAP WWTP personnel regarding the necessary pond water waste characterization, in the event that the pond water can be disposed of at the WWTP. . See Section 5 for details regarding the sampling plan. Prior to excavation, the surface water will be removed using a vacuum truck, or pumps, depending upon the volume of water within the pond at the time of implementation of the IM. The Final RFI/CMS estimates 6,900 gallons of water within the pond requiring removal.

Composite waste characterization samples will be collected from surface and subsurface locations within the area determined during the RFI field activities to contain elevated levels of COCs above residential RGs (Figure 4). The receiving facility may require that one composite sample be collected from the waste materials per 1,000 cubic yards of material; it is assumed that one composite soil sample (composited from three locations) will be collected and analyzed for Toxicity Characteristic Leaching Procedure (TCLP) metals, PCBs, corrosivity as pH, reactivity (including explosives), and ignitability in order to allow direct load-out of soil during excavation activities. Soil sample procedures are presented in Section5.6 of the IMWP. This pre-excavation waste characterization will ensure that soils will be directly loaded and transported to the appropriate disposal facility during implementation of the IM.

During the excavation soil confirmation samples will be collected from both the floor and sidewalls of the excavation along the drainage swale and within the pond. Figure 4 shows the preliminary locations of the planned confirmation samples. Detail of the confirmation sampling is presented in Section 5.6.

3.4 Site Preparation/Stabilization/Fence Removal

Site preparation actions include performing a pre-existing site condition inspection, establishing temporary facilities (if necessary), installing decontamination areas, fence removal, and establishing work zones.

UXB-KEMRON will coordinate with RFAAP personnel and utility locators to identify all utilities in the vicinity of RAAP-022. UXB-KEMRON also will closely coordinate with RFAAP regarding the excavation horizontal and vertical extent, proper equipment placement and other site-specific factors to ensure the overhead piping and any other adjacent site features are not impacted by the interim measures.

A main staging area and access will be established north of SWMU 57 (Figure 5). The main staging area will be designed to have a designated area for employee parking, a non-hazardous solid waste disposal unit (e.g., dumpster), a decontamination area, a fueling area, and a portable toilet.

Site mobilization operations will be completed prior to commencing the prescribed project operations. Site mobilization includes mobilization of required personnel and equipment to the SWMU. Mobilization of most resources will occur at the onset of the project.

KEMRON will mobilize project personnel from areas outside and/or inside the local region. All personnel working at the Site will possess the appropriate skills and knowledge necessary to function in their specified capacity. They will all have completed the necessary health and safety training and physical evaluations to ensure they are able and fit to complete their assigned duties in a safe and efficient manner. The SSHSP included in the project APP presents all health and safety related requirements for project personnel. UXB-KEMRON will provide a complete list of subcontractors to the Army following selection and prior to mobilization. Equipment will be mobilized as needed and released when no longer required.

Equipment required for the work is anticipated to include, but not necessarily limited to:

- Crew vehicles;
- 320 Caterpillar Excavator;
- Front end loader;
- Drill Rig;
- Compactor or Dozer D-5;
- Various support and decontamination equipment.

All equipment will be maintained in good working condition and possess all required safety and operational controls. Inspections upon delivery are required in the SSHSP. Daily safety and operational checklists will be completed for all equipment.

Sediment and erosion controls will be installed and are presented in Section 7.0 of the IMWP. UXB-KEMRON will remove the fencing that surrounds the pond and transport it for recycling or disposal as determined appropriate. UXB-KEMRON will pump the pre-characterized surface water from the pond for appropriate disposal. The waste characterization discussed in Section 5.4 will provide preapproval for the surface water to be transported to the RFAAP WWTP, or, if necessary based on the WWTP permit standards and analytical results from the pond water, the surface water will be containerized and transported for off-site disposal. Any residual material in the pond will be stabilized if/as necessary and will be managed and disposed in conjunction with the soil IDM from the site.

3.5 Excavation

Figure 3, based upon Figure 10-2 of the Final RFI/CMS, illustrates the approximate excavation area with associated depths. Appropriate permits will be applied for and received from RFAAP prior to conduct of any subsurface intrusive activities.

The Final RFI/CMS estimates the soil within the pond area, drainage swale, adjacent to the terra cotta piping, pond asphalt containment, and other associated solids as comprising an in-place volume of approximately 1,685 cubic yards, or 2, 358 tons. Based on information presented in the Final RFI/CMS, UXB-KEMRON anticipates all IDM generated will be nonhazardous.

Excavation depths will be less than or equal to 15 feet bgs. The excavation will begin along the northeast side of the SWMU and excavate south toward the SWMU gradually getting deeper, such that a total depth of 15 feet will be achieved, once the pond is reached. UXB-KEMRON anticipates trench boxes may be required for wall stability within the excavation. A Dozer D-5 or equivalent will be used at the top of the pond and will assist the excavator in reaching materials around the upper reaches of the pond. Supports for the above ground steam lines will be removed, if necessary based upon sampling and analysis, and as determined feasible based upon engineering design. However, engineering limitations determined in conjunction with RFAAP may limit the excavation area within close proximity to the supports or prohibit the removal of steam line supports. If supports must be removed and removal is approved by RFAAP, removed supports will be replaced with temporary supports and cross beams to facilitate soil excavation to achieve RGs.

The exposed portion of the terra cotta pipe up to the soil embankment that underlies the overhead piping on the north side of Building 4931 will be excavated to the extent feasible without disturbing or compromising the soil embankment and related concrete supports to the overlying steam lines. The covered portion of terra cotta pipe will be sealed with a grout plug.

Soil within the remediation area will be removed in approximately 1-ft lifts until RGs are achieved or until the Army's pre-defined limits of the site are reached (15 feet). UXB-KEMRON will coordinate with the Army to ensure all regulatory stakeholders are notified of any changes and modifications needed to complete the IM action. Any such changes or modifications will be documented in writing as well.

Excavation will be performed using a 320 caterpillar excavator or equivalent within the pond area, and a Dozer D-5 or equivalent will be used to assist the excavator, if deemed necessary to expedite the excavator reach near surface soils at the perimeter of the excavation. Contaminated soils will be excavated and directly loaded into dump trucks and transported off site. The sides of all excavations in which employees are exposed to danger from moving ground shall be guarded by a support system, sloping or benching of the ground, or other equivalent means. Sloping and benching, if required, will be in accordance with EM 385-1-1. The portion of the drainage swale where excavations are expected to be less than 5 ft in depth will be examined by a competent person to determine there to be no potential for cave-in do not require protective systems. EM 385-1-1 defines a "competent person" as "one who can identify existing and predictable hazards in the working environment or working conditions that are dangerous to personnel and who has the authority to take prompt corrective measures to eliminate them."

Excavation work will conform to EM 385-1-1 and 29 Code of Federal Regulations (CFR) 1926 Subpart P – *Excavations*, UXB-KEMRON procedures for Excavation and Trenching, and the project APP. Excavations greater than 4 ft may constitute a confined space; therefore personnel will not be allowed to enter the excavation in sections greater than 4 feet. Confirmation sampling will be performed from the bucket of the excavator.

Geotextile fabric (or 5 millimeter polyethylene sheeting) will be used to construct a temporary loading zone on which the trucks will stage while being loaded to ensure contaminated soil spills to the ground surface do not occur. The sheeting/fabric will extend from the truck to the edge of the excavation zone. The temporary loading zone will be moved as the leading edge of the excavation moves forward toward the drainage swale. Backfilling will commence after the excavation has been completed and analytical results from the confirmation samples have demonstrated that soil above the RGs has been removed from the site.

The excavator will be decontaminated prior to use, after completion of the excavation phase, and after completion of the project. When a piece of equipment is removed from an excavation area its tracks, wheels, buckets, etc. will be properly decontaminated. The primary method of decontamination will be to remove clinging soil by using shovels, brooms, and brushes. The decontamination will take place in the excavation area where the soil removed will be picked up and placed into a dump truck for disposal. Once dry decontamination has been performed, decontamination procedures for the excavator will follow those in SOP 80.1 for a drill rig, which requires steam cleaning. A decontamination pad will be set up within the main staging area where pressure washing will occur. The decontamination pad will consist of a lined containment area designed to collect decontamination water, such that it can be collect with a sump pump, containerized until analytical results determine the proper disposal alternative. Note that it is the goal to minimize the use of water so an additional waste stream is not created.

3.6 Monitoring Well Abandonment

After confirmation of ability to achieve clean closure at the site, UXB-KEMRON will permanently close and abandon the monitoring wells 57MW1, 57MW2 and 57MW3. The wells will be abandoned in

accordance with the DEQ Memorandum of January 8, 2008 attached as Appendix C. Well abandonment may occur before or after excavation of impacted soils. The surface pad of each well and associated bollards will be removed. The locations of wells 57MW2 and 57MW3 will be re-verified with a handheld GPS and the geospatial data will be recorded in the project record prior to emplacement of backfill, which may partially or entirely cover these abandoned well locations, based upon the desired finish grade of the site. The well abandonment will be conducted by an appropriately experienced and licensed driller, and the Commonwealth of Virginia Uniform Water Well Completion Report, which includes well abandonment information, will be completed for each well.

IDM generated form well abandonment and decontamination will be handled in accordance with SOP 70.1 for IDM. IDM will be containerized and properly characterized prior to disposal.

3.7 Waste Characterization and Disposal

Samples, as described in *Section 5.4,* will be collected from the soil and used to assess the appropriate disposal options for the soil prior to excavation. Composite samples will be collected from the soil, as specified by the receiving facility. These samples will be collected during the pre-excavation waste characterization phase, as discussed in *Section 5.4*.

The receiving facility may require that all soils at RFAAP be analyzed for explosives (due to the installation mission). Samples were collected for explosives during the RFI. Explosive compounds were not detected in any of the samples collected at SWMU 57. Data from the RFI are fully usable and will be provided in conjunction with generator knowledge regarding waste at SWMU 57 to demonstrate the soils have not been impacted by explosive compounds.

As discussed in *Section 3.5,* direct load-out of the soil will be performed. Each waste type generated during this effort will require a different disposal method based on its waste characterization results. While no hazardous waste is anticipated at the site, should hazardous waste be encountered, it will be appropriately segregated, containerized and either treated to render it nonhazardous or disposed in a RCRA Subtitle C Landfill. The excavated soil that is classified as non-hazardous waste will be disposed in a RCRA Subtitle D Landfill.

The decontamination fluid that is characterized as non-hazardous waste will be containerized and transported for off-site disposal at an appropriately licensed facility or disposed in the RFAAP WWTP after RFAAP approval.

UXB-KEMRON will act as the agent for the Army for treatment and disposal of non-hazardous wastes. UXB-KEMRON will select the final disposal facility for the waste based on several factors including but not necessarily limited to:

1. Subtitle D landfill to accommodate incoming waste.
2. Solicitation of bids.
3. Verification of permits and insurance.
4. The disposal facility must meet the permit compliance requirements.

UXB-KEMRON will coordinate with RFAAP regarding the selection of the disposal facility. Contact information for disposal facilities selected for the SWMU 57 IM will be presented in writing after final selection.

3.8 Backfill and Site Restoration

Following removal of the soil containing COCs above RGs and receipt of confirmation sample laboratory analytical results demonstrating achievement of the CMOs, site restoration activities will commence.

Off-site borrow material will be placed from the grade of the storm water culvert under the adjacent road and sloping toward and into the drainage swale to provide proper drainage. The finished grade will be such that the grass can be maintained and ponding will not occur. The backfilled areas will be seeded with grass and straw will be applied over the seed. Erosion and sediment controls will be left in place to allow sufficient time for the grass to become established.

Clean borrow material will be selected that has physical characteristics consistent with the existing soil at SWMU 57. Details regarding the sampling protocols for borrow material are presented in Section 5.5. After the site restoration activities are completed, UXB-KEMRON will demobilize all equipment off site. The Interim Measures Completion Report will be prepared to document clean closure activities and verification that clean closure to the standards established in the Final RFI/CMS has been achieved. The Interim Measures Completion Report will demonstrate that CMOs for this site have been achieved.

3.9 Prepare Interim Measures Completion Report

All field activities will be documented in conformance to the IAWP, and conducted in conformance to the IAWP, SSHSP, QAPP and other associated site specific and applicable RFAAP requirements. An Interim Measures Completion Report will be prepared to document all activities and verification that clean closure to the standards established in the Final RFI/CMS has been achieved. The Interim Measures Completion Report will demonstrate that the CMOs for this site have been achieved.

4.0 PROJECT SCHEDULE

The field activities to be performed as part of the SWMU 57 IM are scheduled to commence at the end of December 2010. The proposed schedule of project tasks is provided on Appendix A. KEMRON plans to implement the IMWP for SWMU 57 in conjunction with the IMWP for SWMU 40.

[NOTE: The project schedule will be updated in each subsequent edition of this work plan and will be updated and maintained throughout the project.]

5.0 FIELD SAMPLING PLAN

This FSP describes the field defines the procedures and methods that will be used to collect field samples during implementation of the IMWP. Contents included in this FSP include: procedures for collection of confirmation groundwater sampling, soil confirmation samples, and waste characterization samples for soil and groundwater; hand auger borings and soil sampling.

The QAPP (Appendix B) describes the policy, organization, functional activities, analytical methods, and quality assurance (QA) and QC protocols necessary to achieve the project DQO. This QAPP was developed in accordance with USACE *EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans* (USACE, 2001) and the Intergovernmental Data Quality Task Force *Uniform Federal Policy for Implementing Environmental Quality Systems* (UFP QAPP, IDQTF, 2005), and is to be used in conjunction with the FSP. This FSP was developed in accordance with USACE EM 200-1-3, *Requirements for the Preparation of Sampling and Analysis Plans* (USACE, 2001), and is to be used in conjunction with the QAPP (Appendix B).

5.1 Project Description

The project history and the planned Interim Measures work has been presented in the Introduction, *Section 1.0*, and the Organization and Technical Approach Plan, *Section 2.0 and 3.0*, of this IMWP, respectively. Field sampling activities will be conducted as part of the planned Interim Measures work. Sample management will follow SOP 50.1 of the MWP for labels, and 50.2 for packaging. The field sampling activities are discussed below.

5.2 Project Organization

A discussion of project personnel organization and responsibilities was previously provided in *Section 2.0* of this IMWP. Coordination of sample collection activities will be the responsibility of the Field Supervisor, who is responsible for running site operations. Field sampling technicians will be responsible for collection and delivery of samples to the laboratory. After delivery, the Project Chemist will be responsible for ensuring proper analysis and timely delivery of sample results by the laboratory.

5.3 Objectives and Scope

Samples to be collected during IM the include groundwater sampling, soil confirmation sampling, and waste characterization samples for surface water and soil. QC samples, such as field duplicate samples, rinse blanks, matrix spike/matrix spike duplicate (MS/MSD), etc., will also be collected as described in the QAPP (under separate cover). The following sections describe the function of each type of field sample.

5.4 Waste Characterization Samples

Waste characterization samples will be collected and analyzed to determine the appropriate disposal methods of waste streams resulting from the IM at SWMU 57. Two types of waste streams will be generated during the IM: solid (soil) and liquid (decontamination water and pond water).

Soil that will be excavated will be characterized to evaluate whether it is a RCRA characteristic hazardous waste as described in Part 40 of the Code of Federal Regulations (CFR) Part 261, Subpart C (as referenced in the Virginia Hazardous Waste Management Regulations).

Soil waste characterization samples will be collected during site delineation to assess the appropriate disposal options for the soil excavated. The samples will be given the identifiers 57WC1, 57WC2, etc. Soil samples will be collected in the area of historic soil samples 57SB7 and 57SB8 using a hand auger and will follow the SOP 20.11 of the MWP for hand auger borings and soil sampling. Both surface (0-1 foot) and subsurface (greater than 2 feet) samples will be collected at each location. The soil sample(s)

will be submitted to a DoD ELAP certified laboratory as identified in the QAPP (Appendix B), and analyzed for TCLP metals and RCRA waste characteristics (corrosivity as pH, reactivity, and ignitability) and any other parameter required by the approved landfill, which may include TCLP for VOCs and Paint Filter Liquid Test. Liquid waste characterization samples from decontamination procedures will be submitted to the analytical laboratory and analyzed for chemical oxygen demand (COD), TAL metals, and pH.

Surface water characterization samples will be collected during pre-excavation sampling to assess if treatment will be required prior to disposal at the wastewater treatment plant at the installation. The samples will be given the identifiers 57SW1, 57SW2, etc. Surface water samples will be collected in accordance with the procedures outlined in the SOP 30.3 of the MWP. The surface water samples will be submitted to a pre-approved laboratory and analyzed for parameters specified by the RFAAP water treatment facility. UXB-KEMRON will identify a permitted off-site disposal facility for soil disposal, and also will coordinate with RFAAP wastewater treatment plant (WWTP) personnel regarding the necessary waste characterization. This pre-excavation waste characterization will ensure that no pond water, associated residual pond material or soils will need to be temporarily stored on site, but can be directly loaded and transported to the appropriate disposal facility.

5.5 Borrow Material Soil Samples

The borrow material/top soil will be sampled at a rate of 1 sample/1,000 cubic yards (i.e., estimated as one sample from the borrow material, one sample from the top soil). Erosion control measures will be implemented and excavation areas will be seeded. The borrow material and top soil will be analyzed for TAL metals, PAHs, pesticides/PCBs, and pH, and will have the following identifier, 57BF1, 57BF1, etc. A composite soil sample will be collected following procedure SOP 30.1 of the MWP.

5.6 Confirmation Soil Samples

Confirmation soil samples will be collected during 2 phases of the IMWP. Soil samples adjacent to steam line supports within the drainage swale will be collected prior to mobilization. The sampling strategy to be employed is a biased sampling strategy [Standard Operating Procedure (SOP) 30.7], because the source of the contamination is known. Surface and subsurface soil samples will be collected for chemical analysis following the procedures outlined in standard operating procedure (SOP) 30.1 and as described in Section5.2 of the MWP (URS, 2003). Samples will have the identifier 57B1-2.5', 57B1-5', 57B2-2.5, 57B2-5', etc. Soil samples will be collected from the bucket of the excavator, after collecting a sample from the sidewall or floor of the excavation. The sample will be transferred to a stainless steel bowl for compositing

The soil core interval will be examined and classified by the site geologist and recorded in the field logbook and on the boring log consistent with SOPs 10.1 and 10.3, respectively. Soil will be extracted from the appropriate interval; sample containers were filled, labeled, and placed into coolers with ice and maintained at 4 degrees Celsius (°C).

Once the termination depth of the boring is reached and sample collection is completed, the borehole will be backfilled with excess soil from the boring and hydrated bentonite chips. Soil boring logs will be included in the IM Completion Report.

Soil confirmation samples also will be collected during the excavation of the pond and the drainage swale. Confirmation samples will be collected in accordance with the QAPP and will be identified by name (57CS1-57CS29). The sampling strategy to be employed is a biased sampling strategy [Standard Operating Procedure (SOP) 30.7], because the source of the contamination is known. The anticipated excavation area is illustrated in Figure 3; however, the final shape and depth of the excavation will be determined in the field. Planned confirmatory soil sample locations are illustrated on Figure 4. One soil

sample will be collected for laboratory analysis from each 25-ft by 25-ft area of the floor of the remediation area and from every 20-ft section of the wall of the excavation. Sidewall samples will be collected at the midpoint of the sidewall. For example, for an excavation wall that is 15 feet high, the sidewall sample will be collected at 7.5 feet. Samples will also be collected from inflection points and/or corners of the excavation at the base and side wall. Confirmatory soil samples will be analyzed for the site specific COCs, with data compared to the soil RGs specified in the Final RFI/CMS (per Sections 1.2 and 1.3 above). If sample analytical results demonstrate soil remains above the RGs, excavation will continue to a maximum depth of 15 feet bgs. The Final RFI/CMS documents that no exposure pathway for potential future use of the site exists beyond a depth of 15 feet bgs; therefore the maximum excavation depth for the impacted area is 15 ft bgs. The required horizontal extent of the excavation has been delineated by sampling conducted within the RFI, and presented in the Final RFI/CMS.

5.7 Groundwater Samples

Groundwater samples will be collected for chemical analysis in accordance with SOP 30.2 and as described in Section 5.2.10 of the MWP (URS, 2003) to confirm the ability to achieve clean closure. Groundwater sampling information will be recorded in the field logbooks as described in SOPs 10.1 and 10.2. Groundwater sampling field data sheets will be included in the IM Completion Report. Low flow purging and sampling techniques will be used to collect the groundwater samples from monitoring wells 57MW2 and 57MW3, and 57MW1 if possible. Purging in these monitoring wells will be performed at a rate of 500 milliliters per minute (ml/min) or less to minimize excessive drawdown. Sampling will follow the procedures outlined in Section 3.3 of SOP 30.2 of the MWP. Equipment used to purge and sample wells will be thoroughly decontaminated before and after use following SOP 80.1.

All equipment used for monitoring water quality parameters will be calibrated before use according to the manufacturer's instructions and SOP 40.1 of the MWP. Prior to the sampling round, the depth to water will be measured at each well to the nearest 0.01 ft using an electronic water level indicator in accordance with SOP 40.2 of the MWP. Calibration and measurement data will be recorded in the field logbook and on groundwater sampling forms.

A stainless steel, adjustable flow rate submersible pump will be used to collect the groundwater samples. The pump tubing will be connected to an in-line flow-through cell and the multi parameter meter probe will be connected to the flow cell to monitor water quality parameters during purging. Pumping will start and the pump rate will be adjusted to cause minimal drawdown (less than 0.2 ft if possible). A Horiba Model U-22 multi-parameter water quality meter, or equivalent, will be used to monitor pH, specific conductance, temperature, ORP, dissolved oxygen, and turbidity during purging. Purging will continue until parameter stabilization is achieved. Once purging is complete, the pumping rate will be reduced to its lowest steady rate and the in-line flow cell will be disconnected from the tubing to allow for sampling from the tubing directly into sample containers provided the laboratory.

Groundwater samples will be collected and containerized in the order of volatilization sensitivity of the parameters. Samples will be analyzed for TCL VOCs via EPA method SW 846 Method 8260B, antimony, arsenic, chromium, and manganese by EPA SW 846 Method 6010B or 6020. Filtered and unfiltered samples will be collected for metals analysis. The filtered metals samples will be filtered with an in-line high capacity 0.45-micron disposable filter. The appropriate sample containers will be filled, labeled, and placed into coolers with ice and maintained at 4 °C.

Sampling equipment will be decontaminated in accordance with the procedures outlined in the RFAAP MWPSOP 80.1 for non-dedicated sampling equipment. Non-dedicated sampling equipment, including field monitoring deceives, will be decontaminated between each sampling point. A decontamination station will be established for sampling equipment. The exact location of the decontamination station will

be determined in the field and the location will consist of an area outside the SWMU 57 near the proposed main staging area (See Figure 5).

6.0 ENVIRONMENTAL PROTECTION PLAN

This Environmental Protection Plan (EPP) has been prepared by UXB-KEMRON to establish site procedures to protect existing environmental conditions at the RFAAP and to minimize potential harmful impacts caused by interim measures to the environment. During active interim measures, a copy of the EPP will be maintained on-site at all times. The EPP will be supplemented as necessary before land disturbance activities other than those indicated are performed.

This EPP serves the following purposes:

1. To identify potential sources of pollution that could impact the quality of storm water discharges associated with the remediation and construction activities from the site.

2. To describe chemical spill controls and countermeasures associated with construction activity from this site.

3. To detail hazardous substances that could be used on the job site and a contamination prevention plan for these materials.

6.1 Applicable Regulations

The following potentially applicable Federal Regulations will be addressed by UXB-KEMRON through use of appropriate erosion and sediment controls in the field:

- Phase II storm water substantive permitting elements for small construction sites (40 CFR 122)

6.2 Pre Construction Survey of Existing Conditions

UXB-KEMRON will conduct a preliminary survey of the pre existing conditions at the project site prior to any intrusive activities. The initial survey will include a record of existing vegetation, utilities, and other appropriate site conditions. Existing site conditions will be recorded in a photographic log.

6.3 Previously Used Equipment

All UXB-KEMRON equipment will be decontaminated and cleaned prior to arrival at the subject site. Appropriate measures will be conducted to prevent any type of cross contamination from non related sites or improperly cleaned equipment. Dedicated equipment will be utilized when appropriate to minimize potential cross contamination.

6.4 Protection of Land Resources

The interim measures activities will include intrusive actions which may potential affect local land resources. UXB-KEMRON will provide erosion and sedimentation control measures around the perimeter of the excavation area and an improved construction road will minimize the disturbances of land surrounding the work area. The excavation area will be permanently stabilized following the backfilling and grading tasks to return the area to pre existing conditions. The erosion and sedimentation control measures are discussed in greater detail in Section 7.0 of this report.

6.4.1 Work Area Limits/Traffic Control

The soil excavation work area will be isolated from surrounding areas by a perimeter silt fence. UXB-KEMRON will also utilize a main staging area which also will be isolated by a silt fence. The staging area will provide all UXB-KEMRON employees, subcontractors, and site visitors a location to park and stage project specific equipment and materials. An improved construction access road will be installed next to the soil excavation area and the use of a construction entrance will minimize sediment tracking

from the project site onto permanent roadways. An effort will be made to keep all project operations within the soil excavation area and staging area to minimize any disturbances to surrounding land.

6.4.2 Landscape

Vegetation within the project areas, including the staging area and soil excavation area, may be cleared prior the project activities. In the event that particular trees or shrubs are to be protected within this area, then they will be marked with a ribbon.

UXB-KEMRON does not anticipate the disturbance or removal of any vegetation outside of the project area.

6.4.3 Unprotected Erodible Soils

UXB-KEMRON does not anticipate the disturbance of soil outside of the project area. Soils within the excavation area protection measures will be installed when appropriate. Temporary stabilization measures will be implemented and designed to be incorporated into the permanent stabilization plan. Permanent vegetative stabilization is considered part of the site restoration task and will be conducted once all backfilling and grading activities are complete.

6.4.4 Disturbed Areas

Appropriate erosion and sedimentation controls will be installed prior to the disturbance of any soils. The protective measures shall reduce the potential risk of migration of sediments from the excavation area to surrounding land. Temporary erosion control measures will include silt fencing, construction entrances, improved access roads, and other appropriate structures. The erosion and sedimentation control measures are discussed in greater detail in Section 7.0 of this report.

6.4.5 Staging and Work Areas

The work areas for the interim measures will include a soil excavation area and a staging area. An improved access road will be installed between the two. The location of the site features is illustrated in Figure 5.

6.5 Water Resources

The interim measures are designed to remediate a pond area and reduce the potential for contaminants to migrate into surrounding water bodies. The erosion and sedimentation control plan details the protective measures which will be installed to prevent materials from migrating outside the interim measures work area.

6.5.1 Wastewaters

Water will be generated during the interim measures by three distinct processes, including physically removing the waters from the pond, by purging the monitoring wells prior to collection of groundwater samples, and from the decontamination procedures. The water generated from the pond will be pumped out using mobile vacuum trucks and will be disposed either at the RFAAP WWTP or at an appropriately licensed offsite facility. Likewise, the monitoring well purge water will be disposed either at the RFAAP WWTP or an appropriately licensed off-site facility. The fluids generated during decontamination will be contained in tanks or approved containers and will be disposed similarly. All fluids will be properly sampled for disposal characteristics prior to disposal. All disposal records will be maintained in the project file.

6.5.2 Diversion Operations

Water removal operations will be controlled to minimize any impacts or disturbances to downgradient habitats. It is not anticipated that dewatering activities will negatively impact surround areas.

6.5.3 Fish and Wildlife

It is not anticipated that the activities associated with the interim measure will negatively impact surrounding fish or wildlife habitats. There are currently no listed endangered species in the interim measures work area.

6.6 Air Resources

Dust control may be implemented as needed once site excavation activities have been initiated and during windy conditions while site grading and remediation activities are occurring. Dust from the site will be controlled by using a mobile pressure-type distributor truck to apply water to disturbed areas. The mobile unit will apply water at a rate to prevent runoff and ponding. Water will be applied whenever the dryness of the soil warrants it based on air monitoring results.

6.7 Noise

The interim measures will be conducted in an area that will not affect residential areas, and will have limited potential impact to RFAAP workers. While it is anticipated the majority of work will be conducted during normal working hours, noise from the project site, weekend or extended hours of work will not have a negative impact the local environment or onsite workers. Threshold levels for onsite noise and proper protective equipment are detailed in the Site Specific Health and Safety Plan.

6.8 Waste Disposal

Waste will be generated during the interim measures by two distinct processes, first by physically removing the soils from the pond and second from the typical solid waste accumulation. All waste generated will be properly sampled for disposal characteristics prior to disposal. All disposal records will be kept in the project file.

6.8.1 Solid Wastes

Typical solid waste will be contained in an onsite storage device such as a 10 yard roll-off or smaller device. Solid waste will be disposed of on a regular basis at an offsite facility. The soil removed from the pond area will be loaded into dump trucks or appropriate storage containers and transported under manifest to an approved offsite disposal facility. All waste streams will be properly segregated and isolated to prevent any cross contamination.

6.8.2 Chemical Wastes

Any chemical wastes generated during the interim measures will be containerized in appropriated containers such as drums. The chemical wastes shall be stored onsite in the staging area until proper disposal is arranged at an approved offsite disposal facility.

6.8.3 Hazardous Waste

No hazardous wastes are anticipated to be generated during the interim measures. In the event of hazardous waste generation, then all measures will be taken to properly segregate the hazardous from non-hazardous wastes. The hazardous waste will be loaded into appropriate storage containers and transported under manifest to an approved offsite disposal facility or treated to render them nonhazardous

prior to disposal. All waste streams will be properly segregated and isolated to prevent any cross contamination.

6.9 Burning

There will be no burning activities associated with the interim measures.

6.10 Historical, Archaeological, and Cultural Resources

It is not anticipated that any historical resources will be encountered during the interim measures. These resources may include: human remains, bones, structures, and artifacts. However, in the event that such resources are encountered then the project manager will stop work and contact the COR and RFAAP.

6.11 Post-Remedy Cleanup

Once the interim measures are complete UXB-KEMRON will conduct site restoration to bring the project areas back to pre-construction conditions. All solid wastes associated with the project will be removed from the site prior to closeout.

6.12 Restoration of Landscape Damage

Once the interim measures are complete UXB-KEMRON will conduct site restoration to bring the project areas back to pre-construction conditions. Vegetation will be permanently stabilized and erosion control measures will be removed once stabilization is complete.

6.13 Maintenance of Erosion Control Facilities

Any permanent or temporary erosion control measures will be maintained during the entire course of the project and until permanent stabilization is achieved.

6.14 Training of Personnel

All UXB-KEMRON personnel will be provided training for their assigned project tasks. A complete listing of training requirements is included in the project APP.

6.15 Spill Prevention and Response

This Spill Prevention and Response Plan has been developed to prevent the contamination of soils, water, atmosphere, uncontaminated areas, equipment or material by the uncontrolled release of hazardous waste and materials during field operations involved in this project.

6.15.1 Potential Spill Types

The following is a generalized list of materials or substances that are anticipated to be stored on site during the proposed interim measures:

- Detergents for decontamination efforts;
- Diesel fuel, hydraulic oil, and other vehicle maintenance substances; and
- Small quantities of lubricants, cleaners, marking paints, and/or landscaping materials (fertilizer, peat, lime, etc.).

All petroleum products and other hazardous materials will be stored in secondary containment basins, spill pallets, or in double walled tanks to contain the materials in the event of a spill. Fueling operations to fuel heavy earthmoving equipment will be conducted using 100-gallon fuel tanks mounted in pickup trucks. All other materials will be commercially available lubricants, cleaners, or landscaping materials that may be utilized during the remediation effort. Any materials brought onsite will be logged on a Hazardous Materials List. This list will include the MSDS and any appropriate spill protection measures.

6.15.2 Spill Prevention

To prevent the introduction of any potentially hazardous substance into any undisturbed media the following actions will be followed by KEMRON. Detergents and small containers of oil, grease, antifreeze, hydraulic fluids, etc., if any, will be stored within an enclosed storage container. Containers 5-gallons or greater will be stored within a secondary containment area or on secondary containment pallets. Diesel fuel will be stored in portable tanks located in truck beds. Any spill basins/pallets utilized will be designed to contain at least 100 percent of the total contents of all materials stored in the area plus an allowance for precipitation. A small sump or low point will be designed to serve as a monitoring point for any leaks or spills from the containers.

Small quantities of lubricants, cleaners, marking paints, and/or landscaping materials may be stored on site during the interim measures. These materials, when not in use, will be stored in a compatible storage cabinet located in a staging area. Practices that will be followed to reduce risks associated with these materials are as follows:

- Products will be kept in original containers unless they are not re-sealable.
- Original labels and material safety data sheets (MSDS) will be retained onsite.
- If surplus product must be disposed, manufacturers' or local- and State- recommended methods for proper disposal will be followed.

The following Good Housekeeping material management practices will be used to reduce the risk of spills or other accidental exposure of materials and substances to storm water runoff. These practices will be employed during the interim measures:

- An effort will be made to store only enough products required to accomplish the task.
- All materials stored on site will be stored in a neat, orderly manner in their appropriate containers and, if possible, under a roof or other enclosure.
- Products will be kept in their original containers with the original manufacturers' labels.
- Substances will not be mixed with one another unless recommended by the manufacturer.
- Whenever possible, all of a product will be used before disposing of the container.
- Manufacturers' recommendations for proper use and disposal will be followed.
- The assigned individuals will inspect areas daily to ensure proper use and disposal of materials on site.
- Sanitary facilities will be serviced regularly and will be subject to inspection. Sanitary deficiencies will be corrected within 24 hours of the inspection.
- Construction cleanup will proceed as construction progresses and will consist of the removal of mud, oil, grease, soil, gravel, trash, scrap, debris, and excess materials that are unsightly or may cause the slipping or tripping of site workers. Construction cleanup on roadways, work access and staging areas will occur daily.
- Solid waste generated by KEMRON will be placed in containers which are emptied regularly at an approved offsite facility. Handling, storage, and disposal will be conducted to prevent contamination. All environmental and investigational wastes segregated from solid wastes which will be transported in compliance with Federal, State, and local solid waste disposal requirements. A Subtitle D RCRA permitted landfill is the minimum acceptable off-site disposal option.
- Fuel, oil, and chemical dispensing operations will be inspected daily for leaks and spills. Spill wastes will be classified, managed, stored and disposed in accordance with Federal, State, and local solid waste disposal requirements.

6.15.3 Spill Countermeasures

In the event of a spill or release of a solid, UXB-KEMRON will remove and place contaminated materials in a drum with a cover. The container should be appropriately labeled and disposed of as soon as possible.

Liquid spills (e.g., oil from vehicles, etc.) will be absorbed with sand or other appropriate absorbent material (sand would only be used over an impervious substrate such as asphalt paving). The absorbent material will be placed in a drum with a cover. The container should be appropriately labeled and disposed of as soon as possible.

In the event of a discharge of liquid into the soil, UXB-KEMRON's project manager or designee will immediately identify the location of the discharge and take appropriate remedial actions to eliminate further spillage. The discharged liquid material will be controlled and disposed of as described above. If a reportable discharge of any material stored in drums, tanks, or other containers, etc. occurs, the following steps will be followed:

1. Notify the RFAAP representative and RFAAP Security Police Dispatcher (540-639-7323), SSHO, and the Installation Spill Response; in the event of a reportable quantity discharge, notify the National Response Center (800) 424-8802 if authorized by the COR or RFAAP point of contact.
2. Contain and eliminate the discharge (if not prevented by safety considerations).
3. Remove/retrieve any discharged liquids (if not prevented by safety considerations).
4. Isolate the spill area restricting access to unauthorized personnel.
5. Decontaminate the spill area, if necessary.
6. Prepare a spill report.

The project manager, or his designee, will be responsible for completing the spill reporting form and for reporting the spill to the appropriate state or local agency if authorized by the COR or RFAAP point of contact.

The Spill Report will contain the following:

- Description of the material spilled including identity, quantity, and a copy of any waste manifests or bills of lading. Identify the cause of the spill. (If possible, MSDS sheets for spilled material and material used to clean it up will be included in any Spill Reports generated.)
- Exact time and location of the spill and a description of the area involved.
- Containment procedures utilized.
- A description of the corrective actions implemented during the spill including the disposal of the cleanup residues.
- A summary of the communications between UXB-KEMRON and Government officials.

Because no large quantities of hazardous liquids will be involved, no additional supplies or equipment beyond those previously specified are expected to be needed for the duration of this project.

6.15.4 Spill Mitigation Equipment

Spill control equipment will be available in the event of a spill or release at the site. The following quantities of materials are based on a small scale spill, less than 50 gallons of liquid waste and 500 lbs of solid waste:

- Oil absorbent pads (one bale)
- Oil absorbent material (100 lbs.)
- Oil absorbent booms (40 feet)

- Front-end loader or excavator
- 55-gallon drums, DOT UN1A1 UN1A2 (2 total)
- Shovels
- Decontamination supplies and PPE
- Hand operated pump

Spill response equipment will be inspected and maintained as necessary to replace any materials used in spill response activities. Regardless of the type of spill (liquid or solid), the following measures will be taken to isolate the spilled material:

- The PM will be notified immediately when a spill, or the threat of a spill, is observed.
- The PM will assess the situation and determine the appropriate response and contact RFAAP and the Operating Contractor.
- Isolate and contain the spill area.
- Restrict access of unauthorized personnel
- Prevent contact with the spilled material
- Relocate upwind and upgradient of the spilled material
- Take air, soil, or appropriate samples to determine if cleanup is complete

6.15.5 Notification Procedures

In the event of a spill the project manager will contact the COR and RFAAP point of contact. If authorized, UXB-KEMRON will contact the appropriate authorities at the numbers below.

CHEMTREC	800-424-9300
National Response Center	800-424-8802
National Poison Control Center	800-362-9922
Federal Emergency Management Agency	202-646-2400
Centers for Disease Control	800-232-4636
Poison Control Center	800-222-1222
UXB-KEMRON Environmental Services, Inc.	800-548-6938
Radford Army Ammunition Plant Contact – James McKenna	540-731-5782
Installation Fire Department	16 (on post)
Installation Security Police	540 639-7325
Installation Safety Department	540-639-7294
Installation Spill Response	540-639-7324
Installation Medical Facility	540-639-7325
Emergency Fire	911
Emergency Police	911
Emergency Medical Services (EMS)	911

(*) Hospital:
New River Valley Medical Center
2900 Lamb Circle
Christiansburg, VA 24073
(540) 731-2530

(*) The above emergency agencies shall be contacted and notified on the specific hazards on this project. Coordination for special emergency response requirements with these agencies shall be completed upon arrival.

7.0 EROSION AND SEDIMENTATION CONTROL PLAN

7.1 General Purpose

This Erosion and Sediment Control Plan (ESCP) for the RFAAP interim measures has been prepared by UXB-KEMRON to identify and address erosion control regulations and protection measures. The objective of this ESCP is to establish site procedures to control storm water and prevent the transport of sediments or contaminants from the project site. This ESCP was prepared in accordance with the minimum standards and specifications of the Virginia Erosion and Sediment Control Handbook, the Virginia Erosion and Sediment Control Law, Regulations, and Certification (VESCLR&C) Regulations. KEMRON will ensure that all personnel are qualified to perform the work as outlined within the regulations. During active project operations, a copy of this ESCP will be maintained on-site at all times. The ESCP will be supplemented as necessary before land disturbance activities other than those indicated are performed.

7.2 Applicable Regulations and Plan Approval

It is not anticipated that the interim measures for this project will disturb more than one (1) acre of land, therefore, federal regulatory standards for construction sites are not anticipated to be applicable. However, UXB-KEMRON will apply appropriate erosion and sediment controls given the proximity of the site to the New River.

7.3 Erosion and Sedimentation Control Plan

As part of the interim measures being performed at the SWMU 57, UXB-KEMRON will:

- Perform initial delineation soil and groundwater sampling prior to soil excavation;
- Temporarily block culvert under East Pond Road and manage surfacewater collection via pumps, poly tanks and discharge in drainage swale northwest of excavation.
- Excavate impacted soils from the pond, and disposal of soils in containers for transport to an offsite facility;
- Perform verification sampling following the Interim Measures;
- Backfill excavated areas and perform in-place compaction testing;
- Restore the SWMU 57 excavation areas affected by the remedial activities.

The anticipated major land disturbance will consist of the following activities:

1. Installation of construction access road;
2. Installation of perimeter silt fencing and other erosion control devices;
3. Clearing of trees, brush and all surface vegetation within the footprint of the SWMU 57 pond excavation;
4. Excavation and removal impacted soil with heavy equipment;
5. Grading and backfilling of the resulting area following removal; and
6. Restoration of all disturbed areas.

The erosion and sediment control measures will be implemented, installed, inspected, and maintained according to the minimum standards and specifications of the Virginia Department of Conservation *Erosion and Sediment Control Handbook* (1992). The locations of required erosion, sediment, and storm water control measures are shown in Figures 5.

Temporary erosion and sediment control structures must be in place and functional before earth moving disturbance activities begin. Portions of the temporary erosion control measures may be removed at the beginning of each day as required to complete the work, but will be replaced at the end of the day.
Structural, Vegetative, Management Strategies, Material Handling and Waste Management will be

utilized for erosion and sediment control at the SWMU 57 project. A description of the Best Management Practices (BMPs), Installation Schedule, Maintenance and Inspections, and Responsible Personnel for the project are provided below.

The stormwater culvert shown on Figure 5 will be temporarily blocked on the east side of Pond East Road, or diverted around the excavation during the IM. It is anticipated that stormwater run-on to the excavation will be managed by allowing the stormwater to collect east of East Pond Road by blocking the culvert. During rain events, stormwater will be allowed to collect in the existing depression and the water will be pumped into a tank and disposed on in the drainage swale northwest of the excavation.

7.3.1 Structural Practices

Stabilized Construction Access Road

BMP Description: One (1) construction access will be installed adjacent to the soil excavation area and staging area. The construction access will intersect with Pond East Road. This access road and entrance will be installed to control the tracking of dirt off the project site and will be installed according to the Construction/Exit Detail illustrated in Figure 5.

Installation Schedule: The stabilized construction access road will be installed before remedial actions begin on the site. The construction access road will remain in place until the excavation activities are complete and final stabilization vegetation is installed at the site.

Maintenance and Inspection: During active remediation of the site, the construction access road will be inspected every 14 calendar days and within 24 hours after storm events of 0.5 inches or greater or more frequently during periods of heavy use. The construction access road will be maintained in a condition that will prevent tracking offsite. This could require adding additional crushed stone. All sediment tracked, spilled, dropped, or washed onto Pond East Road will be swept daily.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

Silt Fence

BMP Description: Silt fencing will be placed along the perimeter of the soil excavation and staging area(s) as illustrated in Figure 3. Standard silt fence will be utilized for this project unless it becomes necessary to upgrade to a stronger variety. Standard silt fence will be used along the down gradient limits of the staging area.

Installation Schedule: The silt fences will be installed before any remedial actions begin or any materials are brought onsite and as determined necessary by the UXB-KEMRON project manager and the RFAAP representative.

Maintenance and Inspection: After initial installation, the silt fencing will be inspected every 14 calendar days and within 24 hours after storm events of 0.5 inches or greater during active remediation to ensure it is intact and that there are no gaps where the fence meets the ground or tears along the length of the fence. If gaps or tears are found during the inspection, the fabric will be repaired or replaced. Accumulated sediment will be removed from the fence base if it causes "bulging" or reaches one-third the height of the fabric height. If accumulated sediment is creating noticeable strain on the fabric and the fence might fail from a sudden storm event, the sediment will be removed more frequently. Before the fence is removed from the project area, the sediment will be removed. The anticipated life span of the silt fence is 6 months.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

7.3.2 Vegetative Practices

Surface Roughening

BMP Description: The surface of re-graded areas will be roughened to reduce runoff velocity and to aid in the establishment of vegetative cover.

Installation Schedule: To be completed following re-grading activities and prior to ground cover establishment and permanent stabilization.

Maintenance and Inspection: Stabilized areas will be inspected every 14 calendar days and within 24 hours of storm events of 0.5 inches or greater until a dense cover of vegetation has become established.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

Topsoiling

BMP Description: Topsoiling will be placed on all restored surfaces as necessary to allow permanent stabilization and vegetative growth. Imported topsoil will contain 5-10% organic matter. Maximum particle size, ¾ inch with maximum 3% retained on ¼ inch screen. Topsoil shall be free of sticks, stones, roots, and other debris.

Installation Schedule: Topsoil will be installed within 14 days of re-grading following remediation activities.

Maintenance and Inspection: Stabilized areas, topsoiled and seeded, will be inspected every 14 calendar days and within 24 hours of storm events of 0.5 inches or greater until a dense cover of vegetation has become established.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

Temporary Stabilization/Seeding

BMP Description: All re-graded areas that will be left dormant for greater than 30 days shall be seeded with fast-germinating temporary vegetation immediately following grading. Seeding will be performed dependent on the time of year, in accordance with the Seeding Chart in the Virginia Erosion and Sediment Control Handbook.

Installation Schedule: Temporary or permanent stabilization of the SWMU 57 Site will be completed within 14 days of final grading or earth moving activities, unless construction activity will resume on a portion of the site within 30 days from when activities ceased.

Maintenance and Inspection: Temporarily stabilized areas will be inspected every 14 calendar days and within 24 hours of storm events of 0.5 inches or greater until a dense cover of vegetation has become established. If failure is noticed at the seeded area, the area will be reseeded within one week.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

Permanent Stabilization/Seeding

BMP Description: All areas disturbed by construction will be stabilized with permanent seeding following finished grading or when areas are left dormant for more than 30 days. Erosion control matting may be used for steep slopes, as determined necessary by the project manager, and mulch will be used for

mild slopes to secure seeding. The permanent seed mixture for the SWMU 57 interim measures will conform to the Virginia Erosion and Sediment Control Handbook and seasonal considerations.

Installation Schedule: All areas disturbed by the remedial activities will be stabilized with permanent seeding within 14 days of final grading or earth moving activities, unless construction activity will resume on a portion of the site within 30 days from when activities ceased. Sediment control measures will not be removed until permanent vegetative cover is established.

Maintenance and Inspection: All seeded or re-vegetated areas will be inspected every 14 calendar days and within 24 hours after storm events of 0.5 inches or greater until a dense cover of vegetation has become established. If failure is observed, the area will be reseeded, fertilized, and/or mulched. After remedial actions are completed at the site, permanently stabilized areas will be monitored until final stabilization is reached.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

7.3.3 Management Strategies

Initial Site Preparation Work

BMP Description: UXB-KEMRON will be responsible for the implementation and execution of the specified erosion and sediment controls. The work schedule will be sent to RFAAP and regulatory agencies a minimum of 2 weeks in advance to any site work or land disturbance. The site preparation work that will be completed prior to the full scale remedial actions include the installation of erosion control devices and storm water runoff controls such as silt fences. Additional pre-construction activities include surveying and staking of the site. The location and type of control measures to be used are illustrated in Figure 5.

Installation Schedule: Erosion, sediment, and storm water control features will be installed and/or constructed before the start of any earth-disturbance activities. All erosion control features will remain in place until permanent vegetation is established over disturbed surfaces.

Maintenance and Inspection: The UXB-KEMRON project manager will be responsible for ensuring the installation and maintenance of all erosion, sediment, and storm water control practices and all site control measures will be inspected no less than once every fourteen (14) calendar days and within 24 hours after storm events of 0.5 inches or greater until a dense cover of vegetation during final stabilization has become established. Inspections will be documented and any non-functional or damaged control structure will be repaired within 24 hours. Any silt fence control device with 50% accumulated sediment will be either replaced or the sediment removed.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

Water Used to Control Dust

BMP Description: Dust control will be implemented as needed once site excavation activities have been initiated and during windy conditions while site grading and remediation activities are occurring. Dust from the site will be controlled by using a mobile pressure-type distributor truck to apply water to disturbed areas. The mobile unit will apply water at a rate to prevent runoff and ponding. Water will be applied whenever the dryness of the soil warrants it based on air monitoring results.

Installation Schedule: Dust control will be implemented as needed once soil excavation has been initiated and during windy conditions while excavation is occurring. Spraying of water will be performed as the dryness of the soil warrants it based on air monitoring levels for dust.

Maintenance and Inspection: At least one watering unit will be available at all times to distribute water to control dust in the remediation areas. Each mobile unit will be equipped with a positive shutoff valve to prevent over watering of the remediation area.
Responsible Staff: UXB-KEMRON or Subcontractor(s).

Street Cleaning/Sweeping
BMP Description: Street sweeping will be conducted as needed if sediment is observed to be transported onto paved roads or parking areas. Sweeping or cleaning of the roads may be done with air blowers, manually by hand sweeping, or by machine. Sediment recovered by hand methods will be returned to the site.

Installation Schedule: Street cleaning/sweeping will occur as needed as determined by on-site project management.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

Post Construction
BMP Description: The pre- and post-construction runoff volumes will remain relatively the same with no significant changes due to the remedial actions. Project-derived pollutant run-off is not expected to occur after construction operations have been completed and the site has been stabilized. Once final vegetation has developed, all remaining erosion and sediment controls will be removed.

Installation Schedule: Once final vegetation has developed, all remaining erosion and sediment controls will be removed.

Maintenance and Inspection: The UXB-KEMRON project manager will be responsible for ensuring the competency of all erosion, sediment, and storm water control practices at the site. Following remedial activities the protective measures will be inspected no less than once every fourteen (14) calendar days and within 24 hours after storm events of 0.5 inches or greater until a dense cover of vegetation during final stabilization has become established. Inspections will be documented and any non-functional or damaged control structure will be repaired within 24 hours. Any silt fence control device with 50% accumulated sediment will be either replaced or the sediment removed.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

General Maintenance and Inspections
BMP Description: The inspection schedule and documentation procedures have been designed so that vegetation, erosion, sediment control measures, and other protective measures are kept in good and effective operating condition.

The following list includes, but is not limited to, areas that will be inspected by qualified personnel at least once every (14) fourteen calendar days and within 24-hours of the end of a storm that is 0.5 inches or greater:

- Disturbed areas of the construction site that have not undergone final stabilization;
- Areas used for the storage of materials that are exposed to precipitation that have not undergone final stabilization;
- Structural control measures; and
- Locations of vehicle ingress and egress.

Installation Schedule: In general, during the remedial actions, all erosion, sediment, and storm water control measures will be visually inspected daily but at a minimum a formal inspection will occur at least

every fourteen (14) days and after each runoff-producing rainfall event. Any required repairs will be made within 24 hours of detection. Based on the results of the inspection, any inadequate control measures or control measures in disrepair will be replaced, modified, or repaired as soon as practicable (i.e., before the next rain event if possible) but in no case more than twenty-four (24) hours after the need is identified. In general, all repairs to the erosion and sediment control structures shall be made within twenty-four (24) hours or as soon as practicable.

Maintenance and Inspection: The stabilized areas will be checked regularly to ensure that a good stand of vegetation is reached. Areas will be fertilized and reseeded as deemed necessary by the project manager.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

Staging Area

BMP Description: The primary staging area for the project will accommodate parking, work trailers, portajohns, materials containment, and a solid waste dumpster. All hazardous materials such as petroleum products and equipment maintenance fluids will be properly stored in this area. The location of staging area is illustrated in Figure 5.

Installation Schedule: The staging area will be constructed after clearing and grubbing and some light grading work is conducted at the site.

Maintenance and Inspection: The staging area will be inspected every 14 calendar days and within 24 hours after storm events of 0.5 inches or greater. The staging area will be kept clean, well organized, and equipped with ample cleanup supplies as appropriate for the materials being stored. Material safety data sheets, material inventory, and emergency contact numbers will be maintained by the UXB-KEMON project manager.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

Vehicle Fueling and Maintenance

BMP Description: Several types of vehicles and equipment will be used on site throughout the project, including, trucks and trailers, excavators, and bulldozers. All major equipment/vehicle fueling and maintenance will be performed onsite by a subcontractor or UXB-KEMRON personnel. A small, 100-gallon pickup bed fuel tank may be used to refuel equipment. Only minor equipment maintenance will occur on site. Significant equipment repairs will not be conducted on site unless equipment failure occurs and repairs must be completed on site. All equipment fluids generated from maintenance activities will be disposed of into designated drums and stored onsite until proper disposal is setup.

Installation Schedule: Equipment and vehicle maintenance and fueling practices will be implemented at the beginning of active construction on site.

Maintenance and Inspection: Fuel and or dispensing operations will be visually inspected daily for leaks and spills. Inspect equipment/vehicle storage areas, fueling points, and fuel tanks every 14 calendar days and within 24 hours after storm events of 0.5 inches or greater. Vehicles and equipment will be inspected each day of use. Leaks will be repaired as soon as possible, or the problem vehicle(s) or equipment will be removed from the project site. An ample supply of spill-cleanup materials will be kept on site to immediately clean up spills. Absorbent pads will be placed under any tool or vehicle being fueled to catch any incidental drips or spills from reaching the ground surface. All spill cleanup materials used will be disposed of properly.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

7.3.4 Material Handling and Waste Management

General Refuse/Waste Management

BMP Description: All general refuse/waste materials will be collected and disposed of into dumpsters located in the staging area. Dumpsters and other trash receptacles will be placed away from stormwater conveyances and drains, and meet all federal, state, and local solid waste management regulations. Only trash and construction debris from the site will be deposited in the dumpsters and other trash receptacles. All personnel will be instructed, during daily tailgate safety sessions, regarding the correct procedure for disposal of trash and construction debris. On-site management will be responsible for ensuring general refuse and construction wastes are managed appropriately.

Installation Schedule: Dumpsters and other trash receptacles will be installed once the staging area is established at the site.

Maintenance and Inspection: The dumpsters and trash receptacles will be inspected every 14 calendar days and within 24 hours after storm events of 0.5 inches or greater. The dumpsters and trash receptacles will be emptied as needed by a subcontractor in accordance with local, state, and federal regulations.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

Sanitary Waste

BMP Description: At least one (1) temporary sanitary facility (portable toilet) will be provided at the site in the staging area. Additional toilets may also be utilized as is deemed necessary by the UXB-KEMRON project manager. The toilet will be away from concentrated flow paths and traffic flow and will have collection pans underneath to contain waste and provide secondary containment.

Installation Schedule: The portable toilet(s) will be brought to the site once the staging area is established and prior to remedial actions.

Maintenance and Inspection: All sanitary waste will be collected from the portable toilet(s) a minimum of once per week by a local subcontractor. The toilet(s) will be inspected at least every 14 calendar days for evidence of leaking holding tanks. Toilets with leaking holding tanks will be removed from the site and replaced with new portable toilets.

Responsible Staff: UXB-KEMRON or Subcontractor(s).

8.0 WASTE TRANSPORTATION AND DISPOSAL PLAN

This Waste Transportation and Disposal Plan (WTDP) has been prepared by UXB-KEMRON to establish site procedures for handling, transportation, and disposal of waste materials generated from the interim measures actions at the SWMU 57 site. During active interim measures, a copy of the WTDP will be maintained on-site at all times, which may be supplemented as necessary as deemed appropriate by the UXB-KEMRON project manager as site conditions dictate.

8.1 Applicable Regulations and Plan Approval

The wastes generated during the interim measures will be handled in accordance with applicable Federal, State, and local regulations which included:

- 40 CFR 268: Land Disposal Restrictions
- 9 VAC 20-60-262 Adoption of 40 CFR Part 262 by reference

8.2 Anticipated Waste Streams

The wastes generated during the interim measures may include the following waste streams:

- Interim Measures Derived Wastes
- Secondary Wastes

8.2.1 Investigative Derived Materials

Any wastes which are generated by the remedial actions of the interim measures will be considered Investigative Derived Material (IDM). All IDM generated during the project will be properly contained and stored in a designated storage area, which is located near the Shipping and Receiving building. The generated wastes will be removed from the project site daily. The anticipated IDM will include the following waste streams:

- Impacted soils generated during pond excavation
- Purge water generated during groundwater sampling.
- Pond water generated during fluids removal of excavation area.
- Decontamination fluids

8.2.2 Secondary Wastes

Any wastes which are generated by the general activities of the project site during the interim measures will be considered Secondary Wastes. The anticipated Secondary Waste will include the following waste stream:

- Typical non-hazardous solid waste
- Personal Protective Equipment
- Soil and erosion control devices

8.3 Waste Management Procedures

8.3.1 Waste Minimization

Waste minimization practices will be implemented during the interim measures to control the amount of wastes generated and potential to segregate waste streams. The segregated waste streams will include contaminant impacted wastes and non-impacted secondary wastes. The impacted waste can further segregated into two waste streams which include hazardous and non-hazardous wastes. The waste streams will be characterized prior the generation in an effort to minimize the retention times of segregated wastes on the project site.

8.3.2 Onsite Waste Labeling

The following practices will be maintained to indentify and label each container and waste stream generated at the SWMU 57 project site. The information will be used for proper transport and disposal of the materials.

- Generation activity and location
- Type of material
- Comments or Special handling instructions
- Date

8.3.3 Sampling and Characterization

Each of the wastes streams generated during the interim measures at the SWMU 57 site will be sampled for disposal characterization either prior to generation or immediately following waste generation. Waste characterization sampling will follow the procedures specified in the QAPP, or as dictated by the disposal facility. Profiles of each waste stream will be submitted to offsite facilities for approval prior to the transportation of any wastes. Wastes will be transported off site for disposal within 90 days of generation.

8.3.4 Recordkeeping

UXB-KEMRON will maintain records of waste disposal on the project site and in the project file. The records maintained will include the following:

- Waste Characterization Sampling Results
- Signed and Approved Waste Profiles
- Signed Manifests and/or Bills of Lading

Although hazardous waste is not anticipated to be generated, if hazardous waste is encountered, hazardous waste manifests will be coordinated with the operating contractor for their approval, signature and recordkeeping.

8.3.5 Spill Response Materials

Spill response materials will be available in the event of a spill or release at the site. The following quantities of materials are based on a small scale spill, less than 50 gallons of liquid waste and 500 lbs of solid waste:

- Oil absorbed pads (one bale)
- Oil absorbent material (100 lbs.)
- Oil absorbent booms (40 feet)
- Front-end loader or excavator
- 55-gallon drums, DOT UN1A1 Un1A2 (2 total)
- Shovels
- Decontamination supplies and PPE
- Hand operated pump

8.4 Offsite Transportation and Disposal

8.4.1 Identification of Offsite Disposal Facility

UXB-KEMRON will contact waste disposal facilities to determine the most appropriate disposal facility. The factors for selection of the facility will include, but not necessarily be limited to:

- Current facility licensure
- Cost per unit rate of materials
- Transportation costs
- Disposal options (landfilling, incinerations, etc.)
- Types of wastes received

8.4.2 Transportation of Waste

UXB-KEMRON will inspect all containers and maintain the labeling and manifesting of waste has been properly conducted prior to any wastes be removed from the project site. All container loading and segregation of wastes will be conducted under the supervision of UXB-KEMRON employees.

8.5 Documentation and Reporting

8.5.1 Complete Manifest Package

UXB-KEMRON will provide manifest packages for all waste streams which are to be removed from the project site to be disposed of at an offsite facility. All wastes transported from the project site will be contained and labeled in accordance with the DOT requirements. UXB-KEMRON will also maintain a record of the manifest package onsite which will include the following:

- Waste Characterization Sampling Results
- Signed and Approved Waste Profiles
- Signed Manifests and/or Bills of Lading

The final disposal facility tickets will be included into the project file once they have been received by UXB-KEMRON.

8.5.2 Transportation and Disposal Reporting Requirements

UXB-KEMRON will maintain records of all wastes leaving the SWMU 57 site. These records will provide a clear trail of the waste generation and disposal operations. These records will include the following:

- Waste type
- Date generated
- Manifest package
- Date Transported offsite
- Transportation company and driver name
- Date of disposal
- Name of disposal facility

9.0 REFERENCES

Alliant Techsystems, Inc. (ATK), 2005. *Safety, Security and Environmental Rules for Contractors, Subcontractors, Tenants and Government Employees.* March 2005.

Intergovernmental Data Quality Task Force, 2005. *Uniform Federal Policy for Implementing Environmental Quality Systems; Evaluating, Assessing, and Documenting Environmental Data Collection/Use and Technology Program* (UFP QAPP), Final, Version 2. March 2005.

URS Corporation (URS), 2009. SWMU 57 *RCRA Facility Investigation/Corrective Measures Study Report.* Final. September 2009.

URS Corporation (URS), 2003. *Final Master Work Plan, Quality Assurance Plan, Health and Safety Plan*. Radford Army Ammunition Plant, Radford, Virginia. Prepared for the U.S. Army Corps of Engineers, Baltimore District. August 2003.

U.S. Army Corps of Engineers (USACE), 2001. *EM200-1-3, Requirements for the Preparation of Sampling and Analysis Plans*.

U.S. Environmental Protection Agency (USEPA), 2000a. *Permit for Corrective Action and Waste Minimization*: Pursuant to the Resource Conservation and Recovery Act as Amended by the Hazardous and Solid Waste Amendment of 1984, Radford Army Ammunition Plant, Radford, Virginia. VA1210020730.

Virginia Department of Conservation and Recreation, Division of Soil and Water Conservations, 1992. *Virginia Erosion and Sediment Control Handbook.* Third Edition, 1992.

FIGURES

0 325 650 1,300
Feet

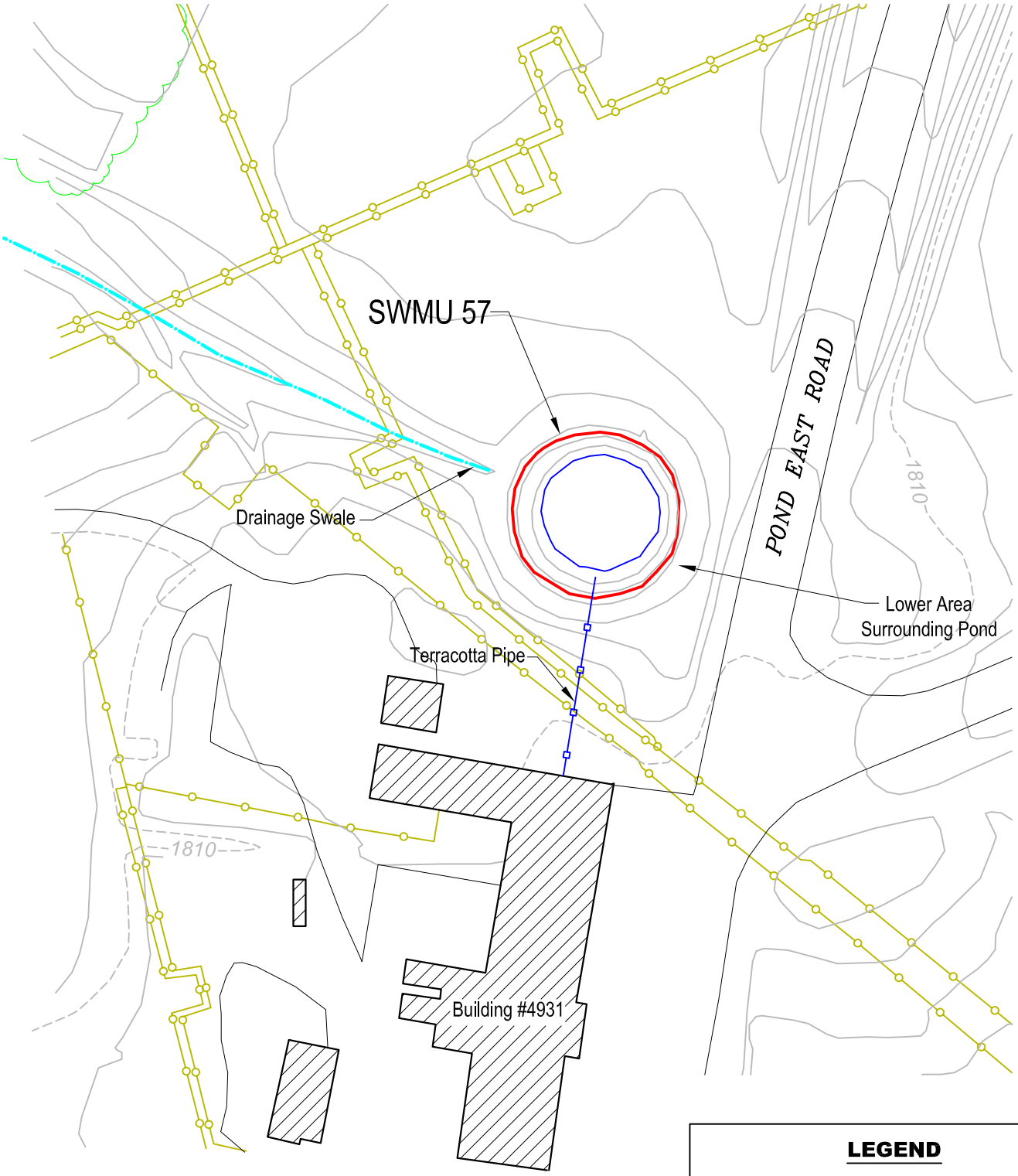
1 inch equals 1,300 feet

N

Source: Google Earth; Imagery Date: February 1, 2007

KEMRON ENVIRONMENTAL SERVICES
KEMRON Environmental Services
1359-A Ellsworth Industrial Blvd.
Atlanta, GA 30318

PROJECT NO. RADFORD	DRAWING DATE: 11/04/2010
DESIGNED DRP	SITE LOCATION MAP
DETAILED DRP	SWMU 57 IMWP
CHECKED KR	LOCATION: RADFORD ARMY AMMUNITION PLANT, RADFORD, VIRGINIA
	FIGURE: 1



LEGEND

- BUILDINGS
- APPROXIMATE SWMU BOUNDARY
- TERRACOTTA PIPE
- VEGETATION
- ABOVEGROUND PIPING
- TOPOGRAPHIC CONTOUR
- DRAINAGE SWALE

Note:
Base map developed from fig. 10-2.
Provided by URS Group Inc., 4-2008

KEMRON ENVIRONMENTAL SERVICES

0 25 50
SCALE 1"=50'
(Approximate)

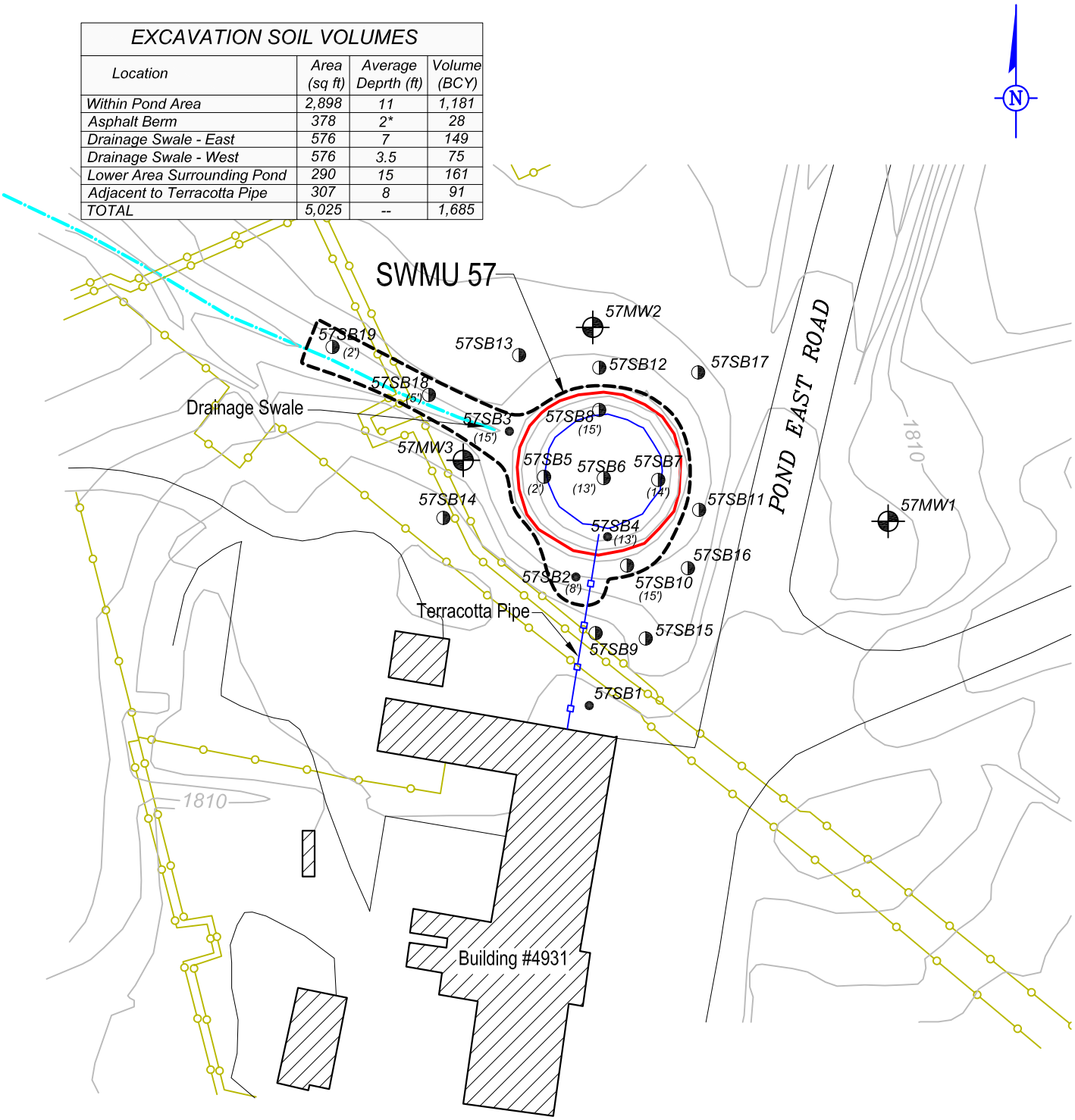
DRAWN BY: KG		DATE 23 AUGUST 2010
REVIEWED: KR	R 4	PROJECT NO. MR0669-400-001
APPROVED:		DWG. FILE NO. SWMU 57.SITE LAYT.

FIGURE 2

IMWP SWMU 57
SITE LAYOUT
RADFORD ARMY AMMUNITION PLANT-022
RADFORD, VIRGINIA

EXCAVATION SOIL VOLUMES

Location	Area (sq ft)	Average Deprth (ft)	Volume (BCY)
Within Pond Area	2,898	11	1,181
Asphalt Berm	378	2*	28
Drainage Swale - East	576	7	149
Drainage Swale - West	576	3.5	75
Lower Area Surrounding Pond	290	15	161
Adjacent to Terracotta Pipe	307	8	91
TOTAL	5,025	--	1,685



LEGEND

- 57MW3 — MONITORING WELL LOCATION
- 57SB14 — RFI SAMPLE LOCATION
- 57SB1 — SSP SOIL SAMPLE LOCATION
- APPROXIMATE SWMU BOUNDARY
- EXCAVATION AREA
- (2') — APPROXIMATE DEPTH OF EXCAVATION
- BUILDINGS
- TERRACOTTA PIPE
- ABOVEGROUND PIPING
- VEGETATION
- TOPOGRAPHIC CONTOUR
- DRAINAGE SWALE

Note:
Base map developed from fig. 10-2.
Provided by URS Group Inc., 4-2008

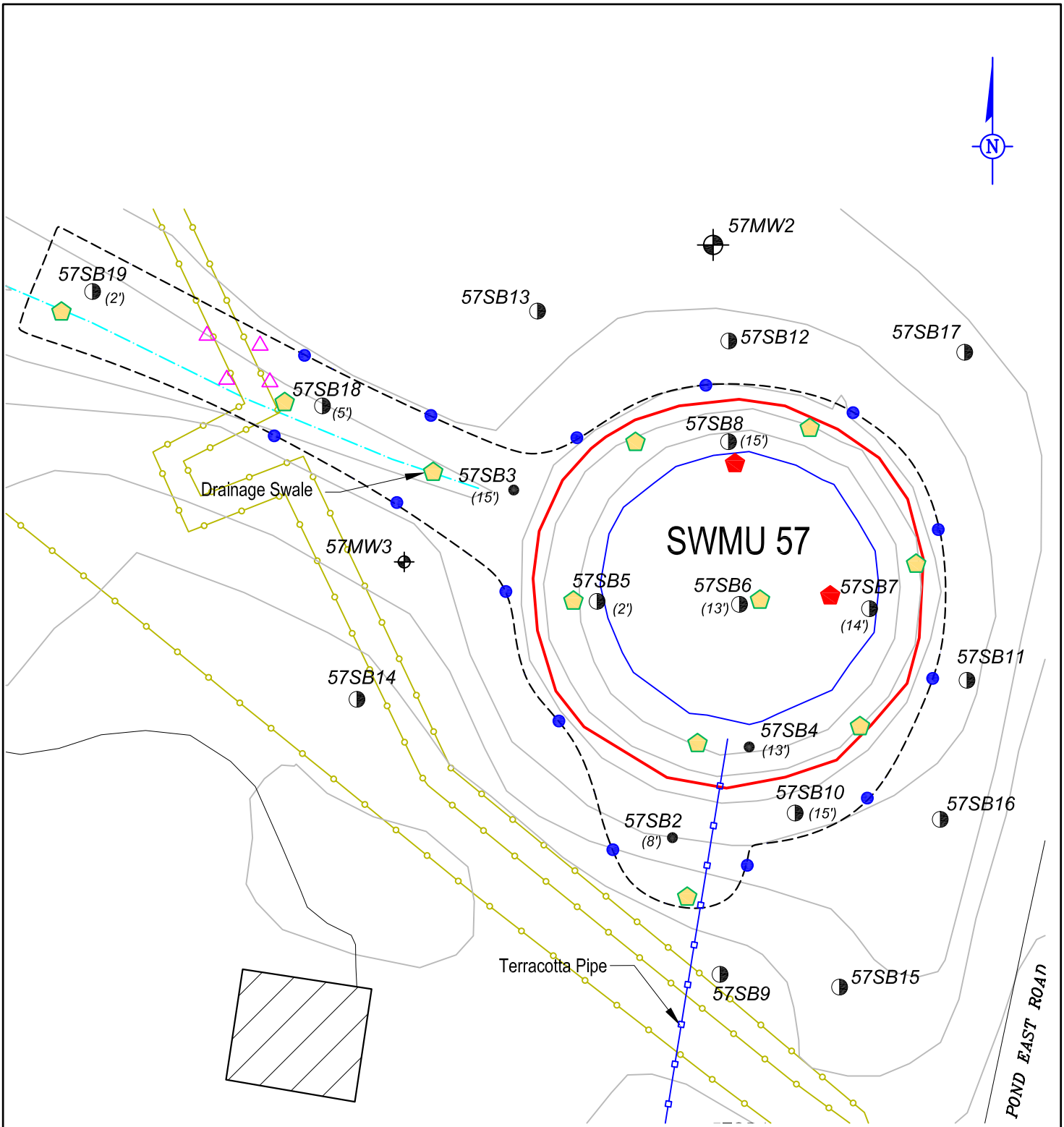
KEMRON ENVIRONMENTAL SERVICES

0 25 50
SCALE 1"=50'
(Approximate)

DRAWN BY: KG		DATE 23 AUGUST 2010	
REVIEWED: KR	R 4	PROJECT NO. MR0669-400-001	
APPROVED:		DWG. FILE NO. ESTIMATED EX. AREA	

FIGURE 3

SWMU 57 IMWP
ESTIMATED EXCAVATION AREA MAP
RADFORD ARMY AMMUNITION PLANT-022
RADFORD, VIRGINIA



LEGEND

- SIDEWALL CONFIRMATION SAMPLE(18)
- WASTE CHARACTERIZATION SAMPLE (Surface & Subsurface)
- FLOOR CONFIRMATION SAMPLE(7)
- HAND AUGER BORING(9)
- 57MW3 — MONITORING WELL LOCATION
- 57SB14 — RFI SAMPLE LOCATION
- 57SB1 — SSP SOIL SAMPLE LOCATION
- BUILDINGS
- TERRACOTTA PIPE
- APPROXIMATE SWMU BOUNDARY
- EXCAVATION AREA
- (2') — APPROXIMATE DEPTH OF EXCAVATION
- ABOVEGROUND PIPING
- TOPOGRAPHIC CONTOUR
- DRAINAGE SWALE

Note:
Base map developed from fig. 10-2.
Provided by URS Group Inc., 4-2008

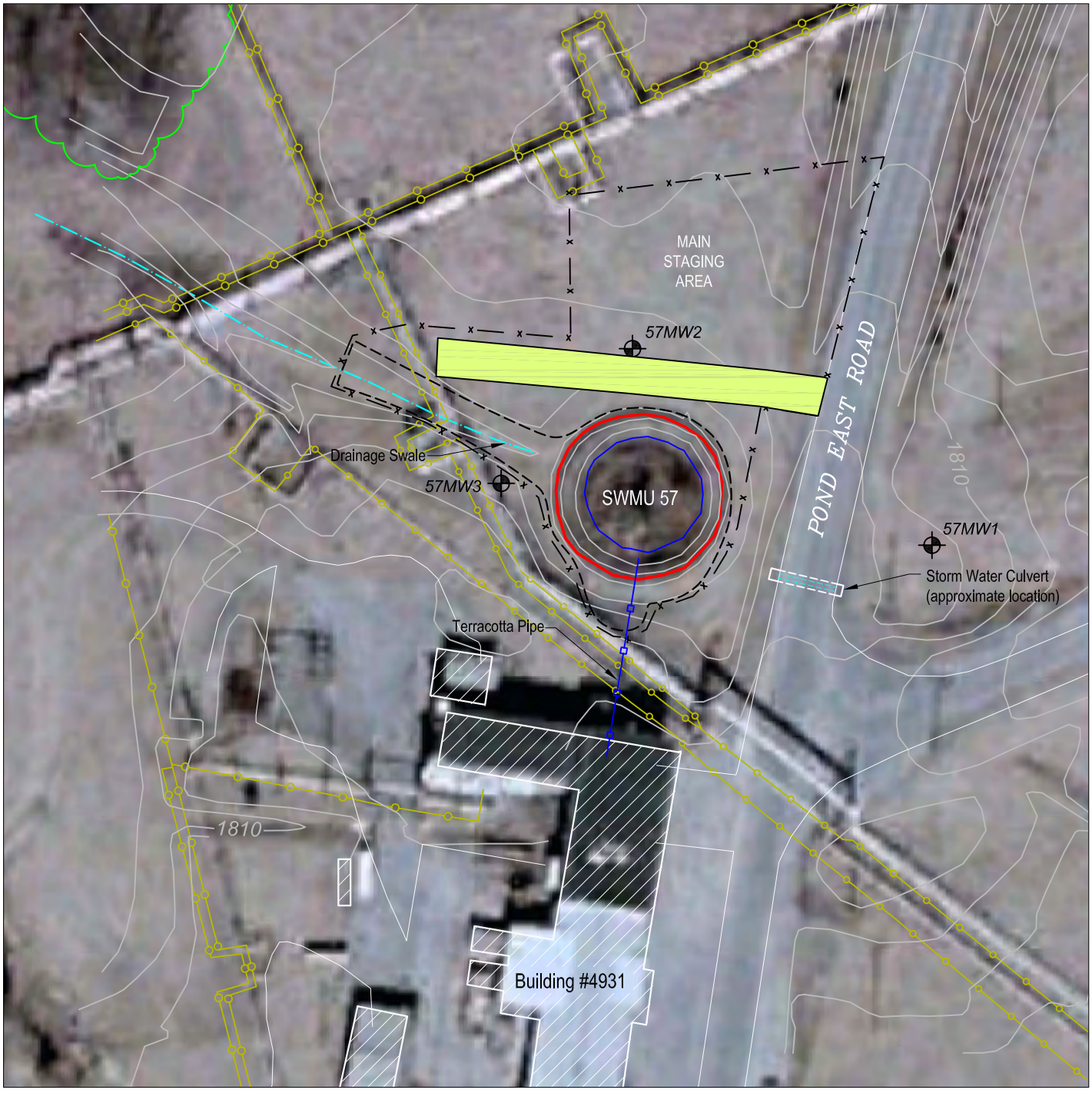
KEMRON ENVIRONMENTAL SERVICES

0 10 20
SCALE 1"=20'
(Approximate)

DRAWN BY: KG		DATE 23 AUGUST 2010	
REVIEWED: KR	R 5	PROJECT NO. MR0669-400-001	
APPROVED:		DWG. FILE NO. PRP. SAMPLING PLAN	

FIGURE 4

IMWP SWMU 57
PROPOSED SAMPLING PLAN
RADFORD ARMY AMMUNITION PLANT-022
RADFORD, VIRGINIA



LEGEND

- BUILDINGS
- TERRACOTTA PIPE
- CONSTRUCTION ENTERANCE/EXIT
- SILT FENCE
- 57MW3 MONITORING WELL LOCATION
- VEGETATION
- APPROXIMATE SWMU BOUNDARY
- EXCAVATION AREA
- ABOVEGROUND PIPING
- TOPOGRAPHIC CONTOUR
- DRAINAGE SWALE

N

Note:
Areal Photo from
Google Earth, 2010

KEMRON ENVIRONMENTAL SERVICES

0 25 50
SCALE 1"=50'
(Approximate)

DRAWN BY: KG		DATE 15 SEPTEMBER 2010	FIGURE 5
REVIEWED: KR	R 5	PROJECT NO. MR0669-400-001	
APPROVED:		DWG. FILE NO. PRP. STAGING AREA	

IMWP SWMU 57
PROPOSED STAGING AREA
RADFORD ARMY AMMUNITION
PLANT-022
RADFORD, VIRGINIA

TABLES

Table 1
Interim Measure Specifications

Process / Task	Unit	Quantity	Specifications	Additional Notes
Site Preparation				
Construction entrance/exit and staging area	CY	60	VDOT #1 coarse aggregate (2- to 3-inch stone) or equivalent	3 inch thick
Liner at loading zone	LF	50	5 millimeter polyethylene plastic sheeting	
Silt fence	LF	600	To comply with Virginia Erosion and Sediment Control Std and Spec 3.05	
Vacuum truck	EA	1	2,500-gallon capacity	
Solid Waste Dumpster	EA	1	10 yard dumpster	
Portable Toilet	EA	1		
Excavation				
Excavator	EA	1	320 caterpillar excavator	
Dozer	EA	1	D-5	
Dump Trucks	EA	2	18 yard dump truck	
Temporary wooden supports	EA	8	wooden 2x4 supports with horizontal bracing. Exact quantity will be determined in field	exact number will be based on hand auger soil results
Backfill	CY	1,685	soil will classify as a CL, CH, or SC in the USCS Classification System	compacted
Seed and straw	TBD	TBD		estimated for 5,025 square foot area.
Monitoring Well Abandonment				
Track mounted drill rig	EA	1	capable of hollow-stem auger and air rotary drilling techniques	
Drums for IDW	EA	50	55-gallon	
Decontamination				
Pressure washer	EA	1		
Alconox powder	LB	1		

Notes:

When brands and model numbers are specified, equivalent equipment may be substituted.

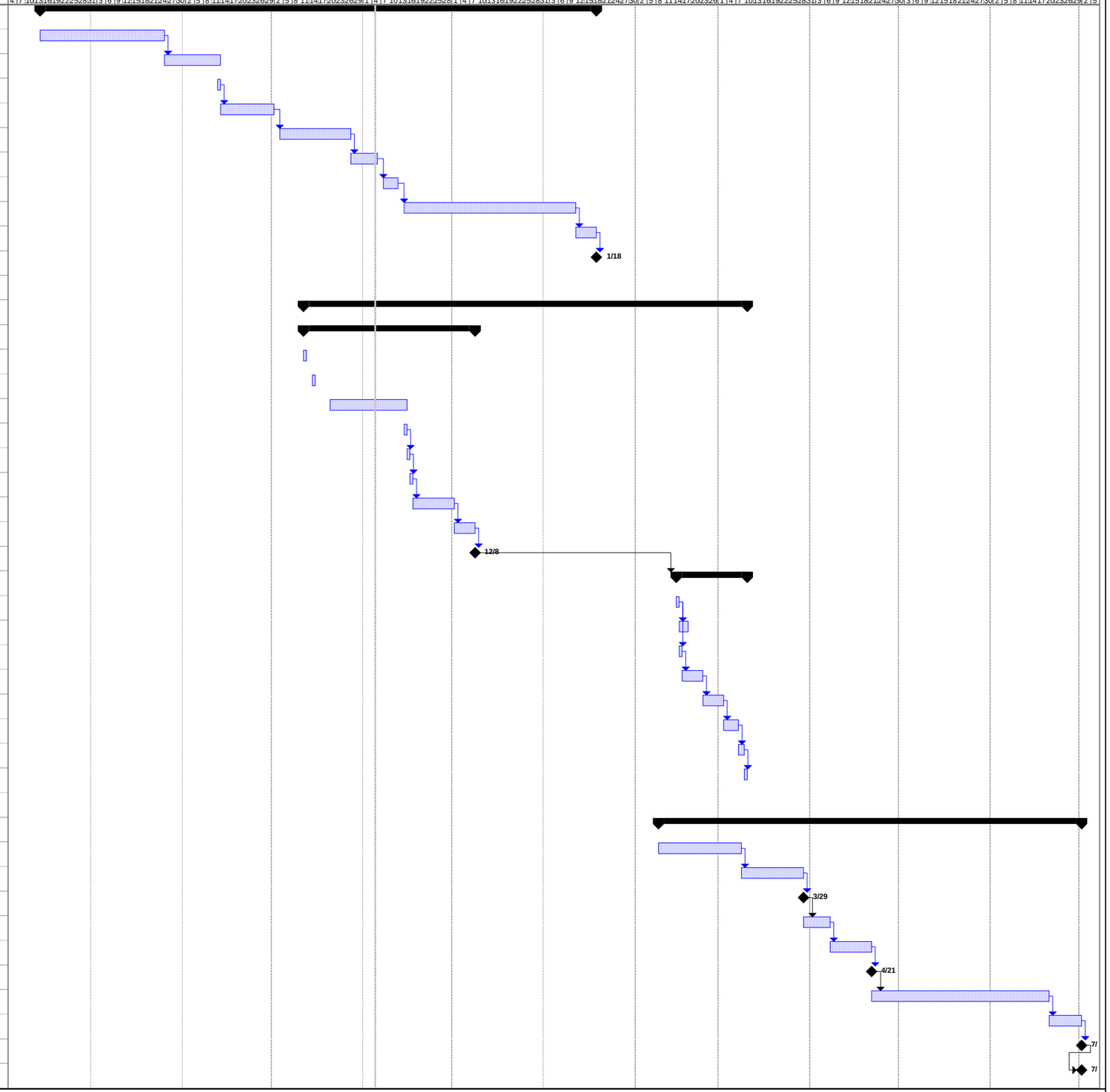
Excavation and backfill volumes estimated assuming excavation will extend to 15 feet bgs and estimated horizontal extent shown in RFI. These values are subject to change based on confirmation sampling results.

CY = cubic yard
EA = each
LB = pound
LF = linear feet
SF = square feet

APPENDIX A

Project Schedule

ID	Task Name	Duration	Start	Finish	Predecessors
1	**(Task 6) Interim Measures Implementation Work Plan (IMI Work Plan) - RAAP-022: Pond by I**	**134 days**	**Thu 7/15/10**	**Tue 1/18/11**	
2	Preparation of Army Draft IMI Work Plan	30 days	Thu 7/15/10	Wed 8/25/10	
3	Army Review	13 days	Thu 8/26/10	Mon 9/13/10	2
4	Army Acceptance of Army Draft IMI Work Plan	1 day	Mon 9/13/10	Mon 9/13/10	
5	Incorporate Army Comments in Stakeholder Draft IMI Work Plan	14 days	Tue 9/14/10	Fri 10/1/10	4
6	Army Review of Stakeholder Draft IMI Work Plan	18 days	Mon 10/4/10	Wed 10/27/10	5
7	Incorporate Army Comments in Stakeholder Draft IMI Work Plan	7 days	Thu 10/28/10	Fri 11/5/10	6
8	Army Second Review of Stakeholder Draft IMI Work Plan, Approved by Army	5 days	Mon 11/8/10	Fri 11/12/10	7
9	Regulator Review	42 days	Mon 11/15/10	Tue 1/11/11	8
10	Incorporate Regulator Comments into Final IMI Work Plan	5 days	Wed 1/12/11	Tue 1/18/11	9
11	Final IMI Work Plan Approved by Army and Regulator	0 days	Tue 1/18/11	Tue 1/18/11	10
12					
13	**(Task 7) Interim Measures Implementation - RAAP-022: Pond by Buildings 4931 and 4928**	**108 days**	**Tue 10/12/10**	**Thu 3/10/11**	
14	**Pre-excavation Sampling**	**42 days**	**Tue 10/12/10**	**Wed 12/8/10**	
15	Notify USEPA of Intent to Conduct Confirmation Groundwater Sampling	1 day	Tue 10/12/10	Tue 10/12/10	
16	USEPA Approval to Conduct Confirmation Groundwater Sampling	1 day	Fri 10/15/10	Fri 10/15/10	
17	Pre-Work Meeting at RFAAP, badging, permitting	18 days	Thu 10/21/10	Mon 11/15/10	
18	Mobilization of personnel and equipment	1 day	Mon 11/15/10	Mon 11/15/10	
19	Conduct sampling for confirmation groundwater	1 day	Tue 11/16/10	Tue 11/16/10	18
20	Demobilize personnel and equipment	1 day	Wed 11/17/10	Wed 11/17/10	19
21	Analytical analysis of samples/data review	10 days	Thu 11/18/10	Wed 12/1/10	20
22	Prepare results and information meeting with RFAAP and Regulator	5 days	Thu 12/2/10	Wed 12/8/10	21
23	Army and Regulator review data and agreement on clean closure decision	0 days	Wed 12/8/10	Wed 12/8/10	22
24	**Implementation of IMWP**	**18 days**	**Tue 2/15/11**	**Thu 3/10/11**	**23**
25	Mobilization of personnel and equipment	1 day	Tue 2/15/11	Tue 2/15/11	
26	Abandon Wells and collect waste characterization samples	3 days	Wed 2/16/11	Fri 2/18/11	25
27	Establish erosion controls	1 day	Wed 2/16/11	Wed 2/16/11	25
28	Excavate pond and drainage swale, confirmation sampling	5 days	Thu 2/17/11	Wed 2/23/11	27
29	Analytical analysis of confirmation samples	5 days	Thu 2/24/11	Wed 3/2/11	28
30	Backfill placement	3 days	Thu 3/3/11	Mon 3/7/11	29
31	Restore Site	2 days	Tue 3/8/11	Wed 3/9/11	30
32	Demobilize personnel and equipment	1 day	Thu 3/10/11	Thu 3/10/11	31
33					
34	**(Task 8) Interim Measures Implementation Report (IMI Report) - RAAP-022: Pond by Buildin**	**103 days**	**Wed 2/9/11**	**Fri 7/1/11**	
35	Preparation of Army Draft IMI Report	20 days	Wed 2/9/11	Tue 3/8/11	
36	Army Review	15 days	Wed 3/9/11	Tue 3/29/11	35
37	Army Acceptance of Army Draft IMI Report	0 days	Tue 3/29/11	Tue 3/29/11	36
38	Incorporate Army Comments in Stakeholder Draft IMI Report	7 days	Wed 3/30/11	Thu 4/7/11	37
39	Army Review of Stakeholder Draft IMI Report	10 days	Fri 4/8/11	Thu 4/21/11	38
40	Stakeholder Draft IMI Report Approved by Army	0 days	Thu 4/21/11	Thu 4/21/11	39
41	Regulator Review	42 days	Fri 4/22/11	Mon 6/20/11	40
42	Incorporate Regulator Comments into Final IMI Report	9 days	Tue 6/21/11	Fri 7/1/11	41
43	Final IMI Report Approved by Army and Regulator	0 days	Fri 7/1/11	Fri 7/1/11	42
44	Site Closure Performance Objective Achieved	0 days	Fri 7/1/11	Fri 7/1/11	43



Project: Project1
Date: Fri 11/5/10

Task | Split | Progress | Milestone | Summary | Project Summary | External Tasks | External Milestone | Deadline

APPENDIX B

Quality Assurance Project Plan

UXB-KEMRON Remediation Services, LLC
2020 Kraft Drive, Suite 2100 • Blacksburg, Virginia 24060
Tel: 540.443.3700 Fax: 540.443.3790

October 08, 2010

Mr. Thomas P. Meyer
Baltimore District, US Army Corp of Engineers
Contracting Officer's Representative
HTRW Military
10 South Howard Street, Room 7000
Baltimore, MD 21201

RE: Draft Final SWMU 40, RAAP-009 and Draft SWMU 57, RAAP-022: Quality Assurance Project Plans, Radford Army Ammunition Plant, Virginia, October 2010

Dear Mr. Meyer:

UXB-KEMRON is pleased to submit the Draft Final Quality Assurance Project Plans (QAPPs), Appendix B to the SWMU 40, RAAP-009 Draft Final Interim Measures Work Plan and SWMU 57, RAAP-022 Draft Final Interim Measures Work Plan. Each Draft Final QAPP has been prepared in accordance with the UXB-KEMRON JV Performance Based Acquisition, Radford Army Ammunition Plant (RFAAP), Virginia.

Based on recent discussion regarding conduct of reviews, UXB-KEMRON has inserted summary comments received from the Army and the UXB-KEMRON response directly into the document using the "insert comment" function in Review mode of Word. The original comments with summary responses also is provided as stand alone document to verify that each comment received has been incorporated into the revised documents. We trust that this format will meet with reviewers' needs, and will best facilitate review of our responses to the Army's comments.

The Draft Final QAPP, Appendix B for each SWMU IMWP, is also being posted to the RFAAP Sharepoint site in editable form. Paper copies are not being issued based on our provision of the documents in editable form. If you wish to receive paper copies, please let us know and we will send them in overnight mail.

Please note that appropriate UXB-KEMRON signatures will be provided on the QAPP signature page following completion of the Army review and comment process on the QAPP.

Please contact me at mrochotte@kemron.com, or by phone at (740) 373-1266, if you have any questions regarding this contract submittal.

Sincerely,
KEMRON Environmental Services, Inc.

Mary Lou Rochotte, C.P.G.
Project Manager

Enclosure

cc: Rich Mendoza, USAEC ERM
Jim McKenna, RFAAP
Jerome Redder, RFAAP
Rich Dugger, UXB-KEMRON Program Manager
Tracy Bergquist, UXB-KEMRON Technical Manager
File

APPENDIX B

RADFORD ARMY AMMUNITION PLANT
RADFORD, VIRGINIA

Performance Based Acquisition
Solid Waste Management Unit 57 (RAAP-022)
Pond by Buildings 4931 & 4928
Quality Assurance Project Plan

DRAFT FINAL
NOVEMBER 2010
REVISION 1

PREPARED BY:

UXB-KEMRON Remediation Services, LLC
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Contract No. W912DY-10-D-0027
Delivery Order Number: DA01

PERFORMANCE BASED ACQUISITION
QUALITY ASSURANCE PROJECT PLAN
TABLE OF CONTENTS

Section ***Page***

PERFORMANCE BASED ACQUISITION
QUALITY ASSURANCE PROJECT PLAN
TABLE OF CONTENTS
(CONTINUED)

LIST OF TABLES

PERFORMANCE BASED ACQUISITION
QUALITY ASSURANCE PROJECT PLAN
TABLE OF FIGURES
(CONTINUED)

LIST OF FIGURES

PERFORMANCE BASED ACQUISITION
QUALITY ASSURANCE PROJECT PLAN
TABLE OF CONTENTS
(CONTINUED)

LIST OF ATTACHMENTS

PERFORMANCE BASED ACQUISITION
QUALITY ASSURANCE PROJECT PLAN
TABLE OF CONTENTS
(CONTINUED)

LIST OF ABBREVIATIONS AND ACRONYMS

AES	Auger Electron Spectrometry
ASTM	American Society for Testing and Materials
bgs	Below Ground Surface
BTEX	Benzene, Toluene, Ethylbenzene, and Xylene
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
CMO	Corrective Measure Objective
COC	Contaminants of Concern
COE	Corps of Engineers
COPC	Constituent of Potential Concern
COR	Contracting Officer Representative
DODELAP	Department of Defense Environmental Laboratory Accreditation
DQI	Data Quality Indicators
DQO	Data Quality Objective
FS	Field Supervisor
GC	Gas Chromatography
H&S	Health and Safety
APP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HPLC	High Pressure Liquid Chromatography
IDW	Investigative-Derived Waste
IMWP	Interim Measures Work Plan
KEMRON	KEMRON Environmental Services, Inc.
kg	Kilogram
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LQAP	Laboratory Quality Manual
LTM	Long-Term Monitoring
LOD	Limit of Detection
LOQ	Limit of Quantitation
MB	Method Blank
MCL	Maximum Contaminant Level
Mg	Milligram
mg/kg	Milligrams per Kilogram
MMRP	Military Munitions Response Program
MPC	Maximum Permissible Concentrations
MRS	Munitions Response Site
MS	Mass Spectrometry
MSR	Management Systems Review
MTBE	Methyl Tertiary Butyl Ether
MW	Monitoring Well
N/A	Not Applicable
NELAP	National Environmental Laboratory Accreditation Program
OSHA	Occupational Safety and Health Administration

OU	Operable Unit
OVA	Organic Vapor Analyzer
OWSER	Office of Solid Waste and Emergency Response (US EPA)
PAH	Polyaromatic Hydrocarbon
PBA	Performance Based Acquisition
PCB	Polychlorinated Biphenyls
PDF	Portable Document Format (Adobe Acrobat)
PE	Professional Engineer
PG	Professional Geologist
PM	Project Manager
PRG	Preliminary Remediation Goal
PQOs	Project Quality Objectives
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
RA	Remedial Action
RFAAP	Radford Army Ammunition Plant
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SOP	Standard Operating Procedure
SSL	Soil Screening Levels
SVOC	Semi Volatile Organic Compounds
TAL	Target Analyte List
TBD	To be determined
TCLP	Total Characteristic Leaching Procedure
TO	Task Order
TOC	Total Organic Carbons
UFP	Uniform Federal Policy
USACE	US Army Corps of Engineers
USAEC	US Army Environmental Command
USEPA	United States Environmental Protection Agency
UXB	UXB International, Inc.
UXB-KEMRON	UXB-KEMRON Remediation Services, LLC
UXO	Unexploded Ordnance
VDEQ	Virginia Department of Environmental Quality
VOC	Volatile Organic Compounds

SECTION 1. INTRODUCTION

The Quality Assurance Project Plan (QAPP) establishes function-specific responsibilities and authorities for data quality and defines procedures that will ensure that site investigative activities will result in the generation of reliable data. Inherent in the quality assurance program (QA) is the implementation of quality control (QC) measures. These measures provide assurance that the monitoring of quality-related events has occurred, and that the data gathered in support of the project are complete, accurate, and precise. The implementation of the QAPP will help ensure the validity of the data collected and will establish a firm foundation for decisions regarding fieldwork performed at Radford Army Ammunition Plant (RFAAP) by UXB-KEMRON under TO # DA01 of Worldwide Environmental Remediation Services contract, number W912DY-10-D-0027.

This QAPP is developed as an addendum to the *Final Master Work Plan, Radford Army Ammunition Plant, Radford, Virginia* (URS, 2003), which has been reviewed and approved by USEPA and VDEQ. This Addendum provide information to supplement existing documentation and is specific to work that will be conducted by UXB-KEMRON at two SWMUs, SWMU 40, RAAP-009 and SWMU 57, RAAP-022.

1.1 Project Objectives

Project objectives are specified in Section 3 of the Interim Measures Work Plan (IMWP), and are taken from the USEPA and VDEQ approved Final RFI/CMS for this site.

1.2 QAPP Distribution List

The following provides a distribution list for this document.

Thomas P. Meyer, USACE Baltimore District, COR
Richard R. Mendoza, USAEC Environmental Restoration Manager
James J. McKenna, RFAAP Environmental Engineer
Jerome Redder, ATK
Will Geiger, USEPA, Region III
Jim Cutler, VDEQ
Richmond H. Dugger, IV, UXB-KEMRON Program Manager
Mary Lou Rochotte, UXB-KEMRON Project Manager
Jeanette Hamm, UXB-KEMRON Project Quality Assurance Officer
Stephanie Mossburg, Project Manager, Microbac Ohio Valley Division
Jeanne Peterson, Senior Chemist, Analytical Quality Associates (AQA)

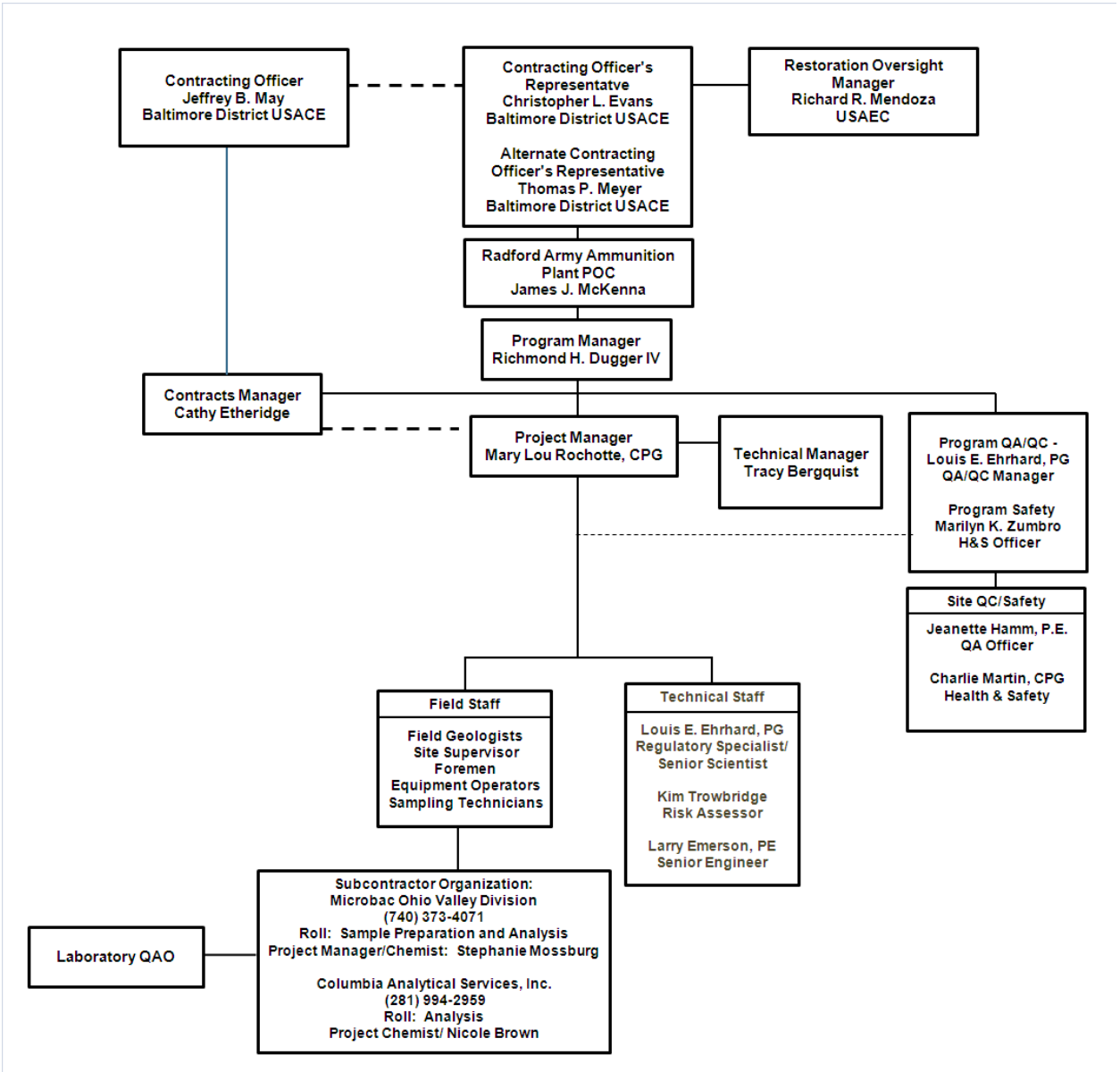
SECTION 2. PROJECT ORGANIZATION AND RESPONSIBILITIES

The UXB-KEMRON project organization strategy consists of a streamlined approach for effective and efficient execution of project activities. The overall project organizational chart can be found in Section 2.1 of the IMWP. Figure 2.1 below illustrates the flow of communication with the laboratory as well. Responsibilities for the implementation of the project QA/QC contractual obligation lie principally with the project staff and subcontractors. The Project Manager (PM) and QA Officer jointly have the primary responsibility to ensure the reliability and the validity of project activities and deliverables in compliance with the project QA program.

2.1 Key Project Personnel

Contractor personnel responsible for implementing technical, quality, and health and safety programs are described in the following subsections, and illustrated in Figure 2.1.

FIGURE 2.1



2.1.1 Contracting Officer Representative

The Contracting Officer Representative (COR) is the main point of contact between the contractor and the U.S. Army Corps of Engineers (USACE). Responsibilities of the COR include:

- Communicating frequently with the contracting project manager (PM) with regard to daily progress of the project;
- Evaluating progress
- Interfacing with regulatory agencies;
- Acting as liaison between USACE, RFAP, and the designated contractor; and
- Requiring completion of corrective actions, when indicated.

2.1.2 UXB-KEMRON Program Manager

The Program Manager is responsible for ensuring that the contract is adhered to throughout project performance. The Program Manager, assisted by the Project Manager, serves as the primary liaison with the COR on issues relating to contract performance. The Program Manager evaluates progress and provides guidance and resources to the Project Manager. The Program Manager is a corporate officer with direct authority to interface with regulatory agencies as needed.

2.1.3 UXB-KEMRON Project Manager

The PM is responsible for ensuring that activities are conducted in accordance with contractual specifications, the Statement of Work (SOW), and approved work plans. The PM will also provide technical coordination with the designated counterpart of the Installation. The PM is responsible for management of operations conducted for this project. In addition, the PM will ensure that personnel assigned to the project, including subcontractors, will review the technical plans prior to each task associated with the project is initiated. The PM will monitor the project budget and schedule and will ensure availability of necessary personnel, equipment, subcontractors, and services. The PM will participate in the development of the field program, evaluation of data, reporting, and the development of conclusions and recommendations.

2.1.4 UXB-KEMRON Quality Assurance Officer

The QA Officer is responsible for ensuring that the QA procedures and objectives in the project-specific work plans are met, reviewing field and analytical data to ensure adherence to QA/QC procedures, and approving the quality of data prior to inclusion in associated reports. This may include the performance of field and laboratory audits during the investigation. In addition, the QA Officer will be responsible for the review, evaluation, and validation of analytical data for the project and will participate in interpreting and presenting analytical data. QC coordination is under the technical guidance of the QA Officer to direct the task leaders on a day-to-day or as-needed basis to ensure the application of QA/QC procedures.

2.1.5 Risk Assessor

Risk Assessment comprises Human Health and Ecological Assessments. The Human Health and Ecological Risk Assessments for this site have been completed and approved by USEPA and VDEQ within the Final RFI/CMS (URS, 2009). Therefore, no further risk assessment work is anticipated.

2.1.6 UXB-KEMRON Field Supervisor

The Field Supervisor will provide management of the field activities during the field work and will report daily progress and concerns to the PM. The Field Supervisor is responsible for ensuring that technical matters pertaining to the field program are addressed. He/she will participate in data interpretation, report writing, and preparation of deliverables, and will ensure that work is being conducted as specified in the technical plans. In addition, the Field Supervisor is responsible for ensuring all field personnel conform to QA/QC procedures, and for safety-related issues. Prior to initiation of field activities, the Field Supervisor will conduct a field staff orientation and briefing to acquaint project personnel with the sites and assign field responsibilities.

2.1.7 Project Chemist

The Project Chemist is an employee of the subcontracted analytical laboratory, and is responsible for knowing and monitoring the laboratory's compliance with the elements of the SOW issued to the laboratory by the contractor, and for day-to-day communications with the Analytical Laboratory. The Project Chemist is the first contact with the laboratory for receiving confirmation of sample delivery, receipt, and analysis; for tracking sample analysis within the required holding times and within the period of performance specified in the SOW; and for the receipt of data deliverables.

2.1.8 Laboratory Quality Manager

The Laboratory Quality Manager will be responsible for the technical quality of the laboratory and adherence to the Laboratory QA Program. The contract laboratory will analyze environmental samples for parameters of interest with complete data documentation. The contractor QA Officer will monitor laboratory activities.

2.1.9 Project Data Coordinator

A UXB-KEMRON Project Data Coordinator (PDC) will be responsible for receipt of field and laboratory data using a computerized data management system or database management system (DBMS). The PDC is responsible for the accuracy of data entered into the data management system. The PDC will also be responsible for providing the PM periodic data management summaries, ensuring all project chemical data are received from the laboratory in the Army's ERIS electronic data deliverable (EDD) format, and uploading the data into ERIS as required by Army standards.

2.1.10 Contract Specialist

The Contract Specialist is responsible for tracking funds for labor and materials procurement. Responsibilities include:

- Oversight of the financial status of the project;

- Preparation of monthly cost reports and invoices;
- Administration of equipment rental, material purchases, and inventory of supplies;
- Administration and negotiation of subcontracts and interaction with the Administration Contracting Officer and Procurement Contracting Officer on contract and subcontract issues;
- Preparation of project manpower estimates; and
- Administration of contract documents.

2.1.11 Health and Safety Officer

The Health and Safety Manager will review and internally approve the Accident Prevention Plan (APP), which includes a Site Specific Health and Safety Plan (SSAPP. In consultation with the PM, the Health and Safety Officer will ensure that an adequate level of personal protection exists for anticipated potential hazards for field personnel. On-site health and safety will the responsibility of the on-site Health & Safety Officer who will work in coordination with the PM and the project Health and Safety Officer.

2.2 Key Points of Contact

Table 2-1 provides the names and points of contact for UXB-KEMRON personnel and subcontractors.

Table 2-1
Contractor and Subcontractor Key Points of Contact
Radford Army Ammunition Plant, Radford, VA

Contractor	Title	Email	Key Point of Contact
Mary Lou Rochotte, CPG	Project Manager	@kemron.com	UXB-KEMRON 156 Starlite Drive Marietta, OH 45750 T (740) 373-1266 F (740) 376-2536
Jeanette Hamm	Project QA Officer	@kemron.com	UXB-KEMRON 1359-A Ellsworth Industrial Boulevard Atlanta, GA 30318 T (404) 636-0928 F (404) 636-7162
Charlie Martin	Project H&S Officer	@kemron.com	UXB-KEMRON 156 Starlite Drive Marietta, OH 45750 T (740) 373-1024 F (740) 376-2536
Subcontractor	**Title**	**Email**	**Key Point of Contact**
Stephanie Mossburg	Laboratory Project Manager	.Mossburg@microbac.com	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 T (740) 373-4071
David Bumgarner	Laboratory QA Officer	.Bumgarner@microbac.com	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 T (740) 373-4071

SECTION 3. PROBLEM DEFINITION AND PROJECT QUALITY OBJECTIVES

At SWMU 57, RAAP-022, historic practices in an adjacent building led to release of contaminants into a treatment unit that consists of a small pond adjacent to Buildings 4931 and 4938. Corrective Measures are being implemented as Interim Measures at the site to prevent potential future human exposure to impacted soils at the site. The contaminants of concern in soil are limited to metals. While limited groundwater sampling is required at the site, USEPA and VDEQ have approved a Final RFI/CMS for the site which concludes that no groundwater Corrective Measures are required.

The Final RFI/CMS for the site defines the Corrective Measures Objectives (CMOs) applicable to the site. The data derived and evaluated through implementation of the Interim Measures, including this QAPP, will be used to demonstrate achievement of the CMOs. The following CMO was developed for SWMU 57 based on the results of the site, risk, and fate and transport assessments and the most likely future land use at the site (industrial):

- Mitigate the potential risks/hazards that have been identified for evaluated future hypothetical industrial receptors for exposure to soil (construction workers) at the site.

The residential exposure pathway also was evaluated in the Final RFI/CMS to assess the remedial effort that would be required to achieve clean closure at SWMU 57 with unrestricted future land use without controls or long-term monitoring (LTM) requirements. The clean closure alternative was selected for the site.

All existing data in the Final RFI/CMS are fully useable and can be relied upon for decision making in this project. All new decision making data will be based upon sampling conducted by UXB-KEMRON on a schedule specified in the IMWP. Groundwater monitoring requirements are as specified in the Final RFI/CMS, Soil sampling at this site will be necessary for waste characterization purposes, and to confirm that Remedial Goals (RGs) for soil have been achieved at the site such that clean closure can be completed. Groundwater sampling data and soil confirmatory sampling data are decision making data. The decision making data will be used by the Army to demonstrate that the established CMOs have been achieved. Data will be reported in summary form in an Interim Measures Completion Report, with complete laboratory analytical reports also provided in electronic form (e.g., CD-ROM or equivalent) and as EDDs uploaded to the Army's ERIS system.

Soil considered for use as backfill at the site will be sampled as discussed in the IMWP to demonstrate it does not contain compounds at levels of concern.

SECTION 4. QUALITY ASSURANCE OBJECTIVES

QA is defined as the overall system of activities for assuring the reliability of data produced. Conformance with appended SOPs, excerpted from the approved *Final Master Work Plan, Radford Army Ammunition Plant, Radford, Virginia* will ensure attainment of QA objectives. The system integrates the quality planning, assessment, and corrective actions of various groups in the organization to provide the independent QA program necessary to establish and maintain an effective system for collection and analysis of environmental samples and related activities. The program encompasses the generation of complete data with its subsequent review, validation and documentation.

The Data Quality Objective (DQO) process is a strategic planning approach to ensure environmental data is of the appropriate type, quantity, and quality for decision making.

The overall QA objective is to develop and implement procedures for sample and data collection, shipment, evaluation, and reporting that will allow reviewers to assess whether the field and laboratory procedures meet the criteria and endpoints established in the DQOs. DQOs are qualitative and quantitative statements that outline the decision-making process and specify the data required to support corrective actions. DQOs specify the level of uncertainty that will be accepted in results derived from environmental data. *Guidance for the Data Quality Objectives Process* (USEPA 1994), and *Data Quality Objective Process for Hazardous Waste Sites* (USEPA 2000a) formed the basis for the DQO process and development of RFAAP data quality criteria and performance specifications.

The DQOs for this project have been designed to assure the data demonstrate achievement of the site CMO per the Final RFI/CMS.

4.1 Chemical Data Measurements

4.1.1 Accuracy

Accuracy is a measure of system bias and is defined quantitatively as the degree of agreement of a measurement (or an average of measurements of the same parameter), X, with an accepted reference or true value, T. The accuracy of RFAAP field activities will be qualitatively controlled through the use of standard operating procedures (SOPs) that have been developed to standardize data collection. For chemical data, accuracy of the extraction and analysis procedures will be checked quantitatively by using matrix and surrogate spikes, and blanks. The accuracy will be calculated based on the percent recovery of the spikes and concentrations of target analytes in the blanks as specified in Equation 1.

Equation 1

$$\text{Percent Recovery (\%R)} = \frac{\text{Test Value}}{\text{True Value}} \times 100$$

Field documentation will be used as evidence that investigative protocols follow the appropriate SOPs as presented in Attachment A. Field inspection performed by the contractor QA Officer will be used to identify deviations and execute corrective action (see Section 10.0). Consistent

and proper calibration of equipment throughout site investigations, as described in this QAPP, will ensure the accuracy of the measurements.

4.1.2 Precision

Precision refers to the level of agreement among repeated measurements of the same parameter. It is usually stated in terms of standard deviation, relative standard deviation, relative percent difference, range, or relative range. The overall precision of data is a mixture of sampling and analytical factors. The analytical precision is easier to control and quantify because sampling precision is unique to each site.

Sampling precision will be evaluated by obtaining one duplicate sample for every ten samples collected for each type of media (10%) and by assessing the relative percent difference (RPD) between results. The RPD will be calculated according to Equation 2:

Equation 2

$$RPD\ (\%) = \frac{(XA - XB)}{XM} \times 100$$

Where:

XA, XB = duplicate analyses

XM = mean value of duplicate analyses (XA + XB)/2

The RPD will be calculated for each analytical parameter that was detected in an environmental sample. Duplicate results for aqueous matrices should have an RPD less than 25% and solid matrices will have a RPD less than 35%. If these criteria are not met, a careful examination of the sampling techniques, sample media, and analytical procedure will be conducted to identify the cause of the high RPD and the usefulness of the data.

Analytical precision will be addressed by analysis of matrix spikes (MS) for inorganic analyses; MS and matrix spike duplicates (MSDs) for organic analyses; and laboratory duplicate sample analyses. The RPD for each MS, MSD, and duplicate analysis will be calculated and compared to control criteria. If these criteria are not met, an examination of the data will be conducted to identify the cause of the variability and usefulness of the data. The data will be qualified in accordance with U.S. Environmental Protection Agency (USEPA) Region III criteria (USEPA 1993a, 2000).

4.1.3 Representativeness

Representativeness is a measure of the degree to which the measured results accurately reflect the medium being sampled. It is a qualitative parameter, which is addressed through the proper design of the sampling program in terms of sample location, number of samples, and actual material collected as a “sample” of the population.

Sampling protocols have been developed to assure that samples collected are representative of the media. Field handling protocols (e.g., storage, handling in the field, and shipping) have also

been designed to protect the representativeness of the collected samples. Proper field documentation and QC inspections will be used to establish that protocols have been followed and that sample identification and integrity have been maintained.

4.1.4 Completeness

Completeness is a measure of the amount of information that must be collected during field investigations to allow for successful achievement of investigation objectives. An adequate amount and type of data must be collected for conclusions to be valid. Missing data may reduce the precision of estimates or introduce bias, thus lowering the confidence level of the conclusions. While completeness has been historically presented as a percentage of the data that is considered valid, this does not take into account critical sample locations or critical analytical parameters.

The amount and type of data that may be lost due to sampling or analytical error cannot be predicted or evaluated in advance. The importance of lost or suspect data will be evaluated in terms of the sample location, analytical parameter, nature of the problem, decision to be made, and the consequence of an erroneous decision. Critical locations or parameters for which data is identified as inadequate will either be re-sampled and re-analyzed or the data will be appropriately qualified based on the decision of the Project Officer. The completeness goal percentage of valid data is set at 90$\pm$2% for field activities at RFAAP and will be quantitatively evaluated using Equation 3:

Equation 3

$$\text{Completeness Goal Percentage (\%C)} = \frac{\text{Number of Successful Analyses (useful data)}}{\text{Number of Requested Analyses}} \times 100$$

4.1.5 Comparability

Comparability is the confidence with which one data set can be compared to another. Comparability will be controlled by using SOPs that have been developed to standardize investigative activities. Consistent and proper calibration of equipment throughout the field exercises, as described in this QAP, will assist in the comparability of measurements. Field documentation, reviews and/or QA audits will be used to establish that protocols for sampling and measurement follow appropriate SOPs.

4.1.6 Sensitivity

Sensitivity requirements are expressed differently for various methods. The instrument detection limits (IDLs), the method detection limits (MDLs), and the practical quantitation limits (PQLs) published within USEPA methods are based upon a reagent water matrix, and are not reflective of typical sample matrices; therefore, care will be taken in establishing limits for laboratory analysis. The published limits may not be achievable for environmental samples, but they should compare reasonably with control samples. This compliance will be verified during data validation. Target analytes detected above the IDL or MDL but less than the PQL limits will be reported as estimated values. Target analytes detected in samples above the upper calibration standard will be diluted and re-run by the contract laboratory.

The overall QA objective is to develop and implement procedures for sample and data collection, sample shipment, and reporting that will allow QA data users to establish whether the field and laboratory data collected during the investigation meet the criteria and endpoints established in the DQOs. The QA objective will be achieved through the implementation of specific procedures for sampling, field data collection, chain-of-custody, calibration, internal QC, audits, preventive maintenance, and corrective actions as described in this QAPP.

4.2 Levels of Concern

An integral part of the identification of DQOs is the determination of Levels of Concern (LOCs). These levels are compared with analytical method reporting limits (RLs) to ensure the method is capable of addressing project DQOs, preclude occurrence of false negative issues, and assess best available technology limitations (refer to Section 6.0).). LOCs have been defined for SWMU 57 soils as site specific RGs. Table 4-2 shows the unrestricted use soil RGs. Additionally, the Final CMS indicates that groundwater sampling and analysis will occur in two monitoring wells to confirm the ability to achieve clean closure of the site. Table 4-3 reflects the sampling and analysis to be conducted in wells

Table 4-2 Unrestricted Use Soil RGs
SWMU 57
Radford Army Ammunition Plant, Radford, Virginia

Medium: SOIL	Compound of Concern	RG
	Total Aluminum	40,041 mg/kg
	Total Antimony	13.2 mg/kg
	Total Cadmium	23.2 mg/kg
	Total Chromium	65.3 mg/kg
	Total Iron	50,962 mg/kg
	Total Manganese	2,543 mg/kg

Table 4-3 Groundwater Sampling and Analysis

Medium: GROUNDWATER	Antimony	Laboratory analytical results provided to verify ability to clean close site; no CMOs for groundwater are necessary.
	Arsenic	
	Manganese	
	Chromium	
	TCL VOCs	

4.2.1 LOCs for Groundwater

Groundwater LOCs include the COPCs established by the Final CMS and listed in Table 4-3 above. No Corrective Measures are required for groundwater, per the Final CMS.

4.2.2 LOCs for Soil Confirmatory Samples

Soil confirmatory sample LOCs include the COCs with established RGs in Table 4-2.

4.2.3 Potential LOCs for Backfill Soil

- USEPA Regional Risk Based Screening Levels.

SECTION 5. SAMPLING LOCATIONS AND RATIONALE

A summary of the sampling locations and rationale are provided in the IMWP, including:

- Background/upgradient samples;
- Contaminants of concern;
- Investigative objective;
- Levels of concern;
- Data use priorities;
- Project scope;
- Associated field and laboratory QA/QC samples;
- Number of samples;
- Sample type;
- Sample location;
- Specific site features that will affect sample collection, such as depth or flow conditions;
- Sampling procedures;
- Analytical methods;

Project staff is responsible for becoming familiar with the details of the IMWP as they pertain to their assigned work. The PM and Field Supervisor will ensure that project staff receive appropriate briefings on the scope of work and project specific requirements.

5.1 Decontamination Procedures

Decontamination procedures will follow those in SOP 80.1 Decontamination.

SECTION 6. SAMPLE MANAGEMENT

6.1 Sample Number and Type

The number and types of environmental and QC samples to be collected during a specific sampling event are identified in the site IMWP. The existing RFI data will be used in conjunction with new data generated by UXB-KEMRON.

For SWMU 57, decision making data will include soil confirmation samples and groundwater monitoring data. These data will be used by UXB-KEMRON and the Army to demonstrate achievement of the CMOs for the site. Chemical data also will be generated to demonstrate that backfill emplaced at SMWU 57 is free of contaminant concentrations that could create new environmental concerns at the site. Waste characterization data will be generated for surface water in the pond, soil and monitoring well purge water; waste characterization data will be used by UXB-KEMRON to determine the appropriate means of waste disposal.

QC samples are discussed in Section 9.0. Sample collection, preservation, handling, storage, and shipping will be performed in a manner that minimizes damage, loss, deterioration, and contamination. Procedures described are designed to eliminate external contamination and to ensure data quality by using approved standardized sampling procedures. References to methods of collection and detailed SOPs are provided in Attachment A.

6.2 Sample Containers

The integrity of containers for aqueous and solid samples is ensured by the use of appropriate cleaning techniques. Microbac will provide pre-cleaned sample bottles for chemical analyses according to good laboratory practices. Table 6-1 specifies the sample containers requirements that may be applicable for the UXB-KEMRON work under this QAPP.

6.3 Sample Preservatives

Preservatives will be used, as applicable, to retard hydrolysis of chemical compounds and complexes, to reduce volatility of constituents, and to retard biological action during transit and storage prior to laboratory analysis. Sample containers will be pre-preserved by the laboratory. In addition to chemical preservatives, samples for chemical analysis will be transported to the laboratory in temperature-controlled coolers. Double-bagged ice will be used to maintain the internal cooler temperature required for preservation. A temperature blank will be included in each shipping container to monitor the internal temperature. Tables 6-1 specifies the sample preservative requirements.

6.4 Holding Times

Sample holding time is defined as the interval between sample collection and sample extraction/analysis, such that a sample may be considered valid and representative of the sample matrix. The laboratory QA program will be responsible for ensuring the adequacy of the sample tracking system in precluding holding time deficiencies. Table 6-1 specifies the holding time requirements. Table 6-2 shows analytical services to be provided.

Table 6-1 Summary of Sample Container and Sample Preservations Requirements – SWMU 57				
Parameter	**Sample Container**[1,2]		**Preservation Methods**	**Holding Times**
	Quantity	**Type**		
Solid Samples				
Pesticides/Aroclors	2	250-mL wide-mouth glass amber container	Cool to 4 ± 2°C	Extraction: 7 days Analysis: 40 days
Metals	1	125-mL glass container, Teflon® lined cap	Cool to 4 ± 2°C	6 months
PAHs	1	125-mL glass amber, Teflon® lined cap	Cool to 4 ± 2°C	Extraction: 14 days Analysis: 40 days
Corrosivity (pH)	1	60-mL glass container	Cool to 4 ± 2°C	Immediately
Waste Samples				
TCLP Metals	1	250-mL wide mouth glass container, Teflon® lined cap	Cool to 4 ± 2°C	Leaching: 14 days Analysis: 2 months Mercury Analysis: 28 days
TAL Metals	1	500 mL high density polyethylene (HDPE) with 10% headspace	HNO_3 to pH <2, Cool to 4 ± 2°C	6 months
TCLP VOCs	1	4 oz. Glass	Cool, 4oC	14 days
Chemical Oxygen Demand (COD)	1	125-mL polyethylene container	H_2SO_4 to pH <2, Cool to 4 ± 2°C	28 days
Corrosivity, Paint Filter	1	250-mL wide mouth glass container, Teflon® lined cap	Cool to 4 ± 2°C	Corrosivity: 7 days Reactivity: 7 days
Reactivity	1	250-mL wide mouth glass container, Teflon® lined cap	--	--
Ignitability	1	250-mL wide mouth glass container, Teflon® lined cap	--	--

Table 6-1 Summary of Sample Container and Sample Preservations Requirements – SWMU 57				
Aqueous Samples				
Volatile Organic Compounds	3	40-mL glass vials, Teflon®-lined septum cap	HCl to pH <2, Cool to 4 ± 2°C	14 days
Metals	1	500 mL high density polyethylene (HDPE) with 10% headspace	HNO_3 to pH <2, Cool to 4 ± 2°C	6 months
Corrosivity (pH)	1	60-mL glass container	Cool to 4 ± 2°C	Immediately

[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.

Notes:
mL = milliliter g=gram HNO_3=Nitric Acid
°C= Celsius HCL=Hydrochloric Acid

Table 6-2 Analytical Services			
Matrix	**Analytical Group**	**Data Package Turnaround Time**	**Laboratory / Organization (Name and Address, Contact Person and Tel. No.)**
Solid (soil, backfill soil and/ or pond residuals) and/or Aqueous (Groundwater or Surface Water)	8082	CD-ROM Level 4 CLP-Like & EDD 30 Days	Microbac Laboratories, Inc. 158 Starlite Drive Marietta, OH 45750 Stephanie Mossburg (740) 373-4071
	8270C		
	8270		
	8260B		
	6010B		
	6020		
	8081A		

6.5 Sample Identification

The sample identification number will consist of an alphanumeric designation related to the sampling location (and/or well identification for groundwater samples), media type, and sequential order according to the sampling event. If previous sampling has been performed at the site, new sample IDs will follow the previous identification scheme as closely as possible. QA/QC sample identification numbers will be numbered as above. The sample identification number should not exceed 20 alphanumeric characters.

Site Location Code: The first two characters will be the SWMU number (i.e., 57 for SWMU 57).

Sample/Media Type: The next two characters will be the sample/media types. In this case, the characters will be GW for groundwater, SC for soil confirmation, SW for surface water, WCS for waste characterization soil, BF for backfill, and SB for soil boring.

Sampling Location Number: The next one or two characters will be the number of the sampling location (e.g., 3,4,5).

Sample Depth: The sample representing 2.5 feet will be designated with -2.5' after the boring number. The sample collected from 5 feet will be designated with -5' following the boring number.

Duplicate: Duplicate samples will be identified with a "D" designation followed by a numeric designation corresponding to the sequence of duplicates collected (e.g., D-1). A record of the sample that corresponds to the duplicate will be kept in the field logbook. In this manner, duplicates will be submitted as blind duplicates, eliminating the potential for laboratory bias in analysis.

Sample Identification Examples:
1) A hand auger soil sample collected at the depth of 2.5 feet of boring location four at SWMU 57 would be identified as sample 57SB4-2.5' (for SWMU 57, soil boring four, and 2.5 feet).

2) Quality Control Samples: QC samples will be identified by date (month, day, year), followed by QC sample type, and sequential order number at one digit. The QC sample types include Matrix Spike (MS), Matrix Spike Duplicate (MSD), Rinse Blank (R), and Trip Blank (T).

6.6 Documentation Requirements

Information pertinent to the sampling effort will be recorded in a field logbook and the sample traced by a chain-of-custody form. Entries will be made in indelible ink on consecutively numbered pages, and corrections will consist of line out deletions that are initialed and dated (refer to SOP 10.1, Attachment A).

At a minimum, field logbook entries include the following:

- Project name (cover);
- Name and affiliation of personnel on site;
- Contractor project number;
- Weather conditions;
- General description of the field activity;
- Sample location;
- Sample identification number;
- Time and date of sample collection;
- Specific sample attributes (e.g. sample collection depth flow conditions, or matrix);
- Sampling methodology (grab or composite sample);
- Sample preservation, as applicable;
- Analytical request/methods;
- Associated QA/QC samples;
- Field measurements/observations, as applicable; and
- Signature and date of personnel responsible for documentation.

SOP 10.2 in Attachment A provides specific protocols for recording for recording soil, surface water, and groundwater sampling information, as well as instrument field calibration data in field logbooks.

Each sample container will be annotated in waterproof ink, with the installation name, sample number, sampling date, analytes, and preservatives. The sample label will be permanently affixed to the sample container using polyethylene tape (refer to SOP 50.1 in Attachment A).

Telephone communication will be documented for scope of work changes and/or conditions that impact task deliverables. Related records include telephone logs, e-mail, and facsimiles.

Specific records related to investigative activities may include:

- Boring logs (refer to SOP 10.3 in Attachment A);
- Well construction diagrams (refer to SOP 20.1 in Attachment A);
- Well development records (refer to SOP 20.2 in Attachment A);
- Monitoring well and boring abandonment (SOP 20.3);
- Aquifer test records;
- Laboratory data; and
- Subcontractor permits and qualifications, utility clearance, and unexploded ordnance (UXO) clearance.

6.7 Chain-of-Custody Requirements

Sampling will be evidenced through the completion of a chain-of-custody form, which accompanies the samples from the field to the laboratory. The chain-of-custody form will be annotated to indicate time and date that samples are relinquished. In addition, shipping containers will be affixed with custody seals. Once samples are relinquished to the laboratory, the laboratory chain-of-custody procedures, as described in Section 9.3, will be followed. SOP 10.4 in Attachment A describes specific protocols for using chain-of-custody forms. An example chain of custody form is provided in Figure 10.4-a.

Table 6-3
Analytical Services Summary

Analytical Services Summary					
Matrix	**Analytical Group**	**Analytical SOP**	**Data Package Turnaround Time**	**Laboratory/Organization (Name and Address, Contact Person, and Telephone Number)**	**Backup Laboratory/Organization (Name and Address, Contact Person, and Telephone Number)**
Groundwater	pH, temperature, specific conductance, redox potential, dissolved oxygen		N/A - Field Screening	UXB-KEMRON 156 Starlite Drive Marietta, OH Mary Lou Rochotte (740) 373-4308	None
Groundwater	Chloroform (VOC)	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Groundwater	Metals COCs (see Table 4-2)	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Soil	TAL Metals	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Soil	TCL VOCs	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Soil	TCL Pesticides/PCBs	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None

Analytical Services Summary					
Matrix	**Analytical Group**	**Analytical SOP**	**Data Package Turnaround Time**	**Laboratory/Organization (Name and Address, Contact Person, and Telephone Number)**	**Backup Laboratory/Organization (Name and Address, Contact Person, and Telephone Number)**
Soil	PAHs	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Leachate	Metals	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Leachate	VOCs	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Water/Soil	Chemical Oxygen Demand (COD)	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Water/Soil	Ignitability	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Water/Soil	Corrosivity	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Water/Soil	Reactivity	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None
Solids	Paint Filter Tests	See Attachment C	30 days from sample receipt	Microbac – Ohio Valley Div. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (740) 373-4071	None

SECTION 7. ANALYTICAL PROCEDURES

7.1 Field Testing and Screening

During the environmental sample collection activities conducted at RFAAP, selected physical and chemical parameters at the site should be measured. For example, specific conductance, pH, temperature, redox potential, and dissolved oxygen measurements will generally be collected in conjunction with the chemical data for groundwater and surface water characterization.

Equipment and general procedures for analysis of field samples should reference appropriate SOPs in Attachment A. Because field instrumentation and analytical methodology are continually being updated, field personnel are required to consult manufacturers' instruction manuals for operating procedures.

7.2 Laboratory Procedures for Chemical Analyses

The choice of analytical method is based on the following consideration:

- Regulatory program requirements as specified in USEPA and VDEQ approved site specific RCRA submittals;
- DQOs; and
- Consideration of other practical constraints (temporal, financial, geographic).

Analytical methods will be derived from standard, published methods having regulatory standing. Examples include USEPA SW-846 Test Methods, test methods specified in the Code of Federal Regulations (Title 40 Code of Federal Regulations Part 136 (40 CFR 136) and 40 CFR 261) Standard Methods for the Examination of Water and Wastewater (APHA 1992), U.S. Geological Survey, etc.

Laboratory analyses of site-specific environmental will be in accordance with USEPA SW-846 Test Methods for the analysis of the COCs with established RGs per Table 4-2.

Samples of IDM (decontamination water and soil) will be characterized for disposal as required by the disposal facility selected, and are anticipated to include:

- TCLP VOCs;
- TCLP Metals;
- TAL Metals (water only);
- Chemical Oxygen Demand (COD) (water only);
- Ignitability;
- Corrosivity;
- Reactivity; and
- Paint Filter Test (solids).

Backfill soil samples will be collected and analyzed as presented in the IMWP, with analyses including:

- TAL metals;
- TCL PAHs;
- Pesticides/PCBs;
- pH.

7.2.1 Organics

The following techniques will be used for determination of organic constituents.

7.2.1.1 Chloroform by SW8260B

The aqueous samples are prepared for analysis by purge-and-trap Method 5030 and the solid samples are prepared by purge-and-trap Method 5035. The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by other methods (see Section 1.2 of Method SW8260B). The analytes are introduced directly to a wide-bore capillary column or cryofocused on a capillary pre-column before being flash evaporated to a narrow-bore capillary for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC). Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. (Wide-bore capillary columns normally require a jet separator, whereas narrow-bore capillary columns may be directly interfaced to the ion source.) Identification of target analytes is accomplished by comparing their mass - spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

7.2.1.2 Pesticides by SW8081A

A measured volume or weight of sample (approximately one liter for liquids, and two to 30 grams for solids) is extracted using the appropriate matrix-specific sample extraction technique. Liquid samples are extracted at neutral pH with methylene chloride using Method 3520C (continuous liquid-liquid extractor), or other appropriate technique. Solid samples are using Method 3540C (Soxhlet) or other appropriate technique. A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes. Suggested cleanups include alumina (Method 3610), Florisil (Method 3620), silica gel (Method 3630), gel permeation chromatography (Method 3640), and sulfur (Method 3660). After cleanup, the extract is analyzed by injecting a one microliter (pL) sample into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (ECD) or an electrolytic conductivity detector (ELCD).

7.2.1.3 PCBs by SW8082

A measured volume or weight of sample (approximately one liter for liquids, and two to 30 grams for solids) is extracted using the appropriate matrix-specific sample extraction technique. Aqueous samples are extracted at neutral pH Method 3520C (continuous liquid-liquid extractor), or other appropriate technique. Solid samples are extracted Method 3540C (Soxhlet) or other appropriate technique. Extracts for PCB analysis may be subjected to a sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphate pesticides. Therefore, Method 8082 is not applicable to the analysis of those compounds. Instead, use Method 8081. After cleanup, the extract is analyzed by injecting a 2 µL aliquot into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and ECD. The chromatographic data may be used to identify the seven Aroclors found in Section 1.1 of Method SW8082, individual PCB congeners, or total PCBs.

7.2.1.4 PAHs by 8270-SIM

Method 8270 SIM is a Gas Chromatography/Mass Spectroscopy (GC/MS) method that relies on a mass-selective detector to measure the breakdown fractions of compound. In the SIM mode

(Selective Ion Monitoring), the GC/MS can look for specific ions of a limited target compound list. This allows the analyst to achieve much lower reporting limits than standard Method 8270 that operates in a full scan mode. Method 8270 SIM provides for the detection of parts per billion (ppb) levels of certain PAHs in water and soil matrices. Samples are extracted then concentrated by evaporation. Compounds of interest are separated by capillary column GC and quantitated using the method of internal standards. The MS is tuned prior to analysis to give an acceptable spectrum.

7.2.2 Inorganics

The following techniques will be used for determination of inorganic constituents.

7.2.2.1 Metals by ICP

Prior to analysis, samples are prepared by Method 3010A for aqueous media and Method 3050B for solid media, or other appropriate methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved before analysis. This method describes multielemental determinations by Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES) using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry.

Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices.

Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be defined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used should be as free as possible from spectral interference and should reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 3.0 of Method 3050B should also be recognized and appropriate corrections made; tests for their presence are described in Section 8.5 of Method 3035B.

Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

7.2.3 Waste Samples

7.2.3.1 TCLP Extraction

For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8-micrometer (µm) glass fiber filter, is defined as the TCLP extract. For wastes containing greater than or equal to 0.5% solids, the liquid, if present, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a

0.6 to 0.8-pm glass fiber filter. If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration. Extracts are analyzed using the analytical methods described above.

7.2.3.2 Ignitability

For liquid wastes, the sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample. For solid wastes, in a preliminary test, the test material is formed into an unbroken strip or powder train 250 millimeters (mm) in length. An ignition source is applied to one end of the test material to learn whether combustion will propagate along 200 mm of the strip within a specified time. Materials that propagate **-** burning along a 200-mm strip within the specified time are then subjected to a burning rate test. Materials that do not ignite or propagate combustion as described above do not require further testing. In the burning rate test, the burning time is measured over a distance of 100 mm and the rate of burning is calculated. The test method described here is based on the test procedure adopted by the U.S. Department of Transportation from the United Nations regulations for the international transportation of dangerous goods and is contained in Appendix E to Part 173 of Title 49 of the Code of Federal Regulations (CFR).

7.2.3.3 Corrosivity

The corrosivity of a sample will be based on its pH. The pH of a liquid sample is either analyzed electrometrically using a glass electrode in combination with a reference potential or a combination electrode. The measuring device is calibrated using a series of standard solutions of known pH. For soil/solid waste samples, the sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured. The same procedure is used for pH determination of water and soil samples.

7.2.3.4 Reactivity

An aliquot of acid is added to a fixed weight of waste in a closed system. The generated gas is swept into a scrubber. The analyte is quantitated for cyanide and sulfide as follows. (1) In the colorimetric measurement, the cyanide is converted to cyanogen chloride (CNCI) by reaction of cyanide with chloramine-T at a pH less than eight. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 578 nm for the complex formed with pyridine-barbituric acid reagent and CNCI. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards. The titration measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver sensitive indicator. (2) Sulfide is extracted from the sample by a preliminary distillation procedure and precipitated in a zinc acetate scrubber as zinc sulfide. The sulfide is oxidized to sulfur by adding a known excess amount of iodine. The excess iodine is quantified by titration with a standard solution of phenyl arsine oxide (PAO) or sodium thiosulfate until the blue iodine starch complex disappears. As the use of standard sulfide solutions is not possible because of oxidative degradation, quantitation is based on the PAO or sodium thiosulfate.

7.3 Method Detection Limits

In order to ensure comparability of analytical results across sites, over time, and regardless of contractor, it is necessary to clearly establish uniform definitions for the various detection limits and uniform terminology. Tables 7-1 through 7-6 identify analytical methods to be used and associated LODs and LOQs.

7.3.1 Limit of Detection

The limit of detection (LOD) is the lowest concentration level that can be established as statistically different from a blank. The standard deviation (used to establish the LOD) was defined by replicate measures of the difference between the lowest concentration of analyte instrumentally detectable and a blank value.

7.3.2 Method Detection Limit

USEPA defines the MDL as, "the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is established from analysis of a sample in a given matrix containing the analyte."

7.3.3 Sample Quantitation Limit

The sample quantitation limit (SQL) is using the MDL modified by such factors as dilution, dry weight, etc.

7.3.4 Limit of Quantitation

The limit of quantitation (LOQ) of an individual analytical procedure is the lowest amount of analyte in a sample, which can be quantitatively established with suitable precision and accuracy. The quantitation limit is a parameter of quantitative assays for low levels of compounds in sample matrices and is used particularly for the determination of impurities and/or degradation products.

7.3.5 Reporting Limit

The RL is using the MDL multiplied by an arbitrary factor intended to assure minimum acceptable levels of precision and accuracy.

Table 7-1
Summary of Reporting Limits and Data Quality Levels for Metals
Soil Samples
Radford Army Ammunition Plant, Radford, Virginia

Analyte	CAS Number	Analytical Method	Achievable Laboratory Limits[1]	
			LODs (mg/kg)	LOQs (mg/kg)
Aluminum	7429-90-5	6010B	10	20
Zirconium	7440-67-7	6010B	12.5	25
Arsenic	7440-38-2	6010B	0.5	5
		6020	0.075	0.3
Barium	7440-39-3	6010B	0.1	0.5
		6020	0.075	0.3
Beryllium	7440-41-7	6010B	0.0125	0.5
Boron	7440-42-8	6010B	2.5	5
Cadmium	7440-43-9	6010B	0.05	0.5
		6020	0.025	0.1
Calcium	7440-70-2	6010B	5	10
Chromium	7440-47-3	6010B	0.125	1
		6020	0.1	0.4

Table 7-1 Summary of Reporting Limits and Data Quality Levels for Metals Soil Samples Radford Army Ammunition Plant, Radford, Virginia				
			Achievable Laboratory Limits[1]	
Analyte	CAS Number	Analytical Method	LODs (mg/kg)	LOQs (mg/kg)
Cobalt	7440-48-4	6010B	0.125	1
		6020	0.125	0.5
Copper	7440-50-8	6010B	0.5	1
		6020	0.15	0.6
Iron	7439-89-6	6010B	1	3
Lead	7439-92-1	6010B	0.5	5
		6020	0.1	0.2
Lithium	7439-93-2	6010B	2.5	5
Magnesium	7439-95-4	6010B	12	25
Manganese	7439-96-5	6010B	0.1	0.5
		6020	0.05	0.2
Molybdenum	7439-98-7	6010B	1.5	5
Nickel	7440-02-0	6010B	0.5	2
		6020	0.2	0.8
Potassium	9/7/7440	6010B	25	50
Selenium	7782-49-2	6010B	0.5	5
		6020	0.1	0.2
Silver	7440-22-4	6010B	0.25	2
		6020	0.05	0.2
Sodium	7440-23-5	6010B	5	25
Strontium	7440-24-6	6010B	0.25	0.5
Thallium	7440-28-0	6010B	1	25
		6020	0.01	0.02
Tin	7440-31-5	6010B	5	25
Titanium	7440-32-6	6010B	0.5	2
Vanadium	7440-62-2	6010B	0.25	0.5
		6020	0.125	0.5
Zinc	7440-66-6	6010B	0.5	1
		6020	0.625	2.5
Phosphorus	7723-14-0	6010B	25	50
Antimony	7440-36-0	6010B	0.5	10
		6020	0.05	0.1
Uranium	7440-61-1	6020	0.1	0.4

[1] Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.

Table 7-2
Summary of Reporting Limits and Data Quality Levels for Metals
Water Samples
Radford Army Ammunition Plant, Radford, Virginia

Analyte	CAS Number	Analytical Method	Achievable Laboratory Limits[1]	
			LODs (mg/L)	LOQs (mg/L)
Calcium	7440-70-2	6010B	0.1	0.2
Vanadium	7440-62-2	6010B	0.005	0.01
		6020	0.00025	0.001
Colbalt	7440-48-4	6010B	0.0025	0.02
		6020	0.00025	0.001
Copper	7440-50-8	6010B	0.005	0.02
		6020	0.0005	0.002
Iron	7439-89-6	6010B	0.025	0.1
Lead	7439-92-1	6010B	0.01	0.1
		6020	0.00025	0.001
Lithium	7439-93-2	6010B	0.05	0.1
Magnesium	7439-95-4	6010B	0.25	0.5
Manganese	7439-96-5	6010B	0.005	0.01
		6020	0.0005	0.002
Molybdenum	7439-98-7	6010B	0.005	0.1
Nickel	7440-02-0	6010B	0.005	0.04
		6020	0.001	0.004
Potassium	7440-09-7	6010B	0.25	1
Selenium	7782-49-2	6010B	0.04	0.08
		6020	0.0005	0.001
Silicon	7440-21-3	6010B	0.25	1
Silver	7440-22-4	6010B	0.005	0.01
		6020	0.00025	0.001
Sodium	7440-23-5	6010B	0.25	0.5
Strontium	7440-24-6	6010B	0.005	0.01
Thallium	7440-28-0	6010B	0.1	1
		6020	0.00005	0.0002
Zinc	7440-66-6	6010B	0.005	0.02
		6020	0.005	0.025
Aluminum	7429-90-5	6010B	0.05	0.1
Antimony	7440-36-0	6010B	0.05	0.2
		6020	0.00025	0.001
Arsenic	7440-38-2	6010B	0.01	0.1
		6020	0.00025	0.001
Barium	7440-39-3	6010B	0.0025	0.01
		6020	0.0005	0.003
Beryllium	7440-41-7	6010B	0.005	0.01
Boron	7440-42-8	6010B	0.05	0.1
Cadmium	7440-43-9	6010B	0.0025	0.01
		6020	0.000125	0.0005
Silica, Calculated as SiO_2		6010B	0.25	1
Phosphorus	7723-14-0	6010B	0.5	1

Table 7-2 Summary of Reporting Limits and Data Quality Levels for Metals Water Samples Radford Army Ammunition Plant, Radford, Virginia				
			Achievable Laboratory Limits[1]	
Analyte	CAS Number	Analytical Method	LODs (mg/L)	LOQs (mg/L)
Zirconium	7440-67-7	6010B	0.25	0.5
Tin	7440-31-5	6010B	0.05	0.5
Titanium	7440-32-6	6010B	0.005	0.03
Chromium	7440-47-3	6010B	0.0025	0.02
		6020	0.0005	0.002
Uranium	7440-61-1	6020	0.0025	0.001

[1] Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method

Table 7-3 Summary of Reporting Limits and Data Quality Levels for Pesticides Soil Samples Radford Army Ammunition Plant, Radford, Virginia				
			Achievable Laboratory Limits[1]	
Analyte	CAS Number	Analytical Method	LODs (ug/kg)	LOQs (ug/kg)
4,4'-DDD	72-54-8	8081A	0.33	1.65
4,4'-DDE	72-55-9	8081A	0.33	1.65
4,4'-DDT	50-29-3	8081A	0.33	1.65
Aldrin	309-00-2	8081A	0.33	1.65
alpha-BHC	319-84-6	8081A	0.33	1.65
alpha-Chlordane	5103-71-9	8081A	0.33	1.65
gamma-Chlordane	5103-74-2	8081A	0.33	1.65
beta-BHC	319-85-7	8081A	0.33	1.65
delta-BHC	319-86-8	8081A	0.33	1.65
Dieldrin	60-57-1	8081A	0.33	1.65
Endosulfan I	959-98-8	8081A	0.33	1.65
Endosulfan II	33213-65-9	8081A	0.33	1.65
Endosulfan sulfate	1031-07-8	8081A	0.33	1.65
Endrin	72-20-8	8081A	0.33	1.65
Endrin aldehyde	7421-93-4	8081A	0.33	1.65
Endrin ketone	53494-70-5	8081A	0.33	1.65
gamma-BHC (Lindane)	58-59-9	8081A	0.33	1.65
Heptachlor	76-44-8	8081A	0.33	1.65
Heptachlor epoxide	1024-57-3	8081A	0.33	1.65
Methoxychlor	72-43-5	8081A	0.33	1.65
Toxaohene	8001-35-2	8081A	16.7	33.0

[1] Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method

Table 7-4
Summary of Reporting Limits and Data Quality Levels for PCBs
Soil Samples
Radford Army Ammunition Plant, Radford, Virginia

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

[1] Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method

Table 7-5
Summary of Reporting Limits and Data Quality Levels for PAHs
Soil Samples
Radford Army Ammunition Plant, Radford, Virginia

Analyte	CAS Number	Analytical Method	Achievable Laboratory Limits[1]	
			LODs (ug/kg)	LOQs (ug/kg)
Phenanthrene	85-01-8	8270	1.25	2.5
Indeno(1,2,3-cd)pyrene	193-39-5	8270	1.25	2.5
Naphthalene	91-20-3	8270	1.25	2.5
Acenaphthene	83-32-9	8270	1.25	2.5
1-Methylnaphthalene	90-12-0	8270	1.25	2.5
Acenaphthylene	208-96-8	8270	1.25	2.5
Anthracene	120-12-7	8270	1.25	2.5
Benzo(a)anthracene	56-55-3	8270	1.25	2.5
Benzo(a)pyrene	50-32-8	8270	1.25	2.5
Benzo(b)fluoranthene	205-99-2	8270	1.25	2.5
Benzo(g,h,i)perylene	191-24-2	8270	1.25	2.5
Benzo(k)fluoranthene	207-08-9	8270	1.25	2.5
Chrysene	218-01-9	8270	1.25	2.5
Pyrene	129-00-0	8270	1.25	2.5
Dibenzo(a,h)anthracene	53-70-3	8270	1.25	2.5
Fluoranthene	206-44-0	8270	1.25	2.5
Fluorene	86-73-7	8270	1.25	2.5
2-Methylnaphthalene	91-57-6	8270	1.25	2.5

[1] Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.

Table 7-6
Summary of Reporting Limits and Data Quality Levels for VOCs
Water Samples
Radford Army Ammunition Plant, Radford, Virginia

Analyte	CAS Number	Analytical Method	Achievable Laboratory Limits[1]	
			LODs (ug/L)	LOQs (ug.L)
1,1,1,2-Tetrachloroethane	630-20-6	8260	0.25	1
1,1,1-Trichloroethane	71-55-6	8260	0.25	1
1,1,2,2-Tetrachloroethane	79-34-5	8260	0.2	1
1,1,2-Trichloroethane	79-00-5	8260	0.25	1
1,1-Dichloroethane	75-34-3	8260	0.125	1
1,1-Dichloroethene	75-35-4	8260	0.5	1
1,1-Dichloropropene	563-58-6	8260	0.25	1
1,2,3-Trichlorobenzene	87-61-6	8260	0.15	1
1,2,3-Trichloropropane	96-18-4	8260	0.5	1
1,2,4-Trichlorobenzene	120-82-1	8260	0.2	1
1,2,4-Trimethylbenzene	95-63-6	8260	0.25	1
1,2-Dibromo-3-chloropropane	96-12-8	8260	1	5
1,2-Dibromoethane	106-93-4	8260	0.25	1
1,2-Dichlorobenzene	95-50-1	8260	0.125	1
1,2-Dichloroethane	107-06-2	8260	0.25	1
1,2-Dichloropropane	78-87-5	8260	0.2	1
1,3,5-Trimethylbenzene	108-67-8	8260	0.25	1
1,3-Dichlorobenzene	541-73-1	8260	0.25	1
1,3-Dichloropropane	142-28-9	8260	0.2	1
1,4-Dichlorobenzene	106-46-7	8260	0.125	1
2,2-Dichloropropane	594-20-7	8260	0.25	1
2-Butanone	78-93-3	8260	2.5	10
2-Chloroethyl vinyl ether	110-75-8	8260	2	10
2-Chlorotoluene	95-49-8	8260	0.125	1
2-Hexanone	591-78-6	8260	2.5	10
4-Chlorotoluene	106-43-4	8260	0.25	1
4-Methyl-2-pentanone	108-10-1	8260	2.5	10
Acetone	67-64-1	8260	2.5	10
Benzene	71-43-2	8260	0.125	1
Bromobenzene	108-86-1	8260	0.125	1
Bromochloromethane	74-97-5	8260	0.2	1
Bromodichloromethane	75-27-4	8260	0.25	1
Bromoform	75-25-2	8260	0.5	1
Bromomethane	74-83-9	8260	0.5	1
Carbon disulfide	75-15-0	8260	0.5	1
Carbon tetrachloride	56-23-5	8260	0.25	1
Chlorobenzene	108-90-7	8260	0.125	1
Chloroethane	75-00-3	8260	0.5	1
Chloroform	67-66-3	8260	0.125	1
Chloromethane	74-87-3	8260	0.5	1
cis-1,2-Dichloroethene	156-59-2	8260	0.25	1
cis-1,3-Dichloropropene	10061-01-5	8260	0.25	1

Table 7-6 Summary of Reporting Limits and Data Quality Levels for VOCs Water Samples Radford Army Ammunition Plant, Radford, Virginia				
Analyte	CAS Number	Analytical Method	Achievable Laboratory Limits[1]	
			LODs (ug/L)	LOQs (ug.L)
Chlorodibromomethane	124-48-1	8260	0.25	1
Dibromomethane	74-95-3	8260	0.25	1
Dichlorodifluoromethane	75-71-8	8260	0.25	1
Ethylbenzene	100-41-4	8260	0.25	1
Hexachlorobutadiene	87-68-3	8260	0.25	1
Isopropylbenzene	98-82-8	8260	0.25	1
m-,p-Xylene	179601-23-1	8260	0.5	1
Methylene chloride	75-09-2	8260	0.25	5
n-Butylbenzene	104-51-8	8260	0.25	1
n-Propylbenzene	103-65-1	8260	0.125	1
Naphthalene	91-20-3	8260	0.2	1
o-Xylene	95-47-6	8260	0.25	1
p-Isopropyltoluene	99-87-6	8260	0.25	1
sec-Butylbenzene	135-98-8	8260	0.25	1
Styrene	100-42-5	8260	0.125	1
tert-Butylbenzene	98-06-6	8260	0.25	1
Tetrachloroethene	127-18-4	8260	0.25	1
Toluene	108-88-3	8260	0.25	1
trans-1,2-Dichloroethene	156-60-5	8260	0.25	1
trans-1,3-Dichloropropene	10061-02-6	8260	0.5	1
Trichloroethene	79-01-6	8260	0.25	1
Trichlorofluoromethane	75-69-4	8260	0.25	1
Vinyl acetate	108-05-4	8260	2.5	10
Vinyl chloride	75-01-4	8260	0.25	1
Chlorobenzene-d5	3114-55-4	8260		
Fluorobenzene	462-06-6	8260		
1,4-Dichlorobenzene-d4	3855-82-1	8260		
4-Bromofluorobenzene	460-00-4	8260		
Toluene-d8	2037-26-5	8260		
1,2-Dichloroethane-d4	17060-07-0	8260		
Dibromofluoromethane	1868-53-7	8260		

7.4 Physical Testing Methods

No physical testing methods are anticipated for this project.

SECTION 8. CALIBRATION PROCEDURES

8.1 Laboratory Calibration

Prior to sample analysis, chemical calibration of each target analyte/compound must be performed to ensure analytical instrumentation is functioning within the established sensitivity range. Laboratory calibration protocols will be specified in an analytical QAPP to be either retained within the contractor's file or appended to the IMWP. Areas to be discussed include solution validation, initial, and continuing calibration. Analytical instruments will be calibrated initially and periodically checked to ensure that the initial calibration remains valid. Generally, this verification will take the form of analysis of at calibration standard, usually at the mid-point of the calibration range, and a comparison of the percent difference or percent drift (both are abbreviated as %D) between the initial calibration response and the calibration check. The %D is calculated as:

Equation 4

$$\text{Percent Difference/Percent Drift (\%D)} = \frac{\text{Calibration Check Response or Amount}}{\text{Initial Calibration Response or Amount}} \times 100$$

8.2 Instrument/Field Equipment Testing, Inspection, and Maintenance

Equipment and supplies purchased in support of these activities will be purchased according to the provisions of this plan, which requires a documented review of the vendor and equipment selection process and receipt inspection and/or testing as appropriate.

In summary, those procedures require that new equipment be tested with an appropriate standard or standards to ensure they function according to intended use. QC sample results from initial field use of equipment and supplies will be reviewed by the Field Manager, Laboratory Manager or his designee to identify potential causes for concern regarding new equipment and supplies received. Corrective actions will be implemented and documented according to the provisions of Section 10.0 as required.

Instrument maintenance, both routine and preventive, will be performed according to manufacturers' instructions. A preventive maintenance plan allows for periodic instrumentation checks for problems that occur frequently. The objective of a preventive maintenance plan is to rectify equipment problems before they become serious. Preventive maintenance also brings attention to those areas of the instrument susceptible to degradation from aging, toxic/corrosive attack, and clogging due to environmental factors.

Procedures for preventive maintenance are contained in each instruments associated manual under the maintenance/troubleshooting sections. Logbooks, such as those described in SOP 10.1 in Attachment A, will be maintained for each instrument used in the laboratory. Maintenance, calibration, and performance data will be entered by the operator and will be periodically reviewed by the Field/Laboratory Manager.

An inventory of critical spare parts will be maintained on-site during field activities. Critical spare parts are defined as those that upon failure would cause a delay in field or laboratory activities of greater than 4 hours. The specific needs of the program will be established at the discretion of the Field/Laboratory Manager.

8.3 Inspection/Acceptance for Supplies and Consumables

The Field/Laboratory Manager and/or his representative will inspect materials and consumables against the purchase order specifications to verify their fitness for use. Materials received will be properly labeled and recorded on the inventory log for accuracy. An expiration date will be assigned immediately to standards, reagents, and solvents. Documentation concerning the quality of materials used on-site will be retained in the central files.

8.4 Field Equipment Calibration and Maintenance

The proper calibration, maintenance, and documentation of field equipment are designed to assure that the field equipment is functioning optimally. Calibration and maintenance will follow manufacturer's specifications. The frequency of calibration is discussed in the APP. Documentation will be kept in equipment logbooks, which are required to record usage, maintenance, calibration, and repair.

8.4.1 Water Quality Parameters

Equipment used for analyzing water quality parameters (e.g., pH, conductivity, temperature, dissolved oxygen, oxidation/reduction potential, turbidity) will be calibrated at the site daily or more frequently as conditions dictate. The calibration for each type of parameter monitored will include a daily initial measurement prior to calibration, a measurement after calibration, and measurement at the end of the day. Measurements will be documented in the field logbooks (SOPs 10.1 and 10.2 in Attachment A) by the field personnel performing the calibration.

8.4.2 Air Quality Parameters

Equipment for analyzing air quality parameters (e.g., organic vapors, lower explosive limit, and percent oxygen) will be calibrated at the site daily. The calibration will include a daily initial measurement prior to calibration, a measurement after calibration, and a measurement at the end of the day. Measurements will be documented in the field logbook forms for meter calibration (SOP 10.2 in Attachment A) by the field personnel performing the calibration. Specific SOPs for air quality instruments are given in SOP Series 90.

8.4.3 Screening

Screening kits, if used, will be calibrated upon arrival according to the manufacturer's instructions.

SECTION 9. INTERNAL QUALITY CONTROL CHECKS

9.1 Laboratory Quality Control Elements

Method quality objectives (MQOs) will be specified in the IMWP. These MQOs provide a basis for project data review and should be evaluated during project planning for data use applicability.

9.2 Special Training Requirements and Certification

In addition to health and safety training as required for hazardous site workers by the Occupational Safety and Health Administration, field and laboratory personnel will receive technical training in the techniques they are expected to carry out. Training will consist of, at a minimum, on the job training by a senior staff member in the SOPs they are expected to implement, documented by virtue of a signed copy of the SOP or cover sheet for the field sampling plan. The QA Manager will retain this documentation on file.

Analysts will receive appropriate training in procedures, safety, and waste disposal. The Laboratory QA Manager, or his designee, will train analysts on analytical methods and operation of laboratory instrumentation. Analysts will be required to prove the ability to execute methods they perform with acceptable precision and accuracy through analysis of performance evaluation samples in quadruplicate, which meet the applicable QC standards of the method. Training completed by the analyst will be documented by the Laboratory QA Manager and maintained on file. These records will serve the additional purpose of providing for validation of non-standard methods of analysis.

9.3 Field Quality Control Samples

Field investigations at RFAAP will require the collection of several types of field QC samples including duplicate samples, rinse blanks, temperature blanks, trip blanks, and source water samples, as outlined in Table 9-1. If a target analyte is detected in a QC blank, data will be evaluated to establish if corrective action measures will be taken. Field QC elements of a QA program for field investigations at RFAAP are summarized in Table 9-2.

Table 9-1
Types of Field Quality Control Samples
Radford Army Ammunition Plant, Radford, VA

Type of Control	Purpose of Sample	Collection Frequency
Duplicate Sample	To ensure precision in sample homogeneity during collection and analysis	10% of field samples per matrix
Rinse Blank	To ensure the decontamination of sampling equipment has been adequately performed; to assess cross contamination and/or incidental contamination to the sample container	One per 20 samples or one per day
Temperature Blank	To verify sample cooler temperature during transport	One temperature blank per cooler
Trip Blank	To evaluate if cross contamination occurs during shipment or storage with volatile organic analyses (VOA) samples	One trip blank per cooler of VOA samples
Source Water	To characterize decontaminated water	One per source

Table 9-2 Field QC Elements of a QA Program Radford Army Ammunition Plant, Radford, VA				
Item	**DQO**	**Parameter**	**Frequency of Association**	**Criteria Requirement**
Source Water	R	Target list of parameters	Per project	Less than USACE reporting limit or if detected approved by USACE
Field Duplicates	P	Target list of parameters	One per ten samples	RPD ≤25% aqueous; ≤35% solid
Trip Blank	A,R	Volatiles in water	One per cooler with volatiles	No target analytes
Rinse Blank	A,R	Target list of parameters	One per 20 samples per matrix per equipment type	No target analytes
Field Logbook	A,C	Target list of parameters	Daily	Filled out and representative of investigation activities
Chain of Custody	R	Target list of parameters	Every sample	Filled out correctly to include signatures; no missing or incorrect info
Chemical Parameter Forms	R	Target list of parameters	Every sample	Filled out correctly to include analytical parameters; and applicable coding info
Field Instrument Calibration Logs	A	Target list of parameters	Every measurement	Measurements must have associated calibration reference

Legend:
A = Accuracy
C = Completeness
P = Precision
R = Representativeness

Table 9-3 QC Guidelines for PAHs Soil Samples					
Sampling Procedure	**Analytical Method**	**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)**
SOP 30.1 Soil Sampling	8270	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	TBD	Field Duplicate	S&A
		Accuracy/Bias /Contamination	<LOQ	Method Blank	A
		Accuracy/Bias	See Table 10-1	LCS	A
		Accuracy/Bias	See Table 10-1	MS/MSD	A
		Precision	See Table 10-1	MS/MSD	A
		Precision	See Table 10-1	Surrogates	A
		Precision	See Table 10-1	Internal Standards	A

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

Table 9-4 QC Guidelines for PCB Soil Samples					
Sampling Procedure	**Analytical Method**	**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)**
SOP 30.1 Soil Sampling	8082	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	TBD	Field Duplicate	S&A
		Accuracy/Bias /Contamination	<LOQ	Method Blank	A
		Accuracy/Bias	See Table 10-1	LCS	A
		Accuracy/Bias	See Table 10-1	MS/MSD	A
		Precision	See Table 10-1	MS/MSD	A
		Precision	See Table 10-1	Surrogates	A

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

Table 9-5 QC Guidelines for Metals Soil Samples					
Sampling Procedure	**Analytical Method**	**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)**
SOP 30.1 Soil Sampling	6010B & 6020	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	TBD	Field Duplicate	S&A
		Accuracy/Bias /Contamination	See Table 10-1	Preparation Blank	A
		Accuracy/Bias /Contamination	See Table 10-1	Calibration Blanks	A
		Accuracy/Bias	See Table 10-1	LCS	A
		Accuracy/Bias	See Table 10-1	MS	A
		Precision	See Table 10-1	Laboratory Duplicate	A
		Accuracy/Bias	See Table 10-1	Serial Dilution	A

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

Table 9-6 QC Guidelines for VOCs Water Samples					
Sampling Procedure	**Analytical Method**	**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-00 SOP for Groundwater Sampling	8260B	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	TBD	Field Duplicate	S&A
		Accuracy/Bias /Contamination	<LOQ	Method Blank	A
		Accuracy/Bias	See Table 10-1	LCS	A
		Accuracy/Bias	See Table 10-1	MS/MSD	A
		Precision	See Table 10-1	MS/MSD	A
		Precision	See Table 10-1	Surrogates	A
		Precision	See Table 10-1	Internal Standards	A

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

Table 9-7 QC Guidelines for Metals Water Samples					
Sampling Procedure	**Analytical Method**	**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-00 SOP for Groundwater Sampling	6010B & 6020	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	TBD	Field Duplicate	S&A
		Accuracy/Bias /Contamination	See Table 10-1	Preparation Blank	A
		Accuracy/Bias /Contamination	See Table 10-1	Calibration Blanks	A
		Accuracy/Bias	See Table 10-1	LCS	A
		Accuracy/Bias	See Table 10-1	MS	A
		Precision	See Table 10-1	Laboratory Duplicate	A
		Accuracy/Bias	See Table 10-1	Serial Dilution	A

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

Table 9-8 QC Guidelines for Pesticides (8081A) Soil Samples			
Procedure	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Initial calibration curve (ICAL)	Set-up, major maintenance	See Table 10-1	Must meet criteria prior to sample analysis
Continuing calibration verification (calibration check)(CCV)	Daily before sample analysis, and after every 10 samples and at the end of the analysis sequence	See Table 10-1	If criteria are not met, reanalyze the daily standard. If the daily standard fails a second time, initial calibration must be repeated.
Independent reference standard (LCS)	1 per matrix/batch: Maximum of 20 samples per batch	See Table 10-1	Qualify associated data biased high or biased low as appropriate.
Method blanks	1 per batch	See Table 10-1	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank Document source of contamination.
Surrogate spikes	Every sample, standard, and quality control sample	See Table 10-1	If any surrogate compounds do not meet criteria, there should be a re-analysis to confirm that the non-compliance is due to the sample matrix effects rather than laboratory deficiencies
Matrix spike and matrix spike duplicate	1 per matrix/batch; maximum of 20 samples per batch	See Table 10-1	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.

Table 9-9 Secondary Data Criteria and Limitations Radford Army Ammunition Plant, Radford, Virginia					
Secondary Data - Title	**Data Source**	**Date**	**Data Generator(s) Originating Org., Data Types, Data Generation / Collection Dates**)	**How Data Will be Used**	**Limitations of Data Use**
SWMU 57					
	Final RFI/CMS	9/2009	URS	Determine waste disposal characterization. Determine pond solids and soil properties. Evaluate COCs and their distribution at the site to assist in design of clean closure specifications.	None

SECTION 10. DATA REDUCTION, VALIDATION, REPORTING, AND MANAGEMENT

The intended use of the data and the associated acceptance criteria for data quality will be established prior to initiation of the data collection. Reported data will include when appropriate, statements of precision, accuracy, representativeness, completeness, and comparability. Data processing procedures will be documented, reviewed, and revised by the QA Officer, as required to meet project-specific DQOs. The laboratory QA Officer will be responsible for data processing at the contract laboratory.

10.1 Method Detection Limit and Reporting Limits

MDL and RL will be included in the IMWP. In general, solid sample results will be reported in micrograms per kilogram (µg/kg) or milligrams per kilogram (mg/kg) depending on the method used for analysis. Aqueous sample results will in general be reported in units of micrograms per liter (µg/L) or milligrams per liter (mg/L) depending on the methods used. The IMWP for site-specific investigations will specify the reporting units for samples along with the associated MDLs and RLs.

The reported data will contain no more than three significant digits and will be rounded to the appropriate number of significant digits, based on certification class and dilution, after calculations have been completed.

10.2 Rounding Rules

The following rules will be used in data validation and reporting for rounding:

- If the figure following those to be retained is less than five, the figure is dropped, and the retained figures are left unchanged;

- If the figure following those to be retained is greater than five, the figure is dropped and the last figure is raised by one. For example, 1.26 is rounded off to 1.3; and

- If the figure following those to be retained is five, and if there are no figures other than the zeros beyond the five, the figure five is dropped, and the last-place figure is increased by one if it is an odd number or it is kept if it is an even number. For example, 1.45 is rounded off to 1.4, while 1.56 is rounded off to 1.6.

10.3 Collection

Collection of analytical data will begin when samples arrive at the laboratory. The Microbac Laboratory in its entirety is a secure area, and all samples received and logged into the laboratory remain in the custody of the Sample Custodian, supervisor or analyst until time of disposal. Refrigerators, freezers and other designated sample storage areas will be securely maintained or locked. Only the designated Sample Custodian or supervisory personnel will have keys to locked sample storage units until removed for sample preparation or analysis. The following minimum custody procedures will be followed:

- All samples are received and inspected.
- The Sample Custodian signs and dates the chain-of-custody form provided by the Field Team.
- The samples are stored in the appropriate storage unit.
- Several laboratory documents are used to document which laboratory personnel handled the samples, including bench sheets, sample preparation logbooks, and instrument run logbooks.

- The original copy of the chain-of-custody form is included in the laboratory report and a copy is maintained in the laboratory files.

10.3.1 Sample Receipt & Inspection

The Sample Custodian will receive all incoming samples and will sign for the container. The Custodian will open the shipping containers and note the presence/absence of chain-of-custody forms and seals, airbills, or bills-of-lading. The sample temperature is then read.

The Custodian will examine the shipping container to verify the integrity of the sample(s) and examine the sample documentation and identification to assure it is correct and the proper preservative has been used. The preservatives are checked by the Sample Custodian. If inspection indicates samples were damaged in transit, the shipping container will be moved to the hood in Log-in, assessments of the damage will be made and the appropriated Technical Services Representative will be notified. The Site Manager will be immediately contacted and determination of the degree of hazard will be made. If damage is minimal and the Remediation Manager requests it, an attempt to salvage the sample(s) will be made if can be done safely. In the event of damaged hazardous samples, Laboratory Spill Response Team will be notified per the laboratory's Chemical Hygiene Plan.

The Sample Custodian will log in the into the LIMS system and will indicate the actual date and time received and the original receipt documentation will be included with the chain-of-custody form.

The Sample Custodian will compare the chain-of-custody forms and labels to verify agreement of information contained therein. If major discrepancies are found, they will be documented on the Cooler Inspection Checklist and the Project Chemist will be immediately notified. Written documentation of all problem resolutions will be placed in the project/case file. If there are no problems with the samples received, the Sample Custodian files the signed chain-of-custody form and Cooler Inspection Checklist in the project/case file. After the sample is logged in, it will be immediately stored under the proper conditions.

Sample labels or other sample documents that appear to be contaminated due to sample breakage or other problems will be dried under a fume hood and be separately sealed in plastic bags, if necessary, prior to being placed in case files. The Project Chemist must also be notified.

10.3.2 Sample Storage

Samples and extracts will be stored in uniquely identified refrigerators that are in secure areas of the laboratory. The Sample Custodian or designated assistant will check the temperature of each refrigerator in the log-in area, twice daily, (once on weekends and holidays) and maintain a record book. This record book will be reviewed on a monthly basis by the Support Services Supervisor to note any trends or inconsistencies. The acceptable range for sample storage is 0 - 6°C, as long as freezing does not occur. The Sample Custodian will notify the Support Services Supervisor of any refrigerator temperature problem that cannot be corrected by simple thermostat adjustment. A list of emergency repair numbers for the refrigeration units is attached to the walk-in refrigerator's exterior.

10.3.3 Sample Distribution and Tracking

Both the preparation and the analysis of samples will be documented using special forms (logbooks). Once analysis is complete, the analyst will return the unused sample to the Sample

Custodian area for return to the main cooler or to the Sample Archive Room whichever is appropriate. Samples will be returned to their original storage units after completion of analyses. Samples that have exceeded their regulated holding period will be placed in the Sample Archive Room. They are routinely stored in this area for a minimum of 14 days after the due date for the analytical report. They are then disposed per appropriate protocol listed. Extended archive beyond 14 days with refrigeration is available for specific projects or as required by contract.

It is important to note that samples received for analysis of volatile organic compounds (VOCs) are segregated from other samples. Standards are also segregated from all samples in designated storage units.

10.3.4 Sample Security

All sample storage refrigerators are equipped with a lock. The units are monitored by the Sample Custodian during business hours. After business hours, the unit is kept locked and only selected personnel have access (by key) to the sample storage unit. All samples, extracts and digests will be stored in segregated areas

10.3.5 Electronic Data Security

Data integrity is insured through LIMS multi-level security. Access to the specific user privileges can be individually controlled. Each user has his/her own user name and password which allows certain privileges. Several pieces of hard copy documentation are generated for verification, but are unsigned. Signatures on the final report indicate that all of these forms have been reviewed. Reports are automatically screened for errors by LIMS at the time of printing. Any case numbers that produce errors at the time of printing are routed to the appropriate person or section for correction. If no errors are produced at the time of printing, the finished report is stamped with the laboratory manager's signature stamp. All level four data undergoes a chemist review before it is available for printing or reporting. Hard copy data, which contains all of the data regarding a group of samples, are kept in a master file, labeled with the login numbers.

10.4 Data Reduction

Data reduction frequently includes computation of analytical results from raw instrument data and summary statistics, including standard errors, confidence intervals, test of hypotheses relative to the parameters, and model validation. Data reduction procedures address the reliability of computations and the overall accuracy of the data reduction. The numerical transformation algorithms used for data reduction will be verified against a known problem set to ensure that the reduction methods are correct. The equations and the typical calculation sequence that should be followed to reduce the data to the acceptable format are instrument and method-specific. Where standard methods are modified, data reduction techniques will be described in a report accompanying the data.

10.4.1 Gas Chromatography/Mass Spectrometry Results

Qualitative identification will be established by obtaining extracted ion current profiles (EICPs) for the primary ion mass to charge ratio (m/z) and the secondary masses for each compound. Positive identification will be based on the following criteria:

- The intensity of the three characteristic masses of each compound must maximize in the same ratio (± 20 percent (%)), within one scan of each other;
- The relative retention time must fall within 30 seconds of the retention time of the authentic compound; and

- The relative peak heights of the three characteristic masses in the EICPs must fall within 20% of the relative intensities of these masses in a reference mass spectrum (e.g., standard analysis or reference library).

Structural isomers to be listed as separate compounds must have acceptable resolution. Acceptable resolution is achieved if, in a standard mix, the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers will be identified as isomeric pairs.

The calculation for the concentration for the suspect peak will be made using the average response factor (RF) for each compound, which was obtained from the daily calibration.

Water

Equation 5

$$C_s = \frac{(A_s)(C_{is})(V_t)(D)}{(A_{is})(RF)(V_o)}$$

Where:

C*w* = Compound concentration (µg/L);

A*s* = Peak area of characteristic in m/z for the compound to be measured;

A*is* = Peak area of characteristic in m/z for the internal standard;

C*is* = Concentration of the internal standard (µg);

V*t* = Final volume of total extract (µL) used in semi-volatile organic compound (SVOC) analyses;

V*o* = Volume of water (L) extracted or purged;

V*i* = Volume of extract injected; SVOC analyses (µL);

D = Dilution factor; and

RF = Compound response factor calculated from Equation 6.

Equation 6

$$C_s = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Where:

A*s* = Area of the characteristic ion for the compound being measured;

A*i* = Area of the characteristic ion for the specific internal standard;

C*is* = Concentration of the specific internal standard; and

C*s* = Concentration of the compound being measured.

Soil

Equation 7

$$C_s = \frac{(A_s)(C_{is})(V_t)(D)}{(A_{is})(RF)(V_i)(W_s)(M)}$$

Where:

C*s* = Compound concentration in the soil sample (µg/g);

A*s* = Area of the characteristic ion for the compound being measured;

C*is* = Internal standard concentration (µg);

A*is* = Area of characteristic ion for the specific internal standard;

V*t* = Volume of total extract (µL);

V*i* = Volume of extract injected (µL);

W*s* = Mass of sample extracted or purged (g);

D = Dilution factor;

M = Percent dry weight of sample/100; and

RF = Compound response factor calculated from the calibration curve using the same equation as that used for water samples.

10.4.2 Gas Chromatographic Results

Calculations will be performed for each compound after it is identified. Identification will be based on the relative retention time (RRT) ratio of the suspect peak compared to the internal standard as compared to the RRT calculated from the calibration curve. The concentration of the compound will be calculated by comparing the relative RFs calculated from the calibration curve and the peak area of the compound using the following equation:

Water

Equation 8

$$C_s = \frac{(A_s)(C_s)(D)}{(A_{is})(RF)(V_s)}$$

Where:

Cw = Concentration of the compound in the sample (µg/L);

As = Compound peak area;

Cs = Standard (µg);

Ais = Internal standard peak area;

Vs = Volume of water extracted (L);

D = Dilution factor; and

RF = Compound response factor calculated from the following equation:

Equation 9

$$RF = \frac{(A_s)(C_i)}{(A_i)(C_s)}$$

Where:

A*s* = Compound response measured in area counts from the calibration curve;

A*i* = Internal standard response measured in area counts from the calibration curve;

C*i* = Internal standard concentration; and

C*s* = Compound concentration from the calibration curve.

Soil

Equation 10

$$C_s = \frac{(A_s)(C_s)(D)}{(Ai)(RF)(W_s)(M)}$$

Where:

C*s* = Compound concentration in the soil sample (µg/g);

A*s* = Compound response measured in area counts;

C*s* = Standard concentration (µg);

A*i* = Standard response measured in area counts;

W*s* = Mass of sample extracted (g);

D = Dilution factor;

M = Percent dry weight of sample/100; and

RF = Compound response factor calculated from the calibration curve using the same equation as that used for water samples.

Atomic Absorption Spectrophotometry Results—Photometric absorbance is governed by the relationship presented in Equation 11.

Equation 11

Absorbance=log 100/%T=2-log%T

Where:

%T = 100 - % absorption.

Percent absorption is based on the amount of light of a particular wavelength absorbed by a specific metal. Its calculation is based on the loss of light after a beam of light of a particular wavelength is passed through a flame into which a solution containing metals of interest has been aspirated.

Calibration curves establishing the absorbance relationship with concentration will be generated at various concentrations. From these curves, a comparison will be made with absorbance from sample measurement. Since absorbance is directly related to concentration, a plot of the two parameters will be linear within operable ranges and will allow for determination of unknown concentrations in solutions (direct samples or extracts) after measurement of absorbance.

Concentrations of contaminants in extracts will be calculated from instrumental responses of the extracts applied to the instrument calibration curve. The resultant concentration will then be modified by applying the appropriate dilution/concentration and sample weight or volume to obtain a final reportable concentration in the original matrix. In general, solid samples will be reported in units of µg/kg or mg/kg and aqueous results will be reported in units of µg/L or mg/L. The units used for a specific project will depend on the methods of analysis, etc.

When samples are diluted into a performance-demonstrated range, the reported concentration will contain one less significant digit than an undiluted sample. Values less than the certified RL will be reported as “less than” the RL. If a sample is diluted, the non-detected results will be reported as “less than” the RL multiplied by the dilution factor to reflect more accurately the observable limit. The dilution factor will be reported with the data.

10.5 Data Validation

10.5.1 Data Review, Validation, and Verification Requirements

The QA Officer will ensure all data are reviewed and verified, and decision-making data are validated in accordance with this section. Upon completion of the data collection activities, the QA Officer will ensure all data are assessed regarding the following, and provide a report to the PM:

- Sampling design – What deviations were observed from the project controlling document (PCDs) in terms of numbers of samples collected, locations of sample collection points, and unexpected events or observations in the field. An assessment will be provided of the impact of such deviations on the usability or interpretation of the results;

- Sampling procedures – What deviations were observed from the PCDs in terms of the method of work applied in the collection of the samples. An assessment will be provided of the impact of such deviations on the usability or interpretation of the results;

- Sample handling – What deviations were observed from the PCDs in terms of the handling and custody of the samples, including containers, preservation, storage, etc. This will include an assessment of potential sample alias problems. An assessment will be provided of the impact of such deviations on the usability or interpretation of the results;

- Analytical procedures – What deviations were observed from the PCDs in terms of the method of work applied in the analysis of the samples. An assessment will be provided of the impact of such deviations on the usability or interpretation of the results;

- QC and calibration – What deviations were observed from the PCDs in terms of conformance to QC and calibration criteria. An assessment will be provided of the impact of such deviations on the usability or interpretation of the results. This assessment will be provided in the form of data validation reports as defined in Section 9.5.2; and

- Data Reduction and processing – What deviations were observed from the PCDs in terms of data reduction and processing specifications. An assessment will be provided of the impact of such deviations on the usability or interpretation of the results.

10.5.2 Validation and Verification Methods

Definitive data will be validated in accordance with USEPA *Region III Modifications to the Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis*, April 1993, and USEPA *Region III Modifications to the USEPA National Functional Guidelines (NFGs) for Organic Data Review Multi-media, Multi-concentration* (OLM01.0-OLM01.9), September 1994, or the appropriate guidance in effect at the time of investigation as modified for the methods of analysis employed. An independent third party will provide data validation services. Ms. Jeanne Peterson, Senior Chemist, AQA will manage data validation and verification for this project. Verification for organic data will be performed at Manual Level M3 and the verification for inorganic data will be performed at Manual Level IM2. Particular emphasis will be placed on holding time compliance, equipment calibration, spike recoveries, and blank results, although each required element of the verification process will be considered. Table 10-1 provides the acceptance criteria which will be applied as applicable to decision making data validation for each method.

The AQA independent reviewer as part of the validation process will fill out a checklist based on the method/protocol. This checklist will document the checks performed and the observations of the reviewer. To the fullest extent possible, the validator will work with the laboratory to resolve anomalies encountered. The validator will then apply data qualifying flags to the data summaries provided by the laboratory and compile a report on each laboratory report. These reports will consist of a summary of the findings, copies of data summaries with data qualifying flags applied (as necessary), a copy of the data validation checklist, and supporting

documentation. The reports will be included in the project documentation submitted following completion of the Interim Measures.

Laboratory deliverable packages for definitive data will be equivalent to USEPA CLP deliverable packages, containing complete QC summary reports, QA documentation, and a complete raw data package.

For first round analytical data and for screening data, an independent review of data packages will be performed to ensure compliance with specified analytical, QC, and data reduction procedures; data reporting requirements; and required accuracy, precision, and completeness measures. At a minimum, the following items will be reviewed to validate the data when applicable:

- Sample custody documents;
- QC data summaries; and
- Reasonableness of analytical results.

10.5.3 Reconciliation with Data Quality Objectives

The QA Officer in association with the PM will provide an assessment of the conformance of data gathered in the course of these activities to objectives of the work. Data determined via the verification and validation processes to be unsuitable for use (i.e., data impacted by unacceptable deviations from plans and protocols and data found to be qualified as unreliable during the validation process) will be clearly identified and excluded from use in downstream decision-making. If, in the judgment of the PM and the technical data users, insufficient data remain for purposes of the work, additional sample collection and analysis may be performed. Data description and assessment tools as described in USEPA QA/G-9, *Guidance for Data Quality Assessment* (USEPA 1996), may be employed in the course of this reconciliation.

10.6 Blank Contamination Assessment

Blank contamination assessment will be performed to evaluate the impact of field sampling and laboratory analysis environments on data quality. Field and laboratory QC blanks will be collected and processed at the frequency specified in Tables 8-1 and 8-2.

Field and laboratory QC blank data will be reviewed in accordance with the NFGs for Organic and Inorganic Data Review (USEPA 1994a), USEPA Region III modifications to the NFGs (USEPA 1993c; 1994b), and USEPA Region III Innovative Approaches to Data Validation (USEPA 1995).

10.6.1 Field Blanks

Equipment Rinse Blanks - The integrity of decontamination events and sample cross-contamination will be evaluated by the rinse blank. The rinse blank will be collected at the beginning of the project when equipment is first decontaminated, and thereafter at a frequency of 5%.

Trip Blanks - Potential contamination during sample collection and shipment, and in the laboratory, will be assessed through the evaluation of trip blanks for volatile contamination. Volatile contaminants detected in a trip blank and the associated samples (associated during collection and shipping) will be flagged.

10.6.2 Laboratory Blanks

Method Blanks - Method blanks will be used to evaluate for potential contamination from the laboratory environment and analytical method used to process the sample. Method blanks will be processed at the beginning and at a frequency of 10% for each analytical run by the laboratory. The blanks will be used to evaluate whether the internal laboratory environment, reagents used during analyses, analytical techniques, or the instrumentation system is sources of contamination that could affect the integrity of the sample.

The criterion for the evaluation of blank contamination applies to blanks associated with the samples, and states that no contamination should be in the blank. If contamination is detected, data associated with the blank will be carefully evaluated to identify if there is an inherent variability in the data for the lot, or if the problem is an isolated occurrence not affecting each sample in the lot.

Examples of USEPA criteria by which the blanks will be reviewed include:

Inorganic:

- Analytes detected in the environmental sample at less than five times the concentration in the associated blank will be qualified "B."

Organic:

- The sample result is qualified "B," when the compound concentration is greater than the RL but less than ten times the amount detected in the associated blank for common laboratory contaminants, (i.e., methylene chloride, acetone, 2-butanone, and common phthalate esters); and
- The sample result for other contaminants are qualified "B," when the sample concentration is greater than the RL but less than five times the amount detected in the associated blank.

In cases where more than one blank is associated with a given sample, qualification will be based upon a comparison with the associated blank having the highest concentration of the contaminant. The mean concentrations and standard deviation will be provided as a reference point. Blank qualification will be added to the data validation and the contractor PM will assess data usefulness based on the project DQOs. The PM will make project decisions (use qualified data, re-sample, re-analyze) based upon the analytical limitations of the data. Contamination assessment results will be presented in the Site Investigation Report.

10.7 Reporting

Data for entry into the project data set will consist of field data and sampling/analytical data. In general, field data will consist of location, well construction and field measurement data generated from field logbooks, boring logs, and field parameter forms used by the UXB-KEMRON team. The original records/data will be retained in the project record, and will be summarized in project reports.

Sampling and analytical data will consist of dates, times and laboratory sample and QC results taken from chain-of-custody records and the laboratory deliverable verified against hardcopy laboratory reports. Laboratory data will be submitted in summary form in project reports, and electronic versions of the complete laboratory analytical report will be included in the reports as well. The chemical data also will be retained by the Army in its ERIS electronic data system.

10.7.1 Field Data Deliverables

Raw data from field measurements and sample collection will be reviewed by UXB-KEMRON and retained in project files. Data relevant to the site-specific objectives will be tabulated and included in reports submitted for regulatory review. Field data from sampling events will be included in reports submitted for regulatory review. These data will be recorded on field data sheets that are 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.

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 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.

10.8 Documentation and Records

Bound logbooks will be used for record keeping purposes both in the field and in the laboratory with the exception of certain standard forms, which will be maintained in three ring binders. Logbooks and binders will contain a unique document control number. Pages, including loose-leaf forms, will be numbered.

Field and laboratory personnel will transmit the bound logbooks to the Field Supervisor or Laboratory QA Manager (or their designees) on a routine basis. The Field or Laboratory QA Manager, as applicable, will review original logbooks at a frequency of at least once every week, and will sign the logbook as proof of said review.

To ease data review, the person making an entry must sign and date the entry. Entries must be recorded in ink or other permanent-marking device. Drawing a line through the incorrect entry, recording the correct information, and initialing and dating the corrected entry will make correction to entries. If the reason for making the change is not immediately evident, an explanation is required. Unused portions of logbook pages must be lined out.

If computerized information is used, a hard copy that has been permanently affixed to the logbook will be acceptable as an original record of sampling and/or laboratory logging.

10.8.1 Field Records

Field records, including sample collection records, chain-of-custody records, etc. will be maintained according to the SOPs applicable to the program. At a minimum, field personnel will keep a personal log of activities, noting conditions that in their judgment may bear on the on the use or interpretation of the data they acquire.

10.8.2 Laboratory Records

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 work order 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.

UXB-KEMRON will provide electronic (CD-ROM) copies of Level 4 reports generated as part of the site-specific scope of work within the final reports submitted to the Administrative Record.

10.8.3 Laboratory Deliverables

Microbac provides four levels of laboratory reports in order to match the documentation level required by specific projects. The laboratory will provide Level 4 CLP-like data packages for all decision making data for the RFAAP sites. 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, LODs and LOQs, data tags/qualifiers, percent solids, sample weight or volume, dilution factor, laboratory batch and sample number, UXB-KEMRON 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 Officer. 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.

Table 10-2 summarizes the content of a Level 4 data package. The complete Level 4 analytical package, including raw data, will be provided electronically (scanned onto a CD) as a .pdf file. The Microbac Laboratory Information Management System (LIMS) will use the project-specific data qualifiers established by this QAPP.

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 10–1
Scheduled QC Samples, Criteria, and Corrective Action

Procedure	Frequency	Acceptance Criteria			Corrective Action
VOCs by SW-846 8260B					
Initial Calibration 5-pt curve	Prior to sample analysis Set-up, major maintenance, and quarterly	RRF > 0.05 RSD ≤ 30% (up to two compounds may be >30% if ≤40%). Lab may use first or higher order regression fit (r ≥ 0.995) if %RSD > 15%.			If RSD of the average RRF for calibration check compounds > 30%, the initial calibration must be repeated. Data reviewer should review and judge all of the target compounds against the acceptance criteria.
Continuing calibration check	Daily before sample analysis, and every 12 hours	%Difference for RF all continuing calibration compounds ±25% from initial calibration average. RRF > 0.05. For first or higher order regressions, %D for recovered concentration must be ±25% of true concentration.			Samples cannot begin until this criterion is met. Data reviewer should review and judge all of the target compounds against the acceptance criteria.
Method blanks	Every 12 hours	No analytes detected ≥ RL.			Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank. Document source of contamination.
Tuning BFB	Prior to calibration	Must meet tuning criteria provided in Innovative Approaches to Data Validation (EPA Region III, June 1995).			Re-tune, re-calibrate.
LCS	Every batch	Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			Qualify associated data biased high or biased low as appropriate.
Internal Standards	Every sample	<u>Standards</u> Fluorobenzene Chlorobenzene-D5 1,4-Dichlorobenzene-D	Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)		Inspect for malfunction. Demonstrate that system is functioning properly. Reanalyze samples with standards outside criteria.
Surrogate	Every Sample	<u>Standards</u> Dibromofluoromethane Toluene-D8 4-Bromofluorobenzene 1,2-DCA-D4	<u>Solid</u> ** 84-138 59-113 70-121	<u>Aqueous</u> ** 88-110 86-115 76-114	If any surrogate compounds do not meet criteria, there should be a re-analysis to confirm that the non-compliance is due to the sample matrix effects rather than laboratory deficiencies.
Matrix Spike and Duplicate	1 per 20 per matrix	Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			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.

**lab limits derived per method SW846 8000 for compounds not listed.

Table 10–1
Scheduled QC Samples, Criteria, and Corrective Action

Procedure	Frequency	Acceptance Criteria			Corrective Action
SVOCs by SW8270C					
Initial calibration curve (5-pt curve)	Set-up, major maintenance	RRF > 0.05 RSD ≤ 30% (up to two compounds may be >30% if ≤40%). Lab may use first or higher order regression fit (r ≥ 0.995) if %RSD > 15%.			Must meet criteria prior to sample analysis. Data review evaluates target compounds against the acceptance criteria.
Continuing calibration standard	12 hours	%Difference for RF all continuing calibration compounds ±25% from initial calibration average. RRF > 0.05. For first or higher order regressions, %D for recovered concentration must be ±25% of true concentration.			If criteria not met, reanalyze the daily standard. If daily standard fails a second time, repeat calibration. Review target compounds against the acceptance criteria.
Internal standards	Every sample	1,4-Dichlorobenzene-D4, Naphthalene-D8, Acenapthene-D10, Phenanthrene-D10 Chrysene-D12, Perylene-D12; Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)			Inspect for malfunction. Demonstrate that system is functioning properly. Reanalyze samples with standards outside criteria.
Tuning DFTPP	12 hours	Must meet tuning criteria provided in Innovative Approaches to Data Validation (EPA Region III, June 1995).			Re-tune, re-calibrate.
Method blanks	Per extraction batch	No analytes detected ≥ RL			Document source of contamination.
LCS	Every batch	Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			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.
Surrogate spikes	Every Sample	Standards 2,4,6-Tribromophenol 2-Fluorobiphenyl 2-Fluorophenol Nitrobenzene-D5 Phenol-D5 Terphenyl-D14	Aqueous (%Rec.) 10-123 43-116 21-110 35-114 10-110 33-141	Solid (%Rec.) 19-122 30-115 25-121 23-120 24-113 18-137	If any two base/neutral or acid surrogates are out of specification, or if any one base/neutral or acid extractable surrogate has a recovery of less than 10%, then there should be a re-analysis to confirm that the non-compliance is due to sample matrix effects rather than laboratory deficiencies.
Matrix spike and duplicate	1 per 20 samples per matrix	Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			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.

Table 10–1
Scheduled QC Samples, Criteria, and Corrective Action

Procedure	Frequency	Acceptance Criteria			Corrective Action
PAHs by SW8270C SIM					
Initial calibration curve (5-pt curve)	Set-up, major maintenance	RRF > 0.05 RSD ≤ 30% (up to two compounds may be >30% if ≤40%). Lab may use first or higher order regression fit (r ≥ 0.995) if %RSD > 15%. The peak for each ion must not be cut off when switching ions.			Must meet criteria prior to sample analysis. Data review evaluates target compounds against the acceptance criteria.
Continuing calibration standard	12 hours	%Difference for RF all continuing calibration compounds ±25% from initial calibration average. RRF > 0.05. For first or higher order regressions, %D for recovered concentration must be ±25% of true concentration.			If criteria not met, reanalyze the daily standard. If daily standard fails a second time, repeat calibration. Review target compounds against the acceptance criteria.
Internal standards	Every sample	1,4-Dichlorobenzene-D4, Naphthalene-D8, Acenapthene-D10, Phenanthrene-D10 Chrysene-D12, Perylene-D12; Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)			Inspect for malfunction. Demonstrate that system is functioning properly. Reanalyze samples with standards outside criteria.
Tuning DFTPP	12 hours	Must meet tuning criteria provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); tailing factors and breakdown not required.			Re-tune, re-calibrate.
Method blanks	Per extraction batch	No analytes detected ≥ RL			Document source of contamination.
LCS	Every batch	Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			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.
Surrogate spikes	Every Sample	Standards 2-Fluorobiphenyl Nitrobenzene-D5 Terphenyl-D14	Aqueous (%Rec.) 43-116 35-114 33-141	Solid (%Rec.) 30-115 23-120 18-137	If any two base/neutral are out of specification, or if any one base/neutral extractable surrogate has a recovery of less than 10%, then there should be a re-analysis to confirm that the non-compliance is due to sample matrix effects rather than laboratory deficiencies.
Matrix spike and duplicate	1 per 20 samples per matrix	All target analytes spiked. Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			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.

**lab limits derived per method SW846 8000

Table 10–1
Scheduled QC Samples, Criteria, and Corrective Action

Procedure	Frequency	Acceptance Criteria			Corrective Action
Organochlorine Pesticides by SW-846 8081A					
Initial calibration curve 5-pt curve	Set-up, major maintenance	%RSD≤20% of the response factor from the initial curve (surrogates ≤30%), (up to two %RSDs may exceed 20% if they are ≤30%).			Must meet criteria prior to sample analysis
Continuing calibration verification (calibration check)	Daily before sample analysis, and after every 10 samples and at the end of the analysis sequence	All analytes within ±25% of expected value			If criteria are not met, reanalyze the daily standard. If the daily standard fails a second time, initial calibration must be repeated.
Resolution Check Mixture	Daily prior to analysis of samples	Contains gamma-chlordane; endosulfan I; 4,4'-DDE; dieldrin; endosulfan sulfate; endrin ketone; methoxychlor; tetrachloro;m;xylene; decachlorobiphenyl. The depth of the valley between two adjacent peaks must be ≥60%.			Perform maintenance; repeat resolution check mixture
Breakdown check (Endrin and DDT)	Daily prior to analysis of samples	Contains gamma-BHC; alpha-BHC; 4,4'-DDT; beta-BHC; endrin; methoxychlor; tetrachloro;m;xylene; decachlorobiphenyl. DDT and endrin degradation ≤20% each for both columns; ≤30% combined for both columns. The depth of the valley between two adjacent peaks must be ≥60%. Absolute RTs must be within RT windows. RPDs between true values and calculated values must be ≤25%.			Repeat breakdown check
Independent reference standard (LCS)	1 per batch	Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			Qualify associated data biased high or biased low as appropriate.
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.
Surrogate spikes	Every sample	<u>Standards</u> TCMX DCB	<u>Aqueous</u> <u>%R</u> 60-150 60-150	<u>Solid</u> <u>%R</u> 60-150 60-150	If any surrogate compounds do not meet criteria, there should be a re-analysis to confirm that the non-compliance is due to the sample matrix effects rather than laboratory deficiencies.
Matrix spike and matrix spike duplicate	1 per 20 samples per matrix	Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			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.

Table 10–1
Scheduled QC Samples, Criteria, and Corrective Action

Procedure	Frequency of QC Procedure	Acceptance Criteria			Corrective Action
PCBs by SW-846 8082					
Initial calibration curve 5-pt curve	Prior to sample analysis Set-up, major maintenance	%RSD≤20% of the response factor from the initial curve (surrogates ≤30%), (up to two %RSDs may exceed 20% if they are ≤30%).			Must meet criteria prior to sample analysis
Continuing calibration (calibration check)	Daily before sample analysis, and After every 10 samples and at the end of the analysis sequence	All analytes within ±25% of expected value			If criteria are not met, reanalyze the daily standard. If the daily standard fails a second time, initial calibration must be repeated.
Independent reference standard (LCS)	1 per batch	Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			Qualify associated data biased high or biased low as appropriate.
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.
Surrogate spikes	Every sample	Standards TCMX DCB	Aqueous %R 60-150 60-150	Solid %R 60-150 60-150	If any surrogate compounds do not meet criteria, there should be a re-analysis to confirm that the non-compliance is due to the sample matrix effects rather than laboratory deficiencies.
Matrix spike and matrix spike duplicate (1016/1260 mix)	1 per 20 samples per matrix	Specified QC limits provided in Innovative Approaches to Data Validation (EPA Region III, June 1995); lab limits derived per method SW846 8000 for compounds not listed.			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.

Table 10–1
Scheduled QC Samples, Criteria, and Corrective Action

Procedure	Frequency of QC Procedure	Acceptance Criteria	Corrective Action
Metals by SW-846 6010B/6020/7470A/7471A			
Initial calibration curve (blank and at least four stds for Hg) (blank and at least one std for ICP)	Daily or major maintenance, instrument modification, replacement of the torch, replacement of the mirror	r > 0.995 for all elements r: linear correlation coefficient	If r < 0.995 for any element, the standards for that element must be prepared again and/or the lower/upper range standard must be used.
Continuing calibration verification (CCV)	Every 10 samples or 2 per 8 hr and end of run.	Recovery ±10% of true value for ICP Recovery ±20% of true value for Hg	Reanalyze CCV. If the CCV fails second time, the terminate analysis, correct problem, re-calibrate instrument, and re-verify calibration prior to continuing sample analyses.
CRDL (CRI for ICP and CRA for Hg)	CRI at 2X CRDL every 10 samples or 2 per 8 hr and end of run, whichever is more frequent. CRA at CRDL at beginning of run.	90-110% of true values.	Perform maintenance; reanalyze.
Interference check	Beginning and end of each sample analytical run or 2 per 8 hr, whichever is more frequent.	Recovery ±20% of true value. For non-spiked analytes, absolute value <IDL.	Terminate the analysis, correct the problem, re-calibrate, re-verify the calibration, and reanalyze the samples.
Continuing calibration blank (CCB)	Every 10 samples, end of analytical run	No target analytes >RL.	If a target analyte is present at a concentration > RL, re-calibrate, and reanalyze all samples analyzed since the last acceptable CCB.
Serial Dilution (ICP)	1 per 20 samples per matrix for samples > 50x IDL	Difference between diluted and undiluted sample <10%.	Chemical or physical interference should be suspected. Investigate to determine cause.
Preparation blank	1 per batch per matrix	No target analytes.	Documented source of contamination.
Laboratory Control Sample	1 per analytical batch	80%≤%Rec.≤120% (aqueous) EPA control limits (solid)	Qualify associated data biased high or biased low as appropriate.
Duplicate Sample	1 per batch per matrix	RPD within ±20% (35% for soil) for sample values >5X the CRDL. ±CRDL (2X CRDL for soil) for sample values <5X the CRDL.	
Matrix spike and duplicate and sample duplicate	1 per analytical batch	75%≤%Rec.≤125%; %RPD<20%; If spike(s) outside of limits, analyze PDS. Spike limits do not apply if sample concentration is >4X the spike amount.	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.

Waste characterization data do not require Level 4 analytical reports.

Table 10-2 Laboratory Report Levels Radford Army Ammunition Plant, Radford, Virginia					
	ICP/6010/6020 Deliverables	**Level 1**	**Level 2**	**Level 3**	**Level 4**
1	Cover Page	x	x	x	x
2	Summary data sheet	x	x	x	x
3	Initial Calibration			x	x
4	ICV/CCV form			x	x
5	CRQL Check Standard			x	x
6	Blanks - ICB/CCB			x	x
7	Prep Blank		x	x	x
8	ICP Interference check			x	x
9	ICP/MS Interference check			x	x
10	Matrix Spike Recovery		x	x	x
11	Post Digestion Spike			x[1]	x[1]
12	Duplicates		x	x	x
13	Laboratory Control Sample		x	x	x
14	Holding Times			x	x
15	ICP Serial Dilutions			x[1]	x[1]
16	Method Detection Limits			x	x
17	ICP-AES Interelement Corr. Fac.			x	x
18	ICP-AES Interelement Corr. Fac.			x	x
19	ICP Linear Ranges			x	x
20	Preparation Log			x	x
21	Analysis Run Log			x	x
22	ICP-MS Tune			x	x
23	ICP-MS Internal Standards			x	x
24	Raw Data Package				x
	AA/7000 Deliverables	**Level 1**	**Level 2**	**Level 3**	**Level 4**
1	Cover Page	x	x	x	x
2	Summary data sheet	x	x	x	x
3	Initial Calibration			x	x
4	ICV/CCV form			x	x
5	CRQL Check Standard			x	x
6	Blanks - ICB/CCB			x	x
7	Prep Blank		x	x	x
8	Matrix Spike Recovery		x	x	x
9	Post Digestion Spike			x[1]	x[1]
10	Duplicates		x	x	x
11	Laboratory Control Sample		x	x	x
12	Holding Times			x	x
13	Method Detection Limits			x	x
14	Preparation Log			x	x
15	Analysis Run Log			x	x
16	Raw Data Package				X
	GC/MS Deliverables	**Level 1**	**Level 2**	**Level 3**	**Level 4**
	8260,8270				
1	Cover Page	x	x	x	X
2	Analysis Data Sheet - Form 1	x	x	x	X

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

X item provided at that laboratory report level.

1 Item to be provided if method requires that this step be performed. ICP Serial Dilutions and Post Digestion Spikes are performed on an as needed basis.

2 Item provided only when specified in remedial action or IMWP; TICs do not apply to this project.

10.8.4 Record Storage and Handling

Records will be designated as “lifetime” or “nonpermanent” prior to temporary or final storage. Nonpermanent records will be retained for three years after the completion of the fieldwork, or three years after the date the record was generated, whichever is longer. A lifetime record will be delivered to the client for retention.
Records of either type will be catalogued prior to shipment to either the client or a storage facility. The storage facility will confirm that the received parcels contain the catalogued records and convey a receipt for the records to the originating office. The records will be made available to the originating office upon request and according to the procedures of the storage facility.

All data generated by the project are the property of the Army and will be delivered for project closeout as specified by the COR. Data, reports and other relevant records are submitted by UXB-KEMRON to the Army for permanent retention by the Army if/as necessary.

10.9 Data Management

Data management will begin when the contractor transmits a request for analytical services to the laboratory, stating the number, type, sample numbers, methods for analysis, and other information necessary for the laboratory to plan a particular job. Data fields of initial input information, including map location files, a certification status check, sample identification number, parameters, dates, etc., will be established as sample containers and chain of custody documentation are prepared for shipment to the sampling team.

While in the process of collecting, documenting, packaging, and shipping samples to the laboratory, the field sampling team will transfer sample data from their notebooks to field parameter forms. Once the samples arrive at the laboratory, this information will be used to create data fields for submission to the data management staff. Status information (e.g., date sampled, date received, data extraction/analysis due) will form a part of the record.

Each step in the analytical process will result in updates to the data files. The operation performed (e.g., preparation, extraction, analysis, data review, data package prepared), the data obtained and the data that each step was completed will be entered into the system and made available for status checks. The laboratory will validate the data, perform error checking and correction, and transmit the files to the contractor, who will also perform the checks. Hard-copy documentation will also be transferred from the laboratory to the contractor.

The laboratory will archive copies of the analytical data, including original instrument magnetic tapes, for a period specified by the contract. Records will also be maintained so that historical summaries of the analyses may be generated by site, by client, or by sample type. ERIS EDDs of chemical data will be uploaded by UXB-KEMRON into ERIS for future Army access and Army record retention. Report will be submitted in Army READ format as well.

SECTION 11. CORRECTIVE ACTION

Corrective or preventive action is required when potential or existing conditions are identified that may adversely impact data quantity or quality. Corrective action could be immediate or long term.

While the specific activities in Table 11-1 may identify a need for corrective action, any member of the project team who identifies a condition adversely affecting quality is responsible for initiating corrective action by notifying his/her supervisor and the PM or Project QA Officer. Whether informally or formally identified, notification of issues resulting in the potential for corrective action will specify the condition and explain how it may affect data quality or quantity.

Table 11-1
Assessment Type and Corrective Action Responses
Radford Army Ammunition Plant, Radford, Virginia

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation[1]	Individual(s) Receiving Corrective Action Response (Name, Title, Organization)	Timeframe for Response
Readiness Review	Written Summary of Findings	FS, QA Manager, PM, UXB-KEMRON	Within 24 hours after review	Memorandum	FS, QA Manager, PM, UXB-KEMRON	Within 24 hours after notification of deficiency requiring CA
Field Sampling Technical Systems Audit	Written Audit Report	FS, QA Manager, PM, UXB-KEMRON	5 business days after audit	Memorandum	FS, QA Manager, PM, UXB-KEMRON	Within 24 hours after notification of significant deficiencies
Data Review and Validation	Data Validation Report and Usability Assessment	Data Validator, AQA	Within 15 business days from receipt of analytical report	CA Report from Analytical Laboratory	AQA Data Validator; FS, QA Manager, UXB-KEMRON	3 business days from identification of deficiency affecting data

Immediate corrective action is applied to spontaneous, nonrecurring problems (e.g., instrument malfunction). The individual who detects or suspects nonconformance to established criteria or protocol in equipment, instruments, data, or methods, will immediately notify his/her supervisor. The supervisor and/or the Task Manager will investigate the extent of the problem and take necessary corrective action. If a large quantity of data is affected, the Field Supervisor will prepare a memorandum to the Project Manager and Project QA Manager. These individuals will collectively decide how to proceed to correct the problem(s). If the problem is limited in scope, the Task Manager will decide on the corrective action measure and document the solution in memorandum form to the PM and QA Officer.

Long-term corrective action procedures are devised and implemented to prevent the recurrence of a potentially serious problem. The Project QA Officer will be notified of the problem and will conduct an investigation to determine the severity and extent of the problem. He will notify the Field Supervisor and Project Manager of the need for corrective action. If the corrective action will adversely impact project schedule, the Project Manager will notify and coordinate with the Army and regulatory personnel.

The development and implementation of preventive and corrective actions will be timed, to the extent possible, so as to not adversely impact project schedules or subsequent data generation/processing activities. Examples of long-term corrective actions include but are not limited to staff training/retraining in technical skills or in implementing the QA Program,

rescheduling of laboratory routine to ensure analysis within allowed holding times, revision of UXB-KEMRON QA Program, or replacement of project personnel.

Corrective actions will be categorized as either routine or non-routine and will require short-term or long-term action. Both types will require administrative coordination between the person initiating the corrective action and the QA staff. Examples of UXB-KEMRON and analytical laboratory corrective action forms are provided in Figures 13-1, 13-2, and 13-3.

11.1 Corrective Action Documentation

The UXB-KEMRON QA Officer will ensure that incidents requiring corrective action are fully documented. Reports will be addressed to the Project Manager as well as the Field Supervisor. The summary of findings will be factual, concise, and complete. Any required supporting information will be appended to the report or cross-referenced to readily available project documents. Depending on the nature of the problem, the corrective action employed may be formal or informal.

Corrective actions reports applicable to the analytical laboratory will be documented in corrective action reports provided within 3 business days, as described in Table 11-1.

Whether requiring formal or informal corrective action, documentation will include the occurrence of the problem, corrective action employed, and verification that the problem has been eliminated. Final resolution of a problem will be documented by the signature of the Project QA Officer, or his/her designee. The QA Officer or his/her designee will sign completed corrective action documentation, verifying that the problem has been resolved.

Significant QC issues are those which require a change to an approved document or procedure, including either field or laboratory procedures, and/or which affect data usability. In cases of significant QC issues, a corrective action report will be completed by the Project QA Officer, reviewed by the Project Manager, and coordinated through the Army and regulatory personnel and/or proposed in an addendum or revision to the FSP/QAPP. For items which are not significant, reports will be issued from the Project Manager to the Project QA Officer and maintained in project records.

11.2 Stop Work Protocols

The contractor Program Manager, PM, and QA Officer have the authority to issue a stop work order. A stop work order will be issued under conditions such that the quality of work jeopardizes the attainment of the project objectives. A stop work order must not create an operational, safety, public health, or environmental hazard.

Under a stop work order, work may not be conducted within affected activities until the responsible manager acknowledges the implementation of a corrective action in accordance with the resolution criteria of the order. Immediate notification of work stoppage will be made to the USACE COR and RFAAP.

SECTION 12. QUALITY ASSESSMENTS

This section discusses the inspection program used to monitor the total measurement system and to evaluate the quality of operation in the field and at the on-site laboratory. A performance inspection is a planned independent check of the operation of a system to obtain a quantitative measure of the quality of data generated, and involves the use of standard reference samples or materials which are certified as to their chemical composition or physical characteristics. Systems inspection is of a qualitative nature and consists of on-site review of a system's quality assurance system and physical facilities for sampling/analysis, calibration, and measurement.

12.1 Document Review

Project plans will be reviewed and approved prior to implementation. The contractor PM and QA Manager will provide a qualitative self-evaluation for establishing whether the prevailing management structure, polices, practices, and procedures are adequate to ensuring that the results needed are obtained. The PM will provide an independent qualitative evaluation of a particular program operation and/or organization to establish whether the prevailing management structure, policies, practices, and procedures are adequate for ensuring that the results needed are obtained.

12.1.1 Document Control

The goal of a Document Control Program is to ensure that the project documents issued or generated will be accounted for upon completion of the project. The program includes a numerical document inventory procedure and a central filing system with a designated person(s) responsible for its maintenance. Documents used or generated during the course of the project are accounted for and become a part of the project files upon completion of the task. These may include, but are not limited to, the following:

- Project deliverables;
- LTM requirements;
- Reports and correspondence material; and
- Contract documents.

For example, QAPP will contain a control footer that includes:

- Document title;
- Document version; and
- Effective date (month year).

A distribution list of controlled documents will be maintained within the document control system. This system will ensure that revisions are distributed to the addressees. After technical work on a task has been completed, the accountable documents generated or used for the task work will be assembled and placed in a secure storage location. The QA Officer or his/her designee will inventory accountable task documentation.

12.2 Readiness Reviews

Documented readiness reviews may be performed by the contracting QA Officer at the beginning of the work schedule start date and in the event of a quality-related stop work order. The readiness review will be performed to verify the following elements:

1. Work plans are approved;
2. Personnel have been suitably trained and qualified; and
3. The proper resources are available.

Work prerequisites for investigation activities include ensuring that necessary permits and licenses have been obtained. The contractor will be responsible for site approvals and preparation, coordinating with RFAAP for the extension of utilities to the study site, and regulatory compliance (i.e., obtaining necessary permits to install monitoring wells). Once site preparation is complete and permits are obtained, the contractor will be responsible for monitoring these facilities and determining compliance with permit requirements.

During the readiness review, actions will be taken as necessary by the contracting QA Officer to ensure that field activities are conducted in accordance with the QAPP. The QA Officer will document deficiencies encountered during the readiness review and actions taken in the field to correct potential problems. Results of readiness reviews and corrective actions will be presented as a memorandum issued to the contracting PM. The memorandum will define deficiencies noted during the inspection and will note the actions taken to meet the QA requirements as defined by this QAPP.

12.3 Field Performance Audits

The contractor's technical audit team will perform a field audit of site activities. During this audit, current field practices will be compared to procedures outlined in the project work plans (i.e., WP, QAP, and other pertinent, industry acceptable, RFAAP-approved standards). The following elements will be evaluated during field activities at RFAAP:

- The overall level of organization and professionalism;
- Project activities;
- Document control and management;
- Level of QC conducted per each field team; and
- Task specific activities.

After audit completion, deficiencies will be discussed with the PM, and corrections will be identified. Corrective action procedures are outlined in Section 11.0.

12.4 Laboratory Performance Audits

Microbac actively participates in several Federal and State performance evaluation testing programs and system audits. External systems audits of the Microbac facility occur on a regular basis. These include audits by the West Virginia Department of Natural Resources (WVDNR), the Ohio Environmental Protection Agency (OEPA), and the Florida Department of Health (FLDOH) (NELAC Primary Accreditor). Additionally, audits are conducted every 2 - 3 years by the California Department of Health Services, and the US Army Corps of Engineers. UXB-KEMRON's QA Manager will be provided copies of the Microbac external audit results during the term of this contract.

Additionally, both Microbac OVD and Columbia Analytical Services have been accredited by the DoD Environmental Laboratory Accreditation Program (DoD ELAP). This program was established by the Assistant Deputy Under Secretary of Defense, and became effective October 01, 2009. Laboratories seeking the DoD ELAP certification undergo stringent audits and reviews. The program is a means for laboratories to demonstrate conformance to the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM). The DoD QSM is based on the National Environmental Laboratory Accreditation Conference Quality Systems standard, which provides guidelines for implementing ISO/IEC 17025, the international standard entitled *General Requirements for the Competence of Testing and Calibration Laboratories*. The DoD QSM is periodically revised, and laboratories are required to achieve conformance with new standards as revisions are issued and become effective. Use of laboratories that have achieved the stringent DoD ELAP certification provides additional assurance that project data will be of the highest quality

SECTION 13. REFERENCES

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FIGURES

FIGURE 10.4-a
EXAMPLE CHAIN-OF-CUSTODY FORM

Project Number	Project Name				Matrix	A	N	A	L	Y	S	E	S		LAB :
Project Contact (Name and Phone Number)															AIRBILL No:
Samplers:															Courier:
Field Sample No.	Date (MM-DD-YY)	Time	Comp	Grab										Subtotal	REMARKS
TOTAL															

Relinquished by:	Date/time	Received by:	Relinquished by:	Date/Time	Received by:
Relinquished by:	Date/time	Received by: (for lab)	Date/Time	Remarks	

Figure 13-1
GENERAL CORRECTIVE ACTION REPORT

KEMRON Environmental Services
General Corrective Action Report

Date:_____ **Department:**_____ **Employee:**_____

I. Description of Non-Conformance:

_____

_____

_____

_____

_____

II. Action Taken:

_____

_____

_____

_____

_____

_____

_____

III. Return to Control?:	Not Applicable _____	Yes _____	No _____

IV. Further Action Required?: (required if no checked in III.)

_____

_____

_____

_____

_____

Approvals:

Department Supervisor: _____ Date: _____
QA/QC Supervisor: _____ Date: _____
Director/Manager: _____ Date: _____

Form NC01

Figure 13-2
ANALYTICAL CORRECTIVE ACTION REPORT

KEMRON Environmental Services
Analytical Corrective Action Report

Date:_____ **Department:**_____ **Employee:**_____

Prep WG#_____ **Preparation Date:**_____ **Parameter:**_____

I. Description of Non-Conformance:

Sample ID(s) _____

☐ Surrogate(s) <10% _____

☐ Multiple Surrogate(s) _____

☐ Method Blank _____

☐ LCS Failures _____analytes out _____

☐ CCV Failures _____

☐ Other _____

II. Action Taken:

☐ Reprep affected samples only _____

☐ Reprep entire workgroup _____

☐ Reanalyze affected samples only _____

☐ Report data with qualifiers _____

☐ Other _____

III. Return to Control?:	Not Applicable	Yes _____	No _____

IV. Further Action Required?: (required if no checked in III.)

Approvals:
Department Supervisor: _____ Date: _____
QA/QC Supervisor: _____ Date: _____
Director/Manager: _____ Date: _____

Form NC02 2101

Figure 13-3
KEMRON PERFORMANCE AUDIT CORRECTIVE ACTION FORM

KEMRON PERFORMANCE AUDIT CORRECTIVE ACTION FORM

PE PROGRAM: _____ Login Number: _____

Analyte: _____ Method: _____

Analyst: _____ Instrument: _____

Acceptable Range

Result (Level 1): _____ _____

Result (Level 2): _____ _____

Reason for Error: _____

Corrective Action: _____

Supervisor Approval: _____ Date: _____

QAO Approval: _____ Date: _____

ATTACHMENT A

SWMU-57 Field Standard Operating Procedures (on CD)

10.1 - Field Log Book

10.2 – Surface Water, Groundwater and Soil/Sediment Field Log Books

10.3 – Boring Logs

10.4 – Chain-of-Custody Form

20.3 – Well and Boring Abandonment

30.1 – Soil Sampling

30.2 – Groundwater Sampling

30.3 – Surface Water Sampling

30.7 – Sampling Strategies

40.1 – Multi-parameter Water Quality Monitoring Instrument

40.2 – Water Level and Well-Depth Measurements

50.1 – Sample Labels

50.2 – Sample Packaging

70.1 – Investigation Derived Materials

80.1 – Decontamination

90.1 – Photo-ionization Detector (HNu Model PI-101 and HW-101)

STANDARD OPERATING PROCEDURE 10.1
FIELD LOGBOOK

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for recording daily site investigation activities.

Records should contain sufficient information so that anyone can reconstruct the sampling activity without relying on the collector's memory.

2.0 MATERIALS

- Field Logbook;
- Indelible ink pen; and
- Clear tape.

3.0 PROCEDURE

Information pertinent to site investigations will be recorded in a bound logbook. Each page/form will be consecutively numbered, dated, and signed. All entries will be made in indelible ink, and all corrections will consist of line out deletions that are initialed and dated. If only part of a page is used, the remainder of the page should have an "X" drawn across it. At a minimum, entries in the logbook will include but not be limited to the following:

- Project name (cover);
- Name and affiliation of personnel on site;
- Weather conditions;
- General description of the field activity;
- Sample location;
- Sample identification number;
- Time and date of sample collection;
- Specific sample attributes (e.g., sample collection depth flow conditions or matrix);
- Sampling methodology (grab or composite sample);
- Sample preservation, as applicable;
- Analytical request/methods;
- Associated quality assurance/quality control (QA/QC) samples;

- Field measurements/observations, as applicable; and
- Signature and date of personnel responsible for documentation.

4.0 MAINTENANCE

Not applicable.

5.0 PRECAUTIONS

None.

6.0 REFERENCES

USEPA. 1990. *Sampler's Guide to the Contract Laboratory Program.* EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, DC.

USEPA. 1991. *User's Guide to the Contract Laboratory Program.* EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response, January.

USEPA. 1998. *EPA Requirements for Quality Assurance Project Plans.* EPA/600/R-98/018, QA/R5, Final, Office of Research and Development, Washington, D.C.

STANDARD OPERATING PROCEDURE 10.2
SURFACE WATER, GROUNDWATER, AND SOIL/SEDIMENT FIELD LOGBOOKS

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for recording surface water, groundwater, and soil/sediment sampling information, as well as instrument calibration data in field logbooks.

2.0 MATERIAL

- Applicable field logbook (see attached forms); and
- Indelible ink pen.

3.0 PROCEDURE

All information pertinent to surface water, groundwater, or soil/sediment sampling will be recorded in the appropriate logbook. Each page/form of the logbook will be consecutively numbered. All entries will be made with an indelible ink pen. All corrections will consist of line out deletions that are initialed and dated.

3.1 SOIL/SEDIMENT

3.1.1 Field Parameters/Logbook (Form 10.2-a)

1. HIGH CONCENTRATION EXPECTED?: Answer "Yes" or "No.";
2. HIGH HAZARD?: Answer "Yes" or "No.";
3. INSTALLATION/SITE: Record the complete name of the installation or site;
4. AREA: Record the area designation of the sample site;
5. INST. NAME: Record the two-letter installation name for Radford Army Ammunition Plant – "RD";
6. SAMPLE MATRIX CODE: Record the appropriate sample matrix code. Common codes are "SD" for solid - sediment, "SI" for soil - gas, "SL for solid sludge, "SO" for surface other, "SS" for solid – soil, "SW" for surface wipe, "WD" for water – potable, "WG" for water – ground, "WS" water – surface, "WT" – water treated and "WW" water -waste;
7. SITE ID: Record a code up to 20 characters or numbers that is unique to the site;
8. ENV. FIELD SAMPLE IDENTIFIER: Record a code up to 20 characters specific for the sample;
9. DATE: Enter the date the sample was taken;
10. TIME: Enter the time (12-hour or 24-hour clock acceptable as long as internally consistent) the sample was taken;

11. AM PM: Circle "AM" or "PM" to designate morning or afternoon (12-hour clock);
12. SAMPLE PROG: Record "RFI" (RCRA Facility Investigation) or other appropriate sample program;
13. DEPTH (TOP): Record the total depth sampled;
14. DEPTH INTERVAL: Record the intervals at which the plug will be sampled;
15. UNITS: Record the units of depth (feet, meters);
16. SAMPLE MEASUREMENTS: Check the appropriate sampling method;
17. CHK: Check off each container released to a laboratory;
18. ANALYSIS: Record the type of analysis to be performed on each sample container;
19. SAMPLE CONTAINER: Record the sample container type and size;
20. NO.: Record the number of containers;
21. REMARKS: Record any remarks about the sample;
22. TOTAL NUMBER OF CONTAINERS FOR SAMPLE: Record the total number of containers;
23. SITE DESCRIPTION: Describe the location where the sample was collected;
24. SAMPLE FORM: Record the form of the sample (i.e., clay, loam, etc.) using The Unified Soil Classification System (USCS);
25. COLOR: Record the color of the sample as determined from standard Munsell Color Charts;
26. ODOR: Record the odor of the sample or "none";
27. PID: Record the measured PID values or other similar measurement instrument value;
28. UNUSUAL FEATURES: Record anything unusual about the site or sample;
29. WEATHER/TEMPERATURE: Record the weather and temperature; and
30. SAMPLER: Record your name.

3.1.2 Map File Form (refer to form 10.2-c)

1. SITE ID: Record the Site ID from the field parameter form;
2. POINTER: Record the field sample number for the sample being pointed to;
3. DESCRIPTION/MEASUREMENTS: Describe the location where the sample was taken, along with distances to landmarks;
4. SKETCH/DIMENSIONS: Diagram the surroundings and record the distances to landmarks;
5. MAP REFERENCE: Record which U.S.G.S. Quad Map references the site;
6. COORDINATE DEFINITION: Write the compass directions and the X- and Y-coordinates of the map run;
7. COORDINATE SYSTEM: Write "UTM" (Universal Transverse Mercator);
8. SOURCE: Record the 1-digit code representing the Map Reference;
9. ACCURACY: Give units (e.g., write "1-M" for 1 meter);
10. X-COORDINATE: Record the X-coordinate of the sample site location;
11. Y-COORDINATE: Record the Y-coordinate of the sample site location;

12. UNITS: Record the units used to measure the map sections;
13. ELEVATION REFERENCE: Record whether topography was determined from a map or a topographical survey;
14. ELEVATION SOURCE: Record the 1-digit code representing the elevation reference;
15. ACCURACY: Record the accuracy of the map or survey providing the topographical information;
16. ELEVATION: Record the elevation of the sampling site;
17. UNITS: Write the units in which the elevation is recorded; and
18. SAMPLER: Write your name.

3.2 SURFACE WATER

3.2.1 Field Parameter Logbook (Forms 10.2-b and 10.2-c)

1. CAL REF: Record the calibration reference for the pH meter;
2. pH: Record the pH of the sample;
3. TEMP: Record the temperature of the sample in degrees Celsius;
4. COND: Record the conductivity of the water;
5. Description of site and sample conditions (refer to 10.2-b);
6. Map File Form (refer to Section 3.1.2).

3.3 GROUNDWATER (FORMS 10.2- D)

3.3.1 Field Parameter Logbook (Form 10.2.b)

Refer to Section 3.2.1.

3.3.2 Map File and Purging Forms

1. WELL NO. OR ID: Record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on pages 18-21 of the IRDMIS User's Guide for chemical data entry;
2. SAMPLE NO.: Record the reference number of the sample;
3. WELL/SITE DESCRIPTION: Describe the location where the sample was taken, along with distances to landmarks;
4. X-COORD AND Y-COORD: Record the survey coordinates for the sampling site;
5. ELEV: Record the elevation where the sample was taken;
6. UNITS: Record the units the elevation was recorded in;
7. DATE: Record the date in the form MM/DD/YY;
8. TIME: Record the time, including a designation of AM or PM;
9. AIR TEMP.: Record the air temperature, including a designation of C or F (Celsius or Fahrenheit);
10. WELL DEPTH: Record the depth of the well in feet and inches;
11. CASING HEIGHT: Record the height of the casing in feet and inches;
12. WATER DEPTH: Record the depth (underground) of the water in feet and inches;

13. WELL DIAMETER: Record the diameter of the well in inches;
14. WATER COLUMN HEIGHT: Record the height of the water column in feet and inches;
15. SANDPACK DIAM.: Record the diameter of the sandpack. Generally, this will be the same as the bore diameter;
16. EQUIVALENT VOLUME OF STANDING WATER: Use one of the following equations to determine one equivalent volume (EV);

    1 EV = volume in casing + volume in saturated sandpack. Or:

    $1\ EV = [\pi R_w^2 h_w + 0.30p(R_s^2 - R_w^2)h_s] * (0.0043)$

Where:

R_s = radius of sandpack in inches
R_w = radius of well casing in inches
h_s = height of sandpack in inches
h_w = water depth in inches

$0.0043 = gal/in^3$

and filter pack porosity is assumed as 30%, or

Volume in casing =
$(0.0043\ gal/in^3)(p)(12\ in/ft)(R_c^2)(W_h)$

Where:

R_c = radius of casing in inches, and
W_h = water column height in feet

Vol. in sandpack =
(0.0043 gal/in3)(p)(12 in/ft)(Rb2 - Rc2)(Wh)(0.30)

(if Wh is less than the length of the sandpack), or

Vol. in sandpack =
(0.0043 gal/in3)(p)(12 in/ft)(Rb2 - Rc2)(Sh)(0.30)

(if Wh is greater than the length of the sandpack).

where:

Rb = radius of the borehole, and
Sh = length of the sandpack.

Show this calculation in the comments section.

1. PUMP RATE: Record pump rate;
2. TOTAL PUMP TIME: Record total purge time and volume;

3. WELL WENT DRY? Write "YES" or "NO";
4. PUMP TIME: Record pump time that made the well go dry;
5. VOLUME REMOVED: Record the volume of water (gal) removed before the well went dry;
6. RECOVERY TIME: Record the time required for the well to refill;
7. PURGE AGAIN?: Answer "YES" or "NO";
8. TOTAL VOL. REMOVED: Record the total volume of water (in gallons) removed from the well;
9. CAL REF.: Record the calibration reference for the pH meter;
10. TIME: Record time started (INITIAL T(0)), 2 times DURING the sampling and the time sampling ended (FINAL);
11. pH: Record the pH at start of sampling (INITIAL), twice DURING the sampling, and at the end of sampling (FINAL);
12. TEMP: Record the water temperature (Celsius) at the start of sampling, twice DURING the sampling, and at the end of sampling (FINAL);
13. COND: Record the conductivity of the water at the start of sampling, twice DURING the sampling, and at the end of sampling (FINAL);
14. D.O.: Record the dissolved oxygen level in the water at the start of sampling, twice DURING the sampling, and at the end of sampling (FINAL);
15. TURBIDITY: Record the readings from the turbidity meter (nephelometer) and units at the start of sampling, twice DURING the sampling, and at the end of sampling (FINAL);
16. ORD: Record the oxidation/reduction (RedOx) potential of the water sample at the start of sampling, twice DURING the sampling, and at the end of sampling (FINAL);
17. HEAD SPACE: Record any positive readings from organic vapor meter reading taken in well headspace before sampling;
18. NAPL: Record the presence and thickness of any non-aqueous phase liquids (LNAPL and DNAPL)
19. COMMENTS: Record any pertinent information not already covered in the form; and
20. SIGNATURE: Sign the form.

3.4 FIELD CALIBRATION FORMS (REFER TO FORM 10.2-E)

1. Record time and date of calibration;
2. Record calibration standard reference number;
3. Record meter ID number;
4. Record initial instrument reading, recalibration reading (if necessary), and final calibration reading on appropriate line;
5. Record value of reference standard (as required);
6. COMMENTS: Record any pertinent information not already covered on form; and
7. SIGNATURE: Sign form.

4.0 MAINTENANCE

Not applicable.

5.0 PRECAUTIONS

None.

6.0 REFERENCE

USEPA. 1991. *User's Guide to the Contract Laboratory Program.* EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response, January.

FIELD PARAMETER/LOGBOOK FORM 10.2-a
SOIL AND SEDIMENT SAMPLES

HIGH CONCENTRATION EXPECTED? _____ HIGH HAZARD?

INSTALLATION/SITE _____ AREA _____

INST NAME _____ FILE NAME _____

SAMPLE MATRIX CODE _____ SITE ID _____
ENV. FIELD SAMPLE IDENTIFIER _____

DATE (MM/DD/YY) __/__/__ TIME _____ AM PM SAMPLE PROGRAM

DEPTH (TOP) _____ DEPTH INTERVAL _____ UNIT _____

SAMPLING METHOD:

SPLIT SPOON __ AUGER __ SHELBY TUBE __ SCOOP __ OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE_____

DESCRIPTION OF SITE AND SAMPLE CONDITIONS

SITE DESCRIPTION:_____

_____

_____

SAMPLE FORM _____ COLOR _____ ODOR _____

PID (HNu)_____ UNUSUAL FEATURES_____

_____

WEATHER/TEMPERATURE

SAMPLER _____

FIELD PARAMETER/LOGBOOK FORM 10.2-b
GROUNDWATER AND SURFACE WATER SAMPLES

HIGH CONCENTRATION EXPECTED? _____ HIGH HAZARD?

INSTALLATION/SITE _____ AREA

INST CODE _____ FILE NAME _____ SITE TYPE

SITE ID _____ FIELD SAMPLE NUMBER

DATE (MM/DD/YY) __/__/__ TIME _____ AM PM SAMPLE PROG.

DEPTH (TOP) _____ DEPTH INTERVAL _____ UNITS

SAMPLING MEASUREMENTS

CAL REF. ____ pH ____ TEMPERATURE °C ____ CONDUCTIVITY ____ REDOX ____

DISSOLVED OXYGEN ____ TURBIDITY ____ OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE_____

DESCRIPTION OF SITE AND SAMPLE CONDITIONS

SITE DESCRIPTION_____

SAMPLING METHOD_____

SAMPLE FORM _____ COLOR _____ ODOR _____

PID (HNu)_____

UNUSUAL FEATURES_____

WEATHER/TEMPERATURE_____ SAMPLER _____

EXAMPLE MAP FILE LOGBOOK FORM 10.2-c
SURFACE WATER, SOIL, AND SEDIMENT SAMPLES

SITE ID_____ POINTER _____

DESCRIPTION/MEASUREMENTS_____

SKETCH/DIMENSIONS :

MAP REFERENCE_____

COORDINATE DEFINITION (X is _____ Y is _____)

COORDINATE SYSTEM _____ SOURCE _____ ACCURACY_____

X-COORDINATE _____ Y-COORDINATE _____ UNITS_____

ELEVATION REFERENCE_____

ELEVATION SOURCE _____ ACCURACY _____ELEVATION_____

UNITS_____

SAMPLER_____

EXAMPLE MAP FILE AND PURGING LOGBOOK FORM 10.2-d
GROUNDWATER SAMPLES

WELL COORD. OR ID _____ SAMPLE NO._____

WELL/SITE DESCRIPTION_____

_____

X-COORD. _____ Y-COORD. _____ ELEV. _____ UNITS

DATE ___/___/___ TIME _____ AIR TEMP.

WELL DEPTH _____ FT. _____ IN. CASING HT. _____ FT. ____ IN.

WATER DEPTH _____ FT. _____ IN. WELL DIAMETER _____ IN.

WATER COLUMN HEIGHT _____ FT. _____ IN. SANDPACK DIAM. _____ IN.

EQUIVALENT VOLUME OF STANDING WATER _____ (GAL) (L)

VOLUME OF BAILER _____ (GAL) (L) or PUMP RATE _____ (GPM) (LPM)

TOTAL NO. OF BAILERS (5 EV) _____ or PUMP TIME _____ MIN.

WELL WENT DRY? [Yes] [No] NUM. OF BAILERS _____ or PUMP TIME

VOL. REMOVED _____ (GAL) (L) RECOVERY TIME

PURGE AGAIN? [Yes] [No] TOTAL VOL. REMOVED _____ (GAL) (L)

DATE & TIME	QUANTITY REMOVED	TIME REQ'D	pH	Cond	Temp	ORD	Turb	DO	Character of water (color / clarity / odor / partic.)
(before)									
(during)									
(during)									
(during									
(after)									

COMMENTS_____

SIGNATURE_____

EXAMPLE FIELD CALIBRATION FORM 10.2-e
FOR pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY,
ORD, AND DISSOLVED OXYGEN METERS

INITIAL CALIBRATION	FINAL CALIBRATION
DATE:	DATE:
TIME:	TIME:

pH METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO:_____

METER ID_____

pH STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
7.0			
10.0			
4.0			

CONDUCTIVITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO:_____

METER ID_____

COND. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

TEMPERATURE METER CALIBRATION

METER ID_____

TEMP. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
ICE WATER			
BOILING WATER			
OTHER _____			

EXAMPLE FIELD CALIBRATION FORM 10.2-e
FOR pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY,
ORD, AND DISSOLVED OXYGEN METERS

TURBIDITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO:_____

METER ID_____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

ORD METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO:_____

METER ID_____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

DISSOLVED OXYGEN METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO:_____

METER ID_____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

COMMENTS_____

SIGNATURE_____

STANDARD OPERATING PROCEDURE 10.3
BORING LOGS

1.0 INTRODUCTION

The purpose of this standard operating procedure (SOP) is to describe the methods to be followed for classifying soil and rock, as well as preparing borehole logs and other types of soil reports.

2.0 MATERIALS

The following equipment is required for borehole logging:

- HTRW ENG Form 5056-R and 5056A-R boring log forms;
- Daily inspection report forms;
- Chain-of-custody forms;
- Request for analysis forms;
- ASTM D 2488 classification flow chart;
- Soil and/or Rock color chart (i.e., Munsell®);
- Grain size and roundness chart;
- Graph paper;
- Engineer's scale;
- Previous reports and boring logs;
- Pocketknife or putty knife;
- Hand lens;
- Dilute hydrochloric acid (10% volume);
- Gloves;
- Personal protective clothing and equipment, as described in work plan addenda health and safety plan;
- Photoionization detector or other appropriate monitoring equipment per site-specific health and safety plan; and
- Decontamination supplies (SOP 80.1).

3.0 PROCEDURE

Each boring log should fully describe the subsurface environment and the procedures used to obtain this description.

Boring logs should be prepared in the field on USACE Engineer Form 5056-R and 5056-R. Logs should be recorded in the field directly on the boring log form and not transcribed from a field book.

A "site geologist" should conduct borehole logging and soil/rock identification and description or other professional trained in the identification and description of soil/rock.

3.1 BORING LOG INFORMATION

As appropriate, the following information should be recorded on the boring log during the course of drilling and sampling activities:

- Project information including name, location, and project number;
- Each boring and well should be uniquely numbered and located on a sketch map as part of the log;
- Type of exploration;
- Weather conditions including events that could affect subsurface conditions;
- Dates and times for the start and completion of borings, with notations by depth for crew shifts and individual days;
- Depths/heights in feet and in decimal fractions of feet;
- Descriptions of the drilling equipment including rod size, bit type, pump type, rig manufacturer and model, and drilling personnel;
- Drilling sequence and descriptions of casing and method of installation;
- Description and identification of soils in accordance with ASTM Standard D 2488;
- Descriptions of each intact soil sample for the parameters identified in Section 3.2;
- Descriptions and classification of each non-intact sample (e.g., wash samples, cuttings, auger flight samples) to the extent practicable;
- Description and identification of rock;
- Description of rock (core(s)) for the parameters identified in Section 3.7;
- Scaled graphic sketch of the rock core (included or attached to log) according to the requirements identified in Section 3.7;
- Lithologic boundaries, with notations for estimated boundaries;
- Depth of water first encountered in drilling, with the method of first determination (any distinct water level(s) below the first zone will also be noted);
- Interval by depth for each sample taken, classified, and/or retained, with length of sample recovery and sample type and size (diameter and length);
- Blow counts, hammer weight, and length of fall for driven samplers;

- Rate of rock coring and associated rock quality designation (RQD) for intervals cored;
- Drilling fluid pressures, with driller's comments;
- Total depth of drilling and sampling;
- Drilling fluid losses and gains should be recorded;
- Significant color changes in the drilling fluid returned;
- Soil gas or vapor readings with the interval sampled, with information on instrument used and calibration;
- Depth and description of any in-situ test performed; and
- Description of other field tests conducted on soil and rock samples.

3.2 SOIL PARAMETERS FOR LOGGING

In general, the following soil parameters should be included on the boring log when appropriate:

- Identification per ASTM D 2488 with group symbol;
- Secondary components with estimated percentages per ASTM D 2488;
- Color;
- Plasticity per ASTM D 2488;
- Density of non-cohesive soil or consistency of cohesive soil;
- Moisture condition per ASTM D 2488 (dry, moist, or wet);
- Presence of organic material;
- Cementation and HCL reaction testing per ASTM D 2488;
- Coarse-grained particle description per ASTM D 2488 including angularity, shapes, and color;
- Structure per ASTM D 2488 and orientation;
- Odor; and
- Depositional environment and formation, if known.

ASTM D 2488 categorizes soils into 13 basic groups with distinct geologic and engineering properties based on visual-manual identification procedures. The following steps are required to classify a soil sample:

1. Observe basic properties and characteristics of the soil. These include grain size grading and distribution, and influence of moisture on fine-grained soil.
2. Assign the soil an ASTM D 2488 classification and denote it by the standard group name and symbol.
3. Provide a written description to differentiate between soils in the same group if necessary.

Many soils have characteristics that are not clearly associated with a specific soil group. These soils might be near the borderline between groups, based on particle distribution or plasticity characteristics. In such a

case, assigning dual group names and symbols (e.g., GW/GC or ML/CL) might be an appropriate method of describing the soil. The two general types of soils, for which classification is performed, coarse- and fine-grained soils, are discussed in the following sections.

3.3 COURSE-GRAINED SOIL IDENTIFICATION

For soils in the coarse-grained soils group, more than half of the material in the soil matrix will be retained by a No. 200 sieve (75-μm).

1. Coarse-grained soils are identified on the basis of the following:
   a) Grain size and distribution;
   b) Quantity of fine-grained material (i.e., silt and clay as a percentage); and
   c) Character of fine-grained material.
2. The following symbols are used for classification:

Basic Symbols	Modifying Symbols
G = gravel	W = well graded
S = sand	P = poorly graded
	M = with silty fines
	C = with clayey fines

3. The following basic facts apply to coarse-grained soil classification.

- The basic symbol G is used if the estimated percentage of gravel is greater than that for sand. In contrast, the symbol S is used when the estimated percentage of sand is greater than the percentage of gravel.
- Gravel ranges in size from 3-inch to 1/4-inch (No. 4 sieve) diameter. Sand ranges in size from the No. 4 sieve to No. 200 sieve. The Grain Size Scale used by Engineers (ASTM Standards D 422-63 and D 643-78) is the appropriate method to further classify grain size as specified by ASTM D 2488.
- Modifying symbol W indicates good representation of all particle sizes.
- Modifying symbol P indicates that there is an excess or absence of particular sizes.
- The symbol W or P is used only when there are less than 15% fines in a sample.
- Modifying symbol M is used if fines have little or no plasticity (silty).
- Modifying symbol C is used if fines have low to high plasticity (clayey).

Figure 10.03a is a flowchart for identifying coarse-grained soils by ASTM D 2488.

3.4 FINED-GRAINED SOIL IDENTIFICATON

If one-half or more of the material will pass a No. 200 sieve (75 μm), the soil is identified as fine-grained.

1. Fine-grained soils are classified based on dry strength, dilatancy, toughness, and plasticity.
2. Classification of fine-grained soils uses the following symbols:

Basic Symbols			Modifying Symbols		
M	=	silt (non plastic)	L	=	low liquid limit (lean)
C	=	clay (plastic)	H	=	high liquid limit (fat)
O	=	organic			
Pt	=	peat			

3. The following basic facts apply to fine-grained soil classification:
   - The basic symbol M is used if the soil is mostly silt, while the symbol C applies if it consists mostly of clay.
4. Use of symbol O (group name OL/OH) indicates that organic matter is present in an amount sufficient to influence soil properties. The symbol Pt indicates soil that consists mostly of organic material.

- Modifying symbols (L and H) are based on the following hand tests conducted on a soil sample:
  - — Dry strength (crushing resistance).
  - — Dilatancy (reaction to shaking).
  - — Toughness (consistency near plastic limit).
- Soil designated ML has little or no plasticity and can be recognized by slight dry strength, quick dilatency, and slight toughness.
- CL indicates soil with slight to medium plasticity, which can be recognized by medium to high dry strength, very slow dilatancy, and medium toughness.

Criteria for describing dry strength per ASTM D 2488 are as follows:

Description	Criteria
None	Dry sample crumbles into powder with pressure of handling
Low	Dry specimen crumbles into powder with some finger pressure
Medium	Dry specimen breaks into pieces or crumbles with considerable finger pressure
High	Dry specimen cannot be broken with finger pressure but will break into pieces between thumb and a hard surface
Very high	Dry specimen cannot be broken between the thumb and a hard surface stiffness

Criteria for describing dilatancy per ASTM D 2488 are as follows:

None	No visible change in the sample
Slow	Water appears slow on the surface of the sample during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the sample during shaking and disappears quickly upon squeezing

Criteria for describing toughness per ASTM D 2488 are as follows:

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit and the thread and lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit and the thread and lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit and the thread and lump have very high stiffness

Figure 10.03b is a flowchart for identifying fine-grained soils by ASTM D 2488.

3.5 DENSITY AND CONSISTENCY

Relative density for coarse-grained soils and consistency for fine-grained soils can be estimated using standard penetration test blow count data (ASTM D 1586). The number of blows required for each 6 inches of penetration or fraction thereof is recorded. If the sampler is driven less than 18 inches, the number of blows per each complete 6-inch interval and per partial interval is recorded.

For partial increments, the depth of penetration should be recorded to the nearest 1 inch. If the sampler advances below the bottom of the boring under the weight of rods (static) and/or hammer, then this information should be recorded on the log.

The following are some "rule-of-thumb" guidelines for describing the relative density of coarse-grained soils:

Blow Count	Relative Density for Sand
0–4	Very loose
4–10	Loose
10–30	Medium dense
30–50	Dense
>50	Very Dense

The following are some "rule-of-thumb" guidelines for describing the consistency of fine-grained soils:

Blow Count	Consistency for Clays	Description
0–2	Very Soft	Sample sags or slumps under its own weight
2–4	Soft	Sample can be pinched in two between the thumb and forefinger
4–8	Medium Stiff	Sample can be easily imprinted with fingers
8–16	Stiff	Sample can be imprinted only with considerable pressure of fingers
16–32	Very Stiff	Sample can be imprinted very slightly with fingers
>32	Hard	Sample cannot be imprinted with fingers; can be pierced with pencil

3.6 OTHER DESCRIPTIVE INFORMATION

The approximate percentage of gravel, sand, and fines (use a percentage estimation chart) should be recorded per ASTM D 2488 as follows:

Modifiers	Descriptions
Trace	Less than 5%
Few	5%–10%
Little	15%–25%
Some	30%–45%
Mostly	50%–100%

Color/discoloration should be recorded and described using a soil color chart, such as the Munsell® Soil Color Charts. A narrative and numerical description should be given from the color chart, such as Brown 10 YR, 5/3 (Munsell®). Odor should be described if organic or unusual.

Plasticity should be described as follows:

Description	Criteria
Non-plastic	A 1/8-inch thread cannot be rolled at any water content
Low	Thread can barely be rolled and lump cannot be formed when drier than plastic limit.
Medium	Thread is easy to roll; plastic limit can be reached with little effort and lump crumbles when drier than plastic limit.
High	Considerable time is required to reach the plastic limit and lump can be formed without crumbling when drier than plastic limit

Moisture condition should be recorded as dry (absence of moisture), moist (damp but no visible water) or wet (visible free water).

Cementation should be recorded (carbonates or silicates) along with the results of HCL reaction testing. The reaction with HCL should be described as none (no visible reaction), weak (some reaction with slowly forming bubbles) or strong (violent reaction with bubbles forming immediately).

Particle description information for coarse-grained soil should be recorded where appropriate per ASTM D 2488 including maximum particle size, angularity (angular, subangular, subrounded, or rounded), shape (flat, elongated or flat and elongated), and color.

Structure (along with orientation) should be reported using the following ASTM D 2488 descriptions:

Description	Criteria
Stratified	Alternating layers of varying material or color with layers greater than 6 millimeters thick
Laminated	Alternating layers of varying material or color with layers less than 6 millimeters thick
Fissured	Breaks along definite planes of fracture with little resistance
Slickensided	Fracture planes that appear polished or glossy, can be striated
Blocky	Inclusion of small pockets of different soils
Homogeneous	Same color and appearance throughout

3.7 ROCK CORE PARAMETERS FOR LOGGING

In general, the following parameters should be included on the boring log when rock coring is conducted:

- Rock type;
- Formation;
- Modifier denoting variety;
- Bedding/banding characteristics;
- Color;
- Hardness;
- Degree of cementation;
- Texture;
- Structure and orientation;
- Degree of weathering;
- Solution or void conditions;
- Primary and secondary permeability including estimates and rationale; and
- Lost core interval and reason for loss.

A scaled graphic sketch of the core should provided on or attached to the log, denoting by depth, location, orientation, and nature (natural, coring-induced, or for fitting into core box) of all core breaks. Where fractures are too numerous to be shown individually, their location may be drawn as a zone.

The RQD values for each core interval (run) should be calculated and included on the boring log. The method of calculating the RQD is as follows per ASTM D 6032:

RQD = [Σ length of intact core pieces > 100 mm (4-inches)] x 100%/total core length.

3.8 PROCEDURES FOR ROCK CLASSIFICATION

For rock classification record mineralogy, texture, and structural features (e.g., biotite and quartz fine grains, foliated parallel to relict bedding oriented 15 to 20 degrees to core axis, joints coated with iron oxide). Describe the physical characteristics of the rock that are important for engineering considerations such as fracturing (including minimum, maximum, and most common and degree of spacing), hardness, and weathering.

1. The following is to be used as a guide for assessing fracturing:

AEG Fracturing	Spacing
Crushed	up to 0.1 foot
Intense	0.1–0.5 foot
Moderate	0.5 foot–10 feet
Slight	1.0 foot–3.0 feet
Massive	>3.0 feet

2. Record hardness using the following guidelines:

Hardness	Criteria
Soft	Reserved for plastic material
Friable	Easily crumbled by finger pressure
Low	Deeply gouged or carved with pocketknife
Moderate	Readily scratched with knife; scratch leaves heavy trace of dust
Hard	Difficult to scratch with knife; scratch produces little powder and is often faintly visible
Very Hard	Cannot be scratched with knife

3. Describe weathering using the following guidelines:

Weathering	Decomposition	Discoloration	Fracture Condition
Deep	Moderate to complete alteration of minerals feldspars altered to clay, etc.	Deep and thorough	All fractures extensively coated with oxides, carbonates, or clay
Moderate	Slight alteration of minerals, cleavage surface lusterless and stained	Moderate or localized and intense	Thin coatings or stains
Weak	No megascopic alteration of minerals	Slight and intermittent and localized	Few strains on fracture surfaces
Fresh	Unaltered, cleavage, surface glistening		

3.9 PROCEDURE FOR LOGGING REFUSE

The following procedure applies to the logging of subsurface samples composed of various materials in addition to soil as may be collected from a landfill or other waste disposal site.

1. Observe refuse as it is brought up by the hollow stem auger, bucket auger, or backhoe.

2. If necessary, place the refuse in a plastic bag to examine the sample.

3. Record observations according to the following criteria:

- Composition (by relative volume), e.g., paper, wood, plastic, cloth, cement, or construction debris. Use such terms as "mostly" or "at least half." Do not use percentages;
- Moisture condition: dry, moist, or wet;
- State of decomposition: highly decomposed, moderately decomposed, slightly decomposed, etc.;
- Color: obvious mottling and/or degree of mottling;
- Texture: spongy, plastic (cohesive), friable;
- Odor;

- Combustible gas readings (measure down hole and at surface); and
- Miscellaneous: dates of periodicals and newspapers, ability to read printed materials, degree of drilling effort (easy, difficult, and very difficult).

3.10 SUBMITTAL REQUIREMENTS

Each original boring log should be submitted to the Contracting Officer Representative (CRO) after completion of the boring. When a monitoring well will be installed in a boring, the boring log and well installation diagram should be submitted together.

4.0 MAINTENANCE

Not applicable.

5.0 PRECAUTIONS

Not applicable.

6.0 REFERENCES

ASTM Standard D 1586–84 (1992). 1992. *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils.*

ASTM Standard D 2488-93. 1993. *Standard Practice for Description and Identification of Soils Visual-Manual Procedure).*

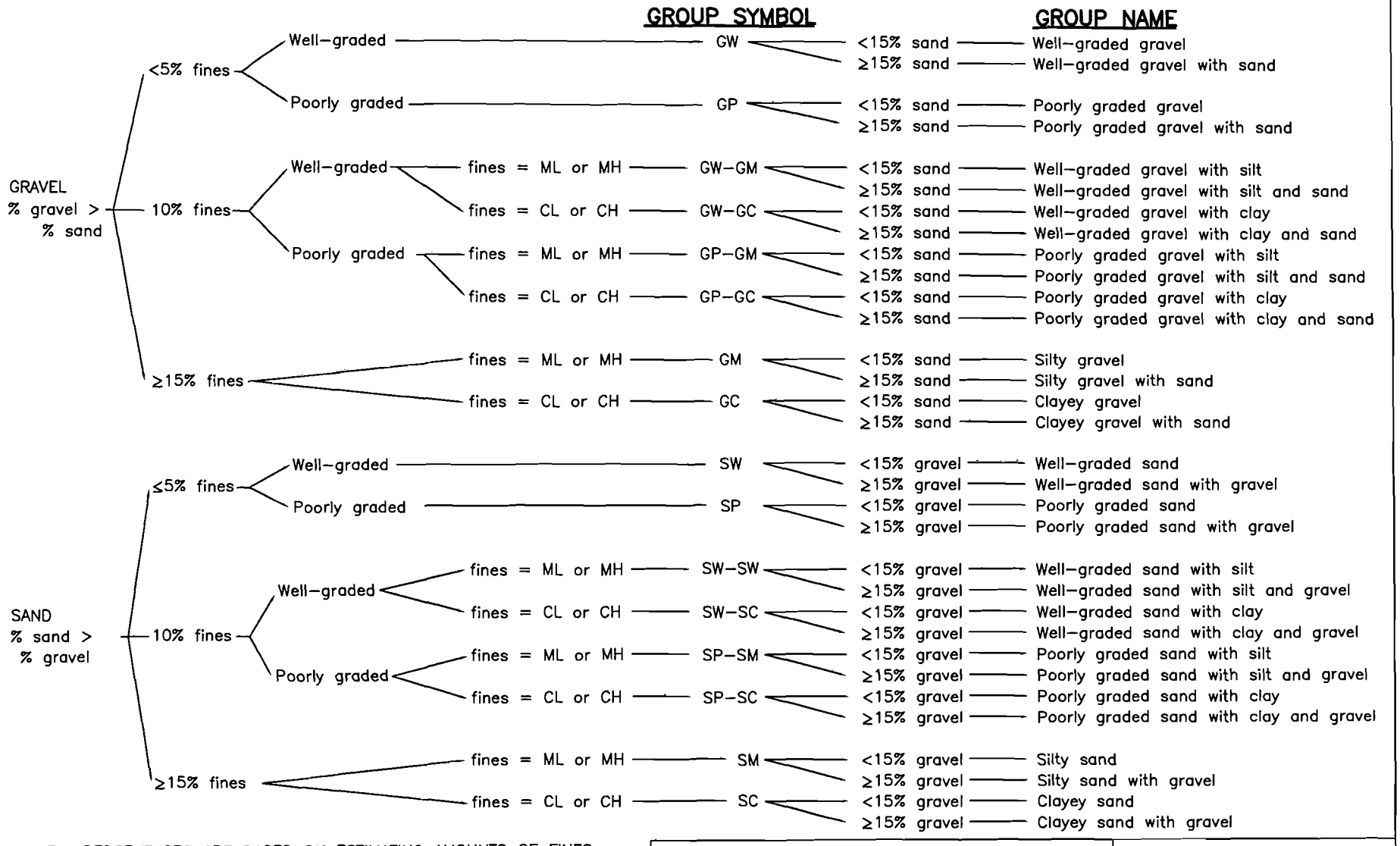
ASTM Standard D 5434-93. 1993. *Guide for Field Logging of Subsurface Explorations of Soil and Rock.*

ASTM Standard D 6032-96. 1996. *Standard Test Method for Determining Rock Quality Designation (RQD) of Rock Core.*

Compton, R. R. 1962. *Manual of Field Geology.* John Wiley & Sons, Inc., New York.

USACE. 1998. *Monitoring Well Design, Installation, and Documentation at Hazardous, Toxic, and Radioactive Waste Sites.* EM 1110-1-4000, 1, November.

U.S. Department of the Interior. 1989. *Earth Manual.* Water and Power Resources Service, Washington, DC.



NOTE: PERCENTAGES ARE BASED ON ESTIMATING AMOUNTS OF FINES, SAND, AND GRAVEL TO THE NEAREST 5%.

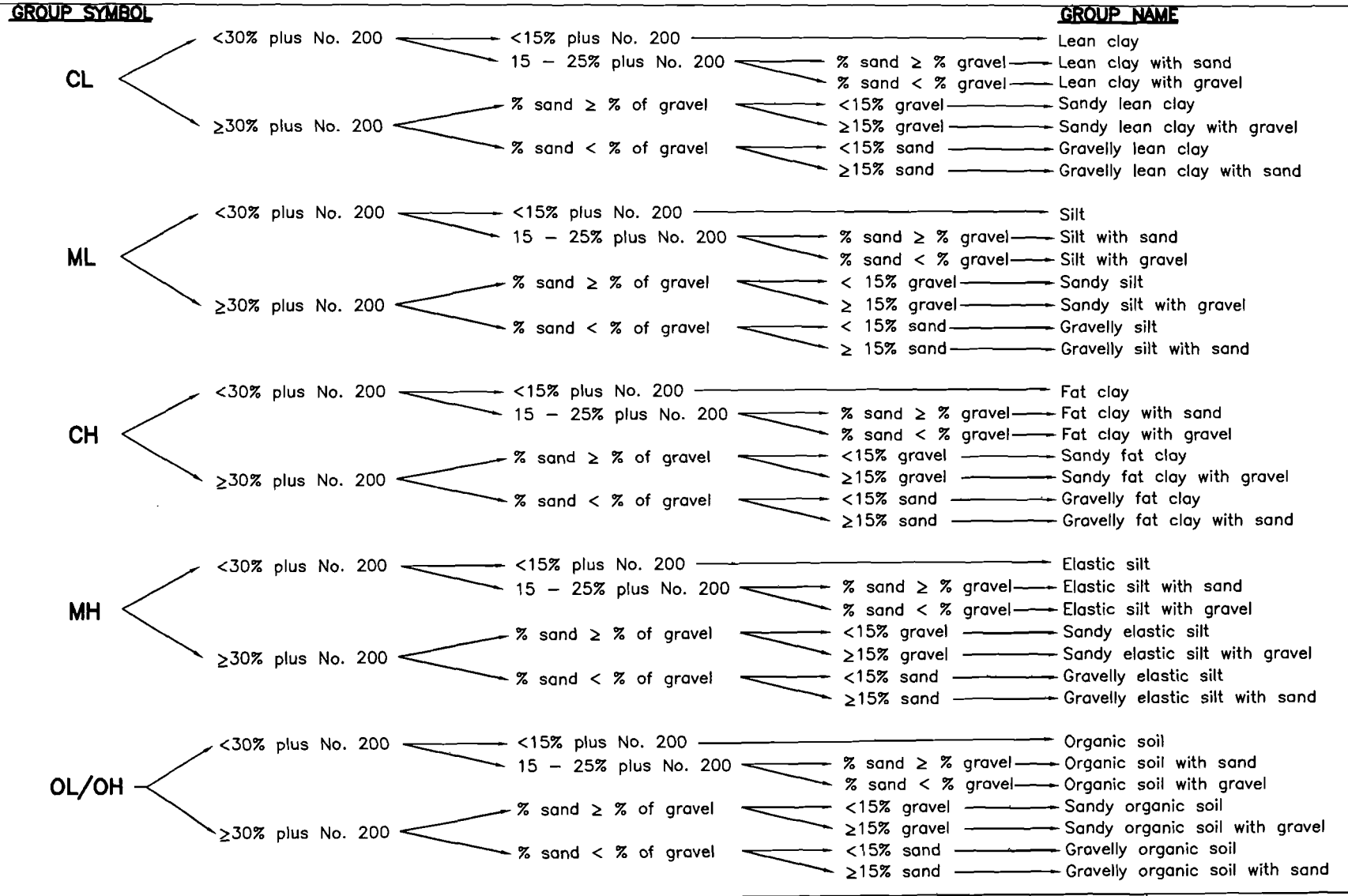
SOURCE: ASTM D 2488

RFAAP
MASTER WORK PLAN

Date: *06/13/01*	**Prepared By:** *URS Corp./DRT*
Scale: *NO SCALE*	**File Name:** *P:\...\Figure XXX.x..*

FIGURE 10.03a

FLOW CHART FOR IDENTIFYING COARSE-GRAINED SOILS



NOTE: PERCENTAGES ARE BASED ON ESTIMATING AMOUNTS OF FINES, SAND, AND GRAVEL TO THE NEAREST 5%.

SOURCE: ASTM D 2488

RFAAP
MASTER WORK PLAN

Date:	Prepared By:
06/13/01	*URS Corp./DRT*
Scale: *NO SCALE*	**File Name:** *P:\...\Figure XXX.x..*

FIGURE 10.03b

FLOW CHART FOR IDENTIFYING FINE-GRAINED SOILS

STANDARD OPERATING PROCEDURE 10.4
CHAIN-OF-CUSTODY FORM

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for use of the chain-of-custody form. An example is provided as part of this SOP. Other formats with similar levels of detail are acceptable.

2.0 MATERIALS

- Chain-of-custody form; and
- Indelible ink pen.

3.0 PROCEDURE

1. Record the project name and number.
2. Record the project contact's name and phone number.
3. Print sampler's names in "Samplers" block.
4. Enter the Field Sample No.
5. Record the sampling dates for all samples.
6. List the sampling times (military format) for all samples.
7. Indicate, "grab" or "composite" sample with an "X."
8. Record matrix (e.g., aqueous, soil).
9. List the analyses/container volume across top.
10. Enter the total number of containers per Field Sample No. in the "Subtotal" column.
11. Enter total number of containers submitted per analysis requested.
12. State the carrier service and airbill number, analytical laboratory, and custody seal numbers.
13. List any comments or special requests in the "Remarks" section.
14. Sign, date, and time the "Relinquished By" section when the cooler is relinquished to the next party.
15. Upon completion of the form, retain the shipper copy and place the forms and the other copies in a zip seal bag to protect from moisture. Affix the zip seal bag to the inside lid of the sample cooler to be sent to the designated laboratory.

4.0 MAINTENANCE

Not applicable.

5.0 PRECAUTIONS

None.

6.0 REFERENCES

USEPA. 1990. *Sampler's Guide to the Contract Laboratory Program.* EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, DC, December 1990.

USEPA. 1991. *User's Guide to the Contract Laboratory Program..* EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response, January 1991.

USEPA. 1998. *EPA Requirements for Quality Assurance Project Plans.* EPA/600/R-98/018, QA/R5, Final, Office of Research and Development, Washington, D.C.

FIGURE 10.4-a
EXAMPLE CHAIN-OF-CUSTODY FORM

Project Number	Project Name				Matrix	A N A L Y S E S									LAB :
Project Contact (Name and Phone Number)															AIRBILL No:
Samplers:															Courier:
Field Sample No.	Date (MM-DD-YY)	Time	Comp	Grab										Subtotal	REMARKS
				TOTAL											

Relinquished by:	Date/time	Received by:	Relinquished by:	Date/Time	Received by:
Relinquished by:	Date/time	Received by: (for lab)	Date/Time	Remarks	

STANDARD OPERATING PROCEDURE 20.3
WELL AND BORING ABANDONMENT

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to establish the protocols by which all borings and wells will be abandoned. The primary objective of boring or well abandonment activities is to permanently abandon the boring or well so that the natural migration of groundwater or soil vapor is not significantly influenced.

2.0 MATERIALS

- Well abandonment equipment including appropriate grout mixing/placement equipment, and heavy equipment as appropriate (drill rig, crane, backhoe, etc.);
- Pure sodium bentonite powder with no additives (bentonite);
- Bentonite pellets (seal);
- Cement (Portland Type II); and
- Approved source water.

3.0 PROCEDURE

The volume of grout required for borehole or well abandonment should be calculated prior to proceeding with abandonment. These calculations should consider loss of material to the formation, changes in borehole diameter, potential zones of washout, and shrinkage of material. Calculations should be recorded on an abandonment record (see Section 3.1.4).

In general, cement grout should be used for boring and well abandonment per the specifications in Section 3.1 and procedures identified in the following sections. Specialized narrow diameter soil borings (3-inches or less) associated with direct push methods or hand augers may be abandoned using bentonite pellets or chips (see Section 3.5).

Any replacement borings or wells associated with the abandonment should be offset at least 20 feet from any abandoned site in a presumed up- or cross-gradient direction.

3.1 GROUT

Grout used in construction will be composed by weight of the following:

- Type II Portland cement (Type IV Portland Cement if sulfate concentrations are greater than 1,500 ppm);
- Bentonite (2 to 5% dry bentonite per 94-lb sack of dry cement); and
- A maximum of 6 to 7 gallons of approved water per 94-lb sack of cement.

Neither additives nor borehole cuttings will be mixed with the grout. Bentonite will be added after the required amount of cement is mixed with the water.

All grout material will be combined in an aboveground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout will be recirculated through the grout pump before placement.

Grout placement will be performed using a commercially available grout pump and a rigid tremie pipe. Removal and grouting will be accomplished in stages, aquifer by aquifer, sealing the boring from the bottom to ground surface. This will be accomplished by placing a grout pipe to the bottom and pumping grout through the pipe until undiluted grout reaches the bottom of the next higher section of casing or, for the top-most section, until grout flows from the boring at ground surface.

After 24 hours, the abandoned drilling site will be checked for grout settlement. Any settlement will be filled with grout and rechecked 24 hours later. This process will be repeated until firm grout remains at the ground surface.

3.2 BORINGS

The term "borings" as used in this SOP applies to any drilled hole made that is not completed as a well. This includes soil test borings, soil sampling borings, and deep stratigraphic borings. Whether completed to the planned depth or aborted for any reason before reaching that depth, borings will be grouted and will be normally closed within 12 hours.

To achieve an effective seal, the borehole to be abandoned should be free of debris and foreign matter that may restrict the adhesion of the grout to the borehole wall. Borehole flushing with a tremie pipe may be required to remove such materials prior to grouting.

Each boring to be abandoned should be sealed by grouting from the bottom of the boring to the ground surface. This will be accomplished by placing a tremie pipe to the bottom of the borehole and pumping grout through the pipe at a steady rate. The grouting should be completed slowly and continuously to prevent channeling of material. The tremie pipe should be raised when pumping pressure increases significantly or when undiluted grout reaches the surface.

After 24 hours of completing the abandonment, the abandoned boring or well should be checked for any grout settlement. The settlement depression should be filled with grout and rechecked 24 hours later. Grout should be placed with a tremie pipe if the open hole is 15 feet or deeper or if the hole is not dry. Otherwise, the grout may be poured from the surface.

3.3 NARROW BORINGS

Narrow borings, those with diameter less than 3 inches, advanced by hand auger or direct push methods, may be sealed using bentonite pellets or chips rather than a grout mixture. Often times a grout pump is not available to mix the grout when these methods have been used. Bentonite pellets or chips will be poured into the boring from the ground surface. Then bentonite will hydrate by absorbing moisture from the ground; unapproved water should not be added to the boring. After 24 hours, the abandoned boring will be checked, and any grout settlement will be topped off with more bentonite. The process will be repeated until bentonite remains at ground surface unless site condition indicates otherwise.

3.4 WELLS

The following procedure applies to wells aborted before completion and existing wells determined to be ineffective or otherwise in need of closure.

General Considerations

A number of techniques are available for abandoning monitoring wells and other monitoring devices including:

- Abandonment in place by grouting the well screen and casing in place;
- Removal of the well by pulling; and
- Overdrilling.

The particular method used for abandonment should be specified in the work plan addenda developed for a site-specific investigation. Several factors must be considered when selecting the appropriate abandonment technique including well construction, well condition, and subsurface conditions.
In general the preferred method for abandonment of wells is to remove all existing well materials to:

- Reduce the potential for the formation of a vertical conduit to occur at the contact between the casing and annular seal;
- Reduce the potential for well materials interfering with the abandonment procedures; and
- Decrease the potential for reaction between the well materials and grout used for abandonment.

In general, all well materials will be removed during abandonment (including screen and casing) by either pulling out the casing, screen, and associated materials or by overdrilling using a rotary or hollow stem auger drilling procedure.

Abandonment with Well Materials In Place

In the event that it is not possible to remove the casing and screen, the casing and screen will be perforated using a suitable tool. A minimum of four rows of perforations several inches long and a minimum of five perforations per linear foot of casing or screen is recommended.

After the screen and casing have been appropriately perforated, the well should be abandoned by grouting from the bottom of the well to the ground surface using a tremie pipe as described in Section 3.2. The tremie pipe should be raised when pumping pressure increases significantly or when undiluted grout reaches the surface.

After 24 hours of completing the abandonment, the abandoned well should be checked for any grout settlement. The settlement depression should be filled with grout and rechecked 24 hours later. Grout should be placed with a tremie pipe if the open hole is 15 feet or deeper or if the hole is not dry. Otherwise, the grout may be poured from the surface.

Abandonment by Removal

Site conditions permitting, relatively shallow monitoring wells may be successfully abandoned by removal providing that the well is generally good condition and sections of casing (including screen) can be successfully removed with materials intact.

This method of abandonment is generally accomplished by removing (pulling) sections of casing and screen out of the subsurface using a drill rig, backhoe, crane, etc. of sufficient capacity. Materials with lower

tensile strength such as polyvinyl chloride (PVC) generally cannot be removed by pulling if they have been appropriately cemented in place.

Once the well materials have been removed from the borehole, the borehole should be abandoned by grouting in the same manner discussed for borings in Section 3.2. If the borehole collapses after removal of well materials, then the borehole should be over drilled to remove all material and then grouted to the surface.

Overdrilling

With this method of abandonment, the well materials are removed by overdrilling (overreaming) the well location. Overdrilling using rotary techniques may be accomplished using an overreaming tool. This tool consists of a pilot bit that is approximately the same size as the inner diameter of well casing and a reaming bit that is slightly larger than the diameter of the borehole. As drilling proceeds, all well materials are destroyed and returned to the surface. After completion of the overdrilling, the borehole should be immediately grouted with a tremie pipe as described in Section 3.2.

In the case of overburden wells, a hollow stem auger may be used for overdrilling providing that this method of drilling appropriate for the subsurface conditions. The hollow stem auger should be equipped with outward facing carbide-cutting teeth with a diameter 2 to 4 inches larger than the well casing. With this method, the casing guides the cutting head and remains inside the auger. When the auger reaches the bottom of the well boring and the well materials have been removed, the borehole may be grouted with a tremie pipe (Section 3.2) through the augers as the augers are gradually withdrawn.

Considerations for Fractured Bedrock and Karst Wells

Multi-cased wells completed into bedrock as screened wells, open wells, or open-lined wells may be abandoned with the outer casing left in place providing that the integrity of this casing and associated annular seal is good. A cement bond log (acoustic amplitude boring geophysical log) may be used to evaluate the integrity of the casing and annular seal, if the outer casing is to be left in place.

Borings or wells completed in karst zones may be difficult to abandon because of the potential presence of large conduits, which may make it difficult to grout. Where large conduits exist or difficulties are encountered when abandoning a boring or well, fill the portion of the borehole penetrating the solution cavity with inert gravel (quartz, claystone, etc.). Packers can be used to isolate critical intervals for filling with grout above and below these zones.

3.5 RESTORATION

All work areas around the borings or wells abandoned should be restored to a condition essentially equivalent to that before the borings and wells were installed.

3.6 INVESTIGATION-DERIVED MATERIAL

Investigation-derived material should be managed in accordance with the requirements of SOP 70.1 and the work plan addenda associated with the site investigation

3.7 DOCUMENTATION

For each abandoned boring or well, a record should be prepared to include the following as appropriate:

- Project and boring/well designation;
- Location with respect to replacement boring well (if any);

- Open depth of well/annulus/boring prior to grouting;
- Casing or items left in hole by depth, description, composition, and size;
- Copy of the boring log;
- Copy of construction diagram for abandoned well;
- Reason for abandonment;
- Description and total quantity of grout used initially;
- Description and daily quantities of grout used to compensate for settlement;
- Disposition of investigation-derived material;
- Water or mud level prior to grouting and date measured; and
- Remaining casing above ground surface, height above ground surface, size, and disposition of each.

Daily investigation activities at the site related to boring and well abandonment should be recorded in field logbooks as described in SOPs 10.1 and 10.2.

4.0 PRECAUTIONS

Refer to the health and safety plan associated with the Work Plan Addenda and the Master Health and Safety Plan.

5.0 REFERENCES

ASTM Standard D 5299-92. 1992. *Standard Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities.*

USACE. 1998. *Monitoring Well Design, Installation, and Documentation at Hazardous, Toxic, and Radioactive Waste Sites.* EM 1110-1-4000, 1 November.

STANDARD OPERATING PROCEDURE 30.1
SOIL SAMPLING

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for sampling surface and subsurface soils.

2.0 MATERIALS

- Stainless steel scoop, spoon, trowel, knife, spatula, (as needed);
- Split-spoon, Shelby tube, or core barrel sampler;
- Hand auger or push tube sampler;
- Drill rig and associated equipment (subsurface soil);
- Stainless steel bowls;
- Photoionization detector or other appropriate instrument as specified in site-specific health and safety plan;
- Sampling equipment for collection of volatile organic samples;
- Appropriate sample containers;
- Appropriate sample labels and packaging material.;
- Personal protective equipment and clothing (PPE) per site-specific health and safety plan; and
- Decontamination equipment and supplies (SOP 80.1).

3.0 PROCEDURE

3.1 DOCUMENTATION

Soil sampling information should be recorded in the field logbooks as described in SOPs 10.1 and 10.2.

3.2 SURFICIAL SOIL SAMPLES

The targeted depths for surficial soil samples (surface and near surface) will be specified in the work plan addenda developed for site-specific investigations.

1. All monitoring equipment should be appropriately calibrated before beginning sampling according to the requirements of the work plan addenda and SOP 90.1 or 90.2.
2. All sampling equipment should be appropriately decontaminated before and after use according to the requirements of the work plan addendum and SOP 80.1.
3. Use a spade, shovel, or trowel or other equipment (manufactured from material, which is compatible with the soil to be sampled) to remove any overburden material present (including vegetative mat) to the level specified for sampling.
4. Measure and record the depth at which the sample will be collected with an engineers scale or tape.

5. Remove the thin layer that was in contact with the overburden removal equipment using a clean stainless steel scoop or equivalent and discard it.
6. Begin sampling with the acquisition of any discrete sample(s) for analysis of volatile organic compounds (VOCs), with as little disturbance as possible. VOC samples will not be composited or homogenized.
7. When a sample will not be collected with a core type of sampler (push tube, split spoon, etc.), the sample for VOC analysis will be collected from freshly exposed soil. The method of collection will follow the procedures specified in SOP 30.8 (Methanol Preservation Method) or 30.9 (En Core® Method) based on the requirements of the work plan addenda.
8. Field screen the sample with properly calibrated photoionization detector (PID) or other appropriate instrument. Cut a cross-sectional slice from the core or center of the sample and insert the monitoring instrument(s). Based on the screening results, collect the VOC fraction, as applicable.
9. Collect a suitable volume of sample from the targeted depth with a clean stainless steel scoop (or similar equipment), push tube sampler, or bucket auger
10. For core type of samplers, rough trimming of the sampling location surface should be considered if the sampling surface is not fresh or other waste, different soil strata, or vegetation may contaminate it. Surface layers can be removed using a clean stainless steel, spatula, scoop, or knife. Samples collected with a bucket auger or core type of sampler should be logged per the requirements of SOP 10.3.
11. If homogenization or compositing of the sampling location is not appropriate for the remaining parameters, the sample should be directly placed into appropriate sample containers with a stainless steel spoon or equivalent.
12. If homogenization of the sample location is appropriate or compositing of different locations is desired, transfer the sample to a stainless steel bowl for mixing. The sample should be thoroughly mixed with a clean stainless steel spoon, scoop, trowel, or spatula and then placed in appropriate sample containers per the requirements for containers and preservation specified in work plan addenda. Secure the cap of each container tightly.
13. Appropriately, label the samples (SOP 50.1), complete the chain-of-custody (SOP 10.4), and package the samples for shipping (SOP 50.2).
14. Return any remaining unused soil to the original sample location. If necessary, add clean sand to bring the subsampling areas back to original grade. Replace the vegetative mat over the disturbed areas.

3.3 SUBSURFACE SAMPLES

All sampling equipment should be appropriately decontaminated before and after use according to the requirements of the work plan addendum and SOP 80.1.

1. All monitoring equipment should be appropriately calibrated before sampling according to the requirement of the work plan addendum and SOP 90.1 or SOP 90.2.
2. All sampling equipment should be appropriately decontaminated before and after use according to the requirements of the work plan addendum and SOP 80.1.
3. Collect split-spoon; core barrel, Shelby tube, sonic core or other similar samples during drilling.
4. Upon opening sampler or extruding sample, immediately screen soil for VOCs using a PID or appropriate instrument. If sampling for VOCs, determine the area of highest concentration; use a

stainless steel knife, trowel, or lab spatula to cut the sample; and screen for VOCs with monitoring instrument(s).

5. Log the sample on the boring log before extracting from the sampler per the requirements of SOP 10.3.
6. Any required VOC samples will be collected first followed by the other parameters. VOC samples will not be composited or homogenized and will be collected from the area exhibiting the highest screening level. The method of VOC sample collection will follow the procedures specified in SOP 30.8 (Methanol Preservation Method) or 30.9 (En Core® Method) based on the requirements of the work plan addenda.
7. Field screen the sample with properly calibrated photoionization detector (PID) or other appropriate instrument. Cut a cross-sectional slice from the core or center of the sample and insert the monitoring instrument(s). Based on the screening results, collect the VOC fraction, as applicable.
8. Rough trimming of the sampling location surface should be considered if the sampling surface is not fresh or other waste, different soil strata, or vegetation may contaminate it. Surface layers can be removed using a clean stainless steel, spatula, scoop, or knife.
9. If homogenization or compositing of the sampling location is not appropriate for other parameters, the sample should be directly placed into appropriate sample containers with a stainless steel spoon or equivalent.
10. If homogenization of the sample location is appropriate or compositing of different locations is desired, transfer the sample to a stainless steel bowl for mixing. The sample should be thoroughly mixed with a clean stainless steel spoon, scoop, trowel, or spatula and placed in appropriate sample containers per the requirements for containers and preservation specified in work plan addenda. Secure the cap of each container tightly.

15. Appropriately, label the samples (SOP 50.1), complete the chain-of-custody (SOP 10.4), and package the samples for shipping (SOP 50.2).
16. Discard any remaining sample into the drums used for collection of cuttings.
17. Abandon borings according to procedures outlined in SOP 20.2.

3.4 INVESTIGATION-DERIVED MATERIAL

Investigation-derived material will be managed in accordance with procedures defined in the work plan addenda for the site being investigated and SOP 70.1.

NOTES: If sample recoveries are poor, it may be necessary to composite samples before placing them in jars. In this case, the procedure will be the same except that two split-spoon samples (or other types of samples) will be mixed together. The boring log should clearly state that the samples have been composited, which samples were composited, and why the compositing was done. In addition, VOC fraction should be collected from the first sampling device.

When specified, samples taken for geotechnical analysis (e.g., percent moisture, density, porosity, and grain size) will be undisturbed samples, such as those collected using a thin-walled (Shelby tube) sampler, sonic core sampler, etc.

4.0 MAINTENANCE

Not applicable.

5.0 PRECAUTIONS

Refer to the site-specific health and safety plan.

Soil samples will not include vegetative matter, rocks, or pebbles unless the latter are part of the overall soil matrix.

6.0 REFERENCES

ASTM Standard D 1586-84. 1984. *Penetration Test and Split-Barrel Sampling of Soils.*

ASTM Standard D 1587-83. 1983. *Thin Walled Sampling of Soils.*

ASTM Standard D 5633-94. 1994. *Standard Practice for Sampling with a Scoop.*

USACE. 2001. *Requirements for the Preparation of Sampling and Analysis Plans.* EM 200-1-3. 1 February.

STANDARD OPERATING PROCEDURE 30.2
GROUNDWATER SAMPLING

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for the collection of groundwater samples from monitoring wells.

2.0 MATERIALS

- Work Plans;
- Field logbooks and field parameter forms;
- Plastic sheeting;
- Decontamination equipment and supplies (SOP 80.1);
- Variable-speed, low-flow submersible pump with safety drop cable;
- Nylon stay-ties;
- Generator;
- Dedicated Teflon tubing or Teflon lined polyethylene tubing;
- Flow-through-cell and probes for measuring pH, temperature, specific conductance, oxidation/reduction potential, dissolved oxygen, and turbidity (SOP 40.1);
- Electronic water-level indicator;
- Appropriate sample bottles, labels, chain-of-custody forms, and sample shipping supplies etc;
- Cooler with ice;
- Silicone tubing;
- 0.45-micron disposable filters (as appropriate).
- Personal protective equipment and clothing (PPE) per site-specific health and safety plan;
- Photoionization detector (PID) or other appropriate monitoring instrument per the site-specific health and safety plan; and
- Appropriate containers for investigation-derived material.

3.0 PROCEDURE

3.1 DOCUMENTATION

Groundwater sampling information should be recorded in the field logbooks as described in SOPs 10.1 and 10.2.

The following are general rules for the field parameter logbook for groundwater, as described in SOP 10.2:

- Only information for one site or installation per logbook. The same book maybe used for more than one sampling event.
- The first five pages will be reserved for index, general notes, etc. Sign and date each entry.
- Fill in the forms.
- Duplicate copies, index pages, and calibration sheets remain intact.

3.2 OVERVIEW OF SAMPLING TECHNIQUES

In general, two different techniques may be used to sample groundwater from monitoring wells at Radford Army Ammunition Plant (RFAAP):

- Low flow purging and sampling (Type I); and
- Conventional purging and low-flow sampling (Type II).

These two sampling techniques are intended to address the different groundwater conditions that may be encountered at RFAAP.

The Type I sampling technique will be used in the following situations:

- In wells where only one discrete water-producing zone is encountered;
- In wells with no discrete water bearing zone and a low yield (generally < 0.5 liters per minute); and
- In wells sampled during seasonal low groundwater conditions with greatly reduced yield.

The Type II sampling technique will be used in the following situations:

- In a well with potential or documented multiple flow zones and where individual flow zones will not be evaluated;
- In moderately producing wells (> 0.5 liters per minute) where no discrete flow zones were documented during drilling; and
- In wells sampled during seasonal high groundwater conditions with enhanced yield (and potentially additional flow zones).

Groundwater samples should be collected no sooner than 14 days after well development. Information from the boring logs, well completion records, and well development records should be reviewed before sampling a well to determine the most appropriate sampling technique. Pertinent information for each well to be sampled includes:

- Well construction;
- Depth and nature of water producing zones;
- Sustainable pumping rate of the well to be sampled;
- Well recharge characteristics; and
- Baseline turbidity.

Because of the heterogeneous nature of the fracture and solution-enhanced fractured bedrock at RFAAP, monitoring well purging and sampling techniques will need to be flexible. This flexibility is necessary to

obtain representative samples that meet the data quality objectives (DQOs) specified in site-specific work plan addenda.

In general, when using the pumps specified in the following sections, situate any gasoline-powered generator on level ground approximately 15 ft downwind from the well. All generator maintenance (oil and fueling) is to be performed off site. If the hose(s) and/or power cord of the pump is not on a reel, place the pump with its hose and power cord on the plastic sheeting downhill from the well.

3.3 TYPE I SAMPLING PROCEDURES

Type I low flow purging and sampling procedures include the following:

- The work area outside the well will be prepared by placing plastic sheeting on the ground around the well casing to avoid cross-contamination.
- All equipment used to purge and sample the wells will be thoroughly decontaminated before and after use according to the requirements of the work plan addenda and SOP 80.1.
- All equipment to be used for monitoring water quality parameters will be calibrated before beginning purging according to the requirements of the work plan addenda and SOP 40.1.
- Note the condition of the well and well head.
- Monitor the headspace of the well with a photoionization detector as the well cap is removed.
- Measure and record the depth to water with an electronic water level indicator. The measurement of well depth will not be taken until after sampling is completed so that potential re-suspension of any settled solids at the bottom of the well is avoided.
- Well depth at the time of purging will be obtained from well construction and existing data.
- Slowly lower a clean, stainless steel, adjustable flow rate, submersible pump and dedicated Teflon or Teflon-lined polyethylene tubing to the desired depth. As the pump is slowly lowered into the well, secure the safety drop cable, tubing, and electrical lines to each other using nylon stay-ties.
- For wells with very low sustainable pumping rates ($\leq$ 0.5 liters per minute), the pump should be set in the middle of the saturated screen section of the well or middle of the water column for open wells. The pump should be set 12 hours prior to purging so that the depth to water equilibrates and sediments disturbed during pump placement have time to settle.
- For wells with sustainable pumping rates ($>$ 0.5 liters per minute), the pumps will be set at a desired depth prior to purging, allowing for the depth to water to equilibrate before sampling. The desired depth will be specified in work plan addenda based on site-specific conditions and DQOs.
- Connect the pump tubing to an in-line flow-through cell(s) and connect the multi-parameter probe to the cell(s). The end of the tubing exiting the in-line flow-through cell should be placed to discharge into a appropriate container(s) to collect purge water.
- Immediately prior to purging, the depth to water will be measured and record. Start pumping the water at a rate of 100 to 400 milliliters per minute. Avoid surging. The pumping rate should cause minimal drawdown (less than 0.2 ft). Water level measurements should be collected continuously to document stabilization of the water level. Pumping rates should, if needed, be reduced to the minimal capabilities of the pump to avoid dewatering the screen interval and ensure stabilization of indicator parameters.

- During purging, water quality indicator parameters will be monitored at the in-line flow-through cell(s) every 3 to 5 minutes. The parameters to be monitored include pH, specific conductance, oxidation/reduction potential (Eh), dissolved oxygen, and turbidity.
- Continue purging until stabilization of indicator parameters is achieved. Stabilization is defined as three consecutive readings that are within the following criteria:
  - ± 0.1 for pH;
  - ±3% for specific conductance;
  - ±10 mV for oxidation/reduction potential (Eh); and
  - ±10% for turbidity and dissolved oxygen.
- If the parameters have stabilized, but the turbidity is not in the range of 5 to 10 NTU, then both filtered and unfiltered samples should be collected for any metals analysis. Filter metal samples should be collected with an in-line filter using a high capacity 0.45-micron particulate filter. This filter should be pre-rinsed according to the manufacturer's instructions.
- Once purging is completed, reduce the pumping rate to its lowest steady rate and disconnect the tubing from the in-line flow-though cell(s).
- Collect groundwater samples directly from the end of the tubing into clean containers provided by the laboratory. The container requirements and preservatives for groundwater samples are specified in work plan addenda. Allowing the pump discharge to flow gently down the inside of the container with minimal turbulence should fill all sample containers. Volatile organic compound (VOC) and gas sensitive parameter samples should be collected first followed by other parameters.
- In general, samples should be collected and containerized in the order of the volatilization sensitivity of the parameters. A preferred collection order for some common parameters is VOCs, extractable organics, metals, cyanide, sulfate and chloride, turbidity, and nitrate and ammonia. The parameters to be collected at any well location are site-specific and are specified in work plan addenda.
- Appropriately, label the samples (SOP 50.1), complete the chain-of-custody (SOP 10.4), and package the samples for shipping (SOP 50.2).
- After the sample collection is complete, remove the pump, tubing, and associated lines. Note: sample tubing will be dedicated to each well.
- Measure and record the total depth of the well.
- Secure the well be replacing and locking the lid.

3.4 TYPE II SAMPLING PROCEDURES

- The work area outside the well will be prepared by placing plastic sheeting on the ground around the well casing to avoid cross-contamination.
- All equipment used to purge and sample the wells will be thoroughly decontaminated before and after use according to the requirements of the work plan addenda and SOP 80.1.
- All equipment to be used for monitoring water quality parameters will be calibrated before beginning purging according to the requirements of the work plan addenda and SOP 40.1.
- Note the condition of the well and well head.
- Monitor the headspace of the well with a photoionization detector as the well cap is removed.

- Measure and record the depth to water with an electronic water level indicator. The measurement of well depth will not be taken until after sampling is completed so that potential re-suspension of any settled solids at the bottom of the well is avoided.
- Well depth at the time of purging will be obtained from well construction and existing data.
- Calculate the standing water column in the well by subtracting the depth to water from the total depth of the well as recorded during completion of the well.
- From the water depth, well diameter, sand pack length, etc., calculate the equivalent volume (1 EV) of water in the well.

  1 EV = volume in casing + volume in saturated sand pack. Therefore; if the water table lies below the top of the sand pack, use the following equation:

  $1\ EV = (pR_w^2h_w) + (0.30p(R_s^2-R_w^2)h_w) * (0.0043)$

  If the water table lies above the top of the sand pack use this equation:

  $1\ EV = [(pR_w^2h_w) + (0.30p(R_s^2-R_w^2)h_s)] * (0.0043)$

  Where: R_s = radius of sand pack in inches
  R_w = radius of well casing in inches
  h_s = height of sand pack in inches
  h_w = water depth in inches

  $0.0043\ gal/in^3$
  Assumed filter pack porosity = 30%

Tables and graphs showing equivalent volumes for typical well constructions are available.

- Slowly lower a clean, stainless steel, adjustable flow rate, submersible pump and dedicated Teflon or Teflon-lined polyethylene tubing to the middle of the saturated screen interval or water column in an open borehole. As the pump is slowly lowered into the well, secure the safety drop cable, tubing, and electrical lines to each other using nylon stay-ties.
- Connect the pump tubing to an in-line flow-through cell(s) and connect the multi-parameter probe to the cell(s). The end of the tubing exiting the in-line flow-through cell should be placed to discharge into an appropriate container to collect purge water.
- Start purging the well at the minimally achievable pumping rate. Gradually increase the pumping rate to achieve the maximum flow rate of the pump or the maximum sustainable flow rate that does not draw down the static water level to a point below the top of the first water bearing zone, whichever is achieved first.
- During purging, water level measurements should be collected periodically to verify water levels in the well.
- During purging, water quality indicator parameters will be monitored at the in-line flow-through cell(s) every 3 to 5 minutes. The parameters to be monitored include pH, specific conductance, oxidation/reduction potential (Eh), dissolved oxygen, and turbidity.
- Note when each indicator parameter stabilizes. Stabilization is defined as three consecutive readings that are within the following criteria:
  - ± 0.1 for pH;
  - ±3% for specific conductance;

- ±10 mV for oxidation/reduction potential (Eh); and
- ±10% for turbidity and dissolved oxygen.

- Three calculated eVs of water in the will be purged prior to sampling. It will be documented if stabilization of the indicator parameters has not occurred after three calculated well volumes have been removed and sampling procedures begin.
- If the turbidity is not in the range of 5 to 10 NTU when purging has been completed, then both filtered and unfiltered samples should be collected for any metals analysis. Filter metal samples should be collected with an in-line filter using a high capacity 0.45-micron particulate filter. This filter should be pre-rinsed according to the manufacturer's instructions.
- Once purging is completed, reduce the pumping rate to its lowest steady rate and disconnect the tubing from the in-line flow-though cell(s).
- Collect groundwater samples directly from the end of the tubing into clean containers provided by the laboratory. The container requirements and preservatives for groundwater samples are specified in work plan addenda. Allowing the pump discharge to flow gently down the inside of the container with minimal turbulence should fill all sample containers. Volatile organic compound (VOC) and gas sensitive parameter samples should be collected first followed by other parameters.
- Appropriately, label the samples (SOP 50.1), complete the chain-of-custody (SOP 10.4), and package the samples for shipping (SOP 50.2).
- After the sample collection is complete, remove the pump, tubing, and associated lines. Note: sample tubing will be dedicated to each well.
- Measure and record the total depth of the well.
- Secure the well be replacing and locking the lid.

3.5 INVESTIGATION-DERIVED MATERIAL

Investigation-derived material will be managed in accordance with procedures defined in the work plan addendum for the site being investigated and SOP 70.1.

4.0 MAINTENANCE

Refer to manufacturer's requirements for maintenance of pumps and generators.

5.0 PRECAUTIONS

Refer to the site-specific health and safety plan.

6.0 REFERENCES

USACE. 2001. *Requirements for the Preparation of Sampling and Analysis Plans.* EM 200-1-3, 1 February.

USEPA. 1997. *Recommended Procedure for Low-flow Purging and Sampling of Groundwater Monitoring Wells.* Bulletin No. QAD023, October.

STANDARD OPERATING PROCEDURE 30.3
SURFACE WATER SAMPLING

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for collecting grab samples of surface water. This procedure can be applied to the collection of surface water samples from streams, rivers, ditches, lakes, ponds, and lagoons.

2.0 MATERIALS

- Work Plans;
- Field logbooks;
- Photoionization detector (PID) or other appropriate monitoring instrument as specified site-specific health and safety plan;
- Appropriate sample bottles, labels, chain-of-custody forms, and sample shipping supplies etc;
- Long-handled dip sampler (polytetrafluoroethylene (PTFE) or stainless steel), as applicable;
- Short-handled dip sampler (PTFE or stainless steel), as applicable;
- Pond sampler (PTFE or stainless steel), as applicable;
- Boat, other stable working platform, personal flotation device, as applicable;
- Sample filtration apparatus, as applicable;
- Peristaltic pump with 0.45-µm filters and disposable PTFE tubing, as applicable;
- Personal protective equipment and clothing (PPE) per site specific health and safety plan; and
- Appropriate containers for investigation-derived material.

3.0 PROCEDURE

3.1 CONSIDERATIONS

Factors that will need to be considered for selection of a surface water sampler include the width, depth, and flow of the surface water body, and whether the sample will be collected from the shore or a vessel. The most appropriate method(s) of sample collection and the appropriate depths of sampling (sampling strategies) will be specified in work plan addenda based on site-specific conditions and data quality objectives (DQOs).

3.2 DOCUMENTATION

Surface water sampling information should be recorded in the field logbooks as described in SOPs 10.1 and 10.2. This information should include a description of the water body characteristics (size, depth, flow, etc.).

Sampling locations should be marked on a site map. Describe each location and place a numbered stake above the visible high water mark on the bank closest to the sampling location and/or mark adjacent trees with surveyor's flagging. The descriptions must be adequate to allow the sampling station to be relocated at some future date by someone other than the original sampling crew.

3.3 SAMPLE LOCATION AND TIMING

Sampling should proceed from downstream locations to upstream locations so that disturbance related to sampling does not affect the samples collected upstream. In addition, if sediment samples are to be collected at the same locations as the surface water samples, the water samples must be collected first. Sampling should be performed quickly and in a manner that minimizes disturbance of bottom sediments to ensure a representative sample.

In general, surface water samples should be collected and containerized in the order of the volatilization sensitivity of the parameters. A preferred collection order for some common parameters is volatile organic compounds (VOCs), extractable organics, metals, cyanide, sulfate and chloride, turbidity, and nitrate and ammonia. The parameters to be collected at any location are site-specific and are specified in work plan addenda.

3.4 SAMPLING METHODS

Various methods may be used to collect samples of surface water and the method used will depend on the considerations discussed in Section 3.1. Some of the more common methods used to collect surface water samples from shallow depths include:

- Submergence of sampling containers;
- Dipper and pond sampler; and
- Peristaltic pump (for non-volatile parameters).

Submergence of Sampling Container

Direct filling the sample containers by submergence is advantageous when the sample might be significantly altered during transfer from a collection device into another container. This method would not be appropriate for sampling at depth.

1. All sampling equipment should be appropriately decontaminated before and after use according to the requirements of the work plan addendum and SOP 80.1.
2. Spread new plastic sheeting on the ground at each sampling location to prevent cross-contamination. If sample access is restricted, use appropriate vessel or another stable working platform adjacent to the area to be sampled.
3. Samples should be collected in order specified in the work plan addenda prepared for the site-specific investigation (also see Section 3.3).
4. Submerge an appropriate sample container with the cap in place with minimal surface disturbance so that the open end is pointing upstream. Sample container requirements are specified in work plan addenda.
5. Allow the container to fill slowly and continuously using the cap to regulate the inflow of water.
6. Retrieve the sample from the surface water with minimal disturbance.
7. Preserve the sample as specified in work plan addenda. Secure the cap on the sample container tightly.

8. Appropriately, label the samples (SOP 50.1), complete the chain-of-custody (SOP 10.4), and package the samples for shipping (SOP 50.2).

Sampling with Dip and Pond Sampler

Dipper and pond samplers prevent unnecessary contamination of the outer surface of the sample container that would occur with the direct submergence method of sampling.

A long handled dipper sampler or a pond sampler can be used to remotely obtain samples where access is poor or non-contact with water is suggested in the health and safety plan.
Sampling with the PTFE or stainless steel dipper or pond sampler (long-handled or measuring cup type):

1. All sampling equipment should be appropriately decontaminated before and after use according to the requirements of the work plan addendum and SOP 80.1.
2. Spread new plastic sheeting on the ground at each sampling location to prevent cross-contamination. If sample access is restricted, use appropriate vessel or another stable working platform adjacent to the area to be sampled.
3. Assemble the sampler.
4. Samples should be collected in order specified in the work plan addenda prepared for the site-specific investigation (also see Section 3.3).
5. Collect samples by slowly submerging the pre-cleaned dipper or pond sampler with minimal surface disturbance. Make sure the open end is pointing upstream.
6. Remove the cap from the sample container and slightly tilt the mouth of the bottle below the edge of the sampler. Sample container requirements are specified in work plan addenda.
7. Empty the sampler slowly. Allow the sample stream to flow gently down the side of the bottle with entry turbulence. Avoid aerating the sample.
8. Continue filling the sample container until the container is filled.
9. Preserve the sample as specified in work plan addenda. Secure the cap on the sample container tightly.
10. Appropriately, label the samples (SOP 50.1), complete the chain-of-custody (SOP 10.4), and package the samples for shipping (SOP 50.2).

Sampling with a Peristaltic Pump

Sampling with a peristaltic pump will extend the lateral reach of the sampler and allow sampling from depths below the water surface. A disadvantage of this method is that it cannot be used to sample for volatile organic compounds because of potential degassing effects.

1. All sampling equipment should be appropriately decontaminated before and after use according to the requirements of the work plan addendum and SOP 80.1.
2. Spread new plastic sheeting on the ground at each sampling location to prevent cross-contamination. If sample access is restricted, use appropriate vessel or another stable working platform adjacent to the area to be sampled.
3. Install clean medical grade silicone tubing in the pump head, as instructed by the manufacturer.
4. Select the length of appropriate suction tubing (PFTE or other) necessary to reach the required sample location/depth and attach to the pump intake or extended section of silicone tubing emanating from the intake side of the pump head.

5. Turn on pump and adjust flow to draw water through tubing. If possible, allow several liters of sample to pass through the system before actual sample collection.
6. Samples should be collected in order specified in the work plan addenda prepared for the site-specific investigation (also see Section 3.3).
7. VOC samples, if required, will be collected using another type of sampling device, as specified in the work plan addenda.
8. Collect surface water samples directly from the end of the tubing into clean laboratory-prepared (preserved) containers. Allowing the pump discharge to flow gently down the inside of the container with minimal turbulence should fill all sample containers. Sample container requirements are specified in work plan addenda.
9. Preserve the sample as specified in work plan addenda. Secure the cap on the sample container tightly.
10. Appropriately, label the samples (SOP 50.1), complete the chain-of-custody (SOP 10.4), and package the samples for shipping (SOP 50.2).

3.5 SAMPLE FILTRATION

If specified in work plan addenda, certain parameters such as metals may need to be collected for both total and dissolved fractions. In this case, filtration will be performed immediately after collecting sample. Set up filtration equipment before collecting sample. Filtration may be accomplished by gravity or, if necessary, due to slow filtering, a peristaltic pump can be used to pressure filter the sample. Vacuum filtration will not be used due to the possibility of analyte volatilization.

Gravity Filtration

1. Using decontaminated forceps, place a 0.45-µm membrane in a decontaminated filter funnel.
2. Slowly pour sample into the funnel and collect filtrate directly into appropriate sample container(s).
3. Preserve the sample as specified in work plan addenda. Secure the cap on the sample container tightly.
4. Appropriately, label the samples (SOP 50.1), complete the chain-of-custody (SOP 10.4), and package the samples for shipping (SOP 50.2).
5. Appropriately dispose of filter membrane.

Filter with a Peristaltic Pump

1. Using previously assembled disposable tubing, 0.45-µm in-line filter, and peristaltic pump, filter sample from collection bucket into appropriate container.
2. Adjust pump rate to avoid aeration of sample.
3. Preserve the sample as specified in work plan addenda. Secure the cap on the sample container tightly.
4. Appropriately, label the samples (SOP 50.1), complete the chain-of-custody (SOP 10.4), and package the samples for shipping (SOP 50.2).

4.0 MAINTENANCE

Refer to manufacturer's specifications for maintenance procedures on generators and pumps.

5.0 PRECAUTIONS

Refer to the site-specific health and safety plan.

6.0 REFERENCES

ASTM Standard D 5358-93. 1993. *Standard Practice for Sampling with a Dipper or Pond Sampler.*

Environmental Monitoring System Laboratory (EMSL), ORD, U.S. Environmental Protection Agency. *Characterization of Hazardous Waste Site-A Method Manual, Volume II-Available Sampling Methods.* 1983.

USACE. 2001. *Requirements for the Preparation of Sampling and Analysis Plans.* EM 200-1-3. 1 February.

STANDARD OPERATING PROCEDURE 30.7
SAMPLING STRATEGIES

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate sampling strategies for sampling various media.

2.0 MATERIALS

- Historical site data;
- Site topography;
- Soil types; and
- Sampled media.

3.0 PROCEDURE

The primary goal of any investigation is to collect samples representative of existing site conditions. Statistics are generally used to ensure samples are as representative as possible. Sampling plans may employ more than one approach to ensure project data quality objectives are adequately addressed. A comparison of sampling strategies is presented in Table 1.

3.1 CLASSICAL STATISTICAL SAMPLING

Classical statistical sampling strategies are appropriately applied to either sites where the source of contamination is known or small sites where the entire area is remediated as one unit. Primary limitations of this sampling approach include (1) inability to address media variability; (2) inadequate characterization of heterogenous sites; and (3) inadequate characterization of sites with unknown contamination characteristics.

3.1.1 Simple Random Sampling

Simple random sampling is generally more costly than other approaches because of the number of samples required for site characterization. This approach is generally used when minimal site information is available and visible signs of contamination are not evident and includes the following features:

- Sampling locations are chosen using random chance probabilities.
- This strategy is most effective when the number of sampling points is large.

3.1.2 Stratified Random Sampling

This sampling approach is a modification to simple random sampling. This approach is suited for large site investigations that encompass a variety of soil types, topographic features, and/or land uses. By dividing the site into homogenous sampling strata based on background and historical data, individual random sampling techniques are applied across the site. Data acquired from each stratum can be used to determine the mean or total contaminant levels and provide these advantages:

- Increased sampling precision results due to sample point grouping and application of random sampling approach.

- Control of variances associated with contamination, location, and topography.

3.1.3 Systematic Grid

The most common statistical sampling strategy is termed either systematic grid or systematic random sampling. This approach is used when a large site must be sampled to characterize the nature and extent of contamination.

Samples are collected at predetermined intervals within a grid pattern according to the following approach:

- Select the first sampling point randomly; remaining sampling points are positioned systematically from the first point.
- Determine the grid design: one or two-dimensional. One-dimensional sample grids may be used for sampling along simple man-made features. Two-dimensional grid systems are ideal for most soil applications.
- Determine the grid type: square or triangular. Sampling is usually performed at each grid-line intersection. Other strategies include sampling within a grid center or obtaining composite samples within a grid.
- Each stratum is sampled based on using the simple random sampling approach but determined using a systematic approach.

3.1.4 Hot-Spot Sampling

Hot spots are small, localized areas of media characterized by high contaminant concentrations. Hot-spot detection is generally performed using a statistical sampling grid. The following factors should be addressed:

- Grid spacing and geometry. The efficiency of hot-spot searches is improved by using a triangular grid. An inverse relationship exists between detection and grid point spacing, e.g., the probability of hot-spot detection is increased as the spacing between grid points is decreased.
- Hot-spot shape/size. The larger the hot spot, the higher the probability of detection. Narrow or semi-circular patterns located between grid sampling locations may not be detected.
- False-negative probability. Estimate the false negative (β-error) associated with hot-spot analysis.

3.1.5 Geostatistical Approach

Geostatistics describe regional variability in sampling and analysis by identifying ranges of correlation or zones of influence. The general two-stage approach includes the following:

- Conducting a sampling survey to collect data defining representative sampling areas.
- Defining the shape, size, and orientation of the systematic grid used in the final sampling event.

3.2 NON-STATISTICAL SAMPLING

3.2.1 Biased Sampling

Specific, known sources of site contamination may be evaluated using biased sampling. Locations are chosen based on existing information.

3.2.2 Judgmental Sampling

This sampling approach entails the subjective selection of sampling locations that appear to be representative of average conditions. Because this method is highly biased, it is suggested that a measure of precision be included through the collection of multiple samples.

4.0 MAINTENANCE

Not applicable.

5.0 REFERENCES

USACE. 2001. *Requirements for the Preparation of Sampling and Analysis Plans.* EM200-1-3. 1 February.

STANDARD OPERATING PROCEDURE 40.1
MULTIPARAMETER WATER QUALITY MONITORING INSTRUMENT

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for field operation with the multiparameter water quality logging system (data transmitter and visual display). This system can monitor up to eleven basic parameters, including dissolved oxygen, percent saturation, temperature, pH, specific conductance, resistivity, salinity, total dissolved solids, redox, level, and depth.

2.0 MATERIALS

- Visual display;
- Data transmitter;
- Underwater cables; and
- Field logbooks.

3.0 PROCEDURE

3.1 CALIBRATION

Calibration will be performed in the field daily before use according to manufacturer's specifications. The following parameters are calibrated to the following standards:

- Temperature—none required;
- Specific conductance—KCl or seawater standards;
- pH—pH 7 buffer plus a slope buffer;
- Dissolved oxygen—saturated air or saturated water;
- Redox—quinhydrone or transfer;
- Depth—set zero in air;
- Level—set zero in air; and
- Salinity—uses calibration for specific conductance.

3.2 OPERATION

1. Attach the cable to the transmitter.
2. Connect the other end of the cable to the display.
3. Press the On/Off key on the display panel. Allow a few seconds for the transmitter to start sending data to the display screen.
4. Calibrate the transmitter.
5. Deploy the sensor into a minimum of 4 in. of water.

6. Write data values from the display screen in the appropriate field logbook.
7. Retrieve sensor and clean the transmitter to prevent cross-contamination.
8. Move to the next sampling location. If travel time is great, turn off display by pressing On/Off key. Check condition of probes after each deployment.
9. Disconnect the transmitter when finished sampling for the day.

4.0 MAINTENANCE

Maintain according to specific manufacturer's specifications.

5.0 PRECAUTIONS

- Check condition of probes frequently between sampling; and
- Don't force pins into the connectors; note the keying sequence.

STANDARD OPERATING PROCEDURE 40.2
WATER LEVEL AND WELL-DEPTH MEASUREMENTS

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for measuring water level and well depth. This procedure is applicable to the sampling of monitoring wells and must be performed before any activities that may disturb the water level, such as purging or aquifer testing.

2.0 MATERIALS

- Work Plans;
- Well construction diagrams;
- Field logbook;
- Photoionization detector (PID) or other monitoring instruments per site-specific health and safety plan;
- Decontamination equipment and supplies (SOP 80.1);
- Electric water level indicator (dipmeter) with cable measured at 0.01 ft increments;
- Oil-water interface probe (if non-aqueous phase liquid (NAPLs) are suspected to be present); and
- Plastic sheeting.

3.0 PROCEDURE

3.1 PRELIMINARY STEPS

1. Locate the well and verify its position on the site map. Record whether positive identification was obtained, including the well number and any identifying marks or codes contained on the well casing or protective casing. Gain access to the top of the well casing.
2. Locate the permanent reference mark at the top of the casing. This reference point will be scribed, notched, or otherwise noted on the top of the casing. If no such marks are present, measure to the top of the highest point of the well casing and so note this fact in field logbook. Determine from the records and record in the notebook the elevation of this point.
3. Record any observations and remarks regarding the completion characteristics and well condition, such as evidence of cracked casing or surface seals, security of the well (locked cap), and evidence of tampering.
4. Keep all equipment and supplies protected from gross contamination; use clean plastic sheeting. Keep the water level indicator probe in its protective case when not in use.

3.2 OPERATION

1. Sample the air in the well head for gross organic vapors by lifting the well cap only high enough for an organic vapor meter (PID or FID) probe to be entered into the well casing. This will indicate the presence of gross volatile contaminants as well as indicating potential sampler exposure.

2. Remove cap. Allow well to vent for 60–90 seconds. Resample headspace. Record both readings. If the second reading is lower than the first, use the second reading to determining whether respiratory protection will be required during subsequent water level and well depth determinations and sampling.
3. Note that all headspace sampling must be performed at arm's length and from the upwind side of the well if possible.
4. If NAPL contamination is suspected, use an interface probe to determine the existence and thickness of NAPLs.
    - Open the probe housing, turn the probe on, and test the alarm. Slowly lower the probe into the well until the alarm sounds. A continuous alarm indicates a NAPL, while an intermittent alarm indicates water. If a NAPL is detected, record the initial level (first alarm). Mark the spot by grasping the cable with the thumb and forefingers at the top of the casing. If a mark is present on the casing, use the mark as the reference point. If no mark is present, use the highest point on the casing as the reference point. Withdraw the cable sufficiently to record the depth.
    - Continue to slowly lower the probe until it passes into the water phase. Slowly retract the probe until the NAPL alarm sounds and record that level in the manner as described above.
    - Record the thickness of the LNAPL (see Section 3.3.1).
    - Continue to slowly lower the interface probe through the water column to check for the presence of DNAPL.
    - Measure and record the thickness of the DNAPL layer (if any) as described above.
    - Slowly raise the interface probe, recording the depth to each interface as the probe is withdrawn. If there is a discrepancy in depths, clean the probe sensors and re-check the depths.
    - NOTE: Air-liquid interface depth is more reliable if probe is lowered into liquid. NAPL-water depths are more accurate if probe is moved from water into NAPL.
    - Always lower and raise interface probe slowly to prevent undue mixing of media.
    - Always perform NAPL check in wells installed in areas with suspected NAPL contamination. Always perform NAPL check if headspace test reveals presence of volatiles. Always perform NAPL check the first time a well is sampled. If a well has been sampled previously and no NAPLs were present and none of the proceeding conditions are met, the NAPL check may be omitted.
5. If no NAPL is present, use an electronic water level detector as follows.
    - Remove the water level indicator probe from the case, turn on the sounder, and test check the battery and sensitivity scale by pushing the red button. Adjust the sensitivity scale until you can hear the buzzer.
    - Slowly lower the probe and cable into the well, allowing the cable reel to unwind. Continue lowering until the meter buzzes. Very slowly, raise and lower the probe until the point is reached where the meter just buzzes. Marking the spot by grasping the cable with the thumb and forefingers at the top of the casing. If a mark is present on the casing, use the mark as the reference point. If no mark is present, use the highest point on the casing as the reference point. Withdraw the cable and record the depth.

6. To measure the well depth, lower electric water level indicator probe or tape until slack is noted. Very slowly raise and lower the cable until the exact bottom of the well is "felt." Measure (cable) or read the length (tape) and record the depth.
7. Note that if the electric water level indicator is used to determine depth of well, the offset distance between the tip of the probe and the electrode must be added to the reading to determine actual depth.
8. Withdraw the probe or tape.
9. Decontaminate the probe(s) and cable(s), in accordance with SOP 80.1.

3.3 DATA RECORDING AND MANIPULATION

Record the following information in the field logbook and appropriate sampling forms:

- Date and time;
- Weather;
- Method of measurement;
- Casing elevation;
- NAPL surface elevation = casing elevation - depth to NAPL;
- Apparent measured LNAPL thickness = depth to bottom of NAPL - depth to top of NAPL;
- Water level elevation = casing elevation - depth to water; and
- Well bottom elevation = casing elevation - depth to bottom (or read directly from tape).

4.0 CALIBRATION

No calibration is required. Ensure operability of electric water level indicator by testing sounder before use.

5.0 PRECAUTIONS

- Depending upon the device used, correction factors may be required for some measurements;
- Check instrument batteries before each use; and
- Exercise care not to break the seals at the top of the electric water level indicator probe.

6.0 REFERENCES

ASTM Standard D 4750-87. 1987. *Standard Test Method for Determining Subsurface Liquid Levels in a Borehole or Monitoring Well (Observation Well).*

M^{c}Alary, T. A., and Barker, J.F. 1987. "Volatilization Losses of Organics During Ground Water Sampling from Low Permeability Materials" in *Ground Water Monitoring Review.* Fall 1987.

Thornhill, Jerry T. 1989. *Accuracy of Depth to Groundwater Measurements;* in "EPA Superfund Ground Water Issue" EPA/540/4-89/002.

STANDARD OPERATING PROCEDURE 50.1
SAMPLE LABELS

1.0 SCOPE AND APPLICATION

Every sample will have a sample label uniquely identifying the sampling point and analysis parameters. The purpose of this standard operating procedure (SOP) is to delineate protocols for the use of sample labels. An example label is included as Figure 50.1-A. Other formats with similar levels of detail are acceptable.

2.0 MATERIALS

- Sample label; and
- Indelible marker.

3.0 PROCEDURE

The use of preprinted sample labels is encouraged and should be requested from the analytical support laboratory during planning activities.
As each sample is collected, fill out a sample label ensuring the following information has been collected:

- Project name;
- Sample ID: enter the SWMU number and other pertinent information concerning where the sample was taken. This information should be included in site-specific work plan addenda;
- Date of sample collection;
- Time of sample collection;
- Initials of sampler(s);
- Analyses to be performed (NOTE: Due to number of analytes, details of analysis should be arranged with lab *a priori*); and
- Preservatives (water samples only).

Double-check the label information to make sure it is correct. Detach the label, remove the backing and apply the label to the sample container. Cover the label with clear tape, ensuring that the tape completely encircles the container.

4.0 MAINTENANCE

Not applicable.

5.0 PRECAUTIONS

None.

6.0 REFERENCES

USEPA. 1998. *EPA Requirements for Quality Assurance Project Plans.* EPA/600/R-98/018, QA/R5, Final, Office of Research and Development, Washington, D.C.

FIGURE 50.1-A
SAMPLE LABEL

PROJECT NAME _____

SAMPLE ID _____

DATE: ___/___/___ TIME: ____:____

ANALYTES: VOC SVOC P/P METALS CN
PAH D/F HERBs ANIONS TPH
ALK TSS

PRESERVATIVE: [HCl] [HNO_3] [NaOH] [H_2SO_4]

SAMPLER: _____

STANDARD OPERATING PROCEDURE 50.2
SAMPLE PACKAGING

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for the packing and shipping of samples to the laboratory for analysis.

2.0 MATERIALS

- Waterproof coolers (hard plastic or metal);
- Metal cans with friction-seal lids (e.g., paint cans);
- Chain-of-custody forms;
- Chain-of-custody seals (optional);
- Packing material;
- Sample documentation;
- Ice;
- Plastic garbage bags;
- Clear Tape;
- Zip-top plastic bags; and
- Temperature blanks provided by laboratory for each shipment.

3.0 PROCEDURE

1. Check cap tightness and verify that clear tape covers label and encircles container.
2. Wrap sample container in bubble wrap or closed cell foam sheets. Samples may be enclosed in a secondary container consisting of a clear zip-top plastic bag. Sample containers must be positioned upright and in such a manner that they will not touch during shipment.
3. Place several layers of bubble wrap, or at least 1 in. of vermiculite on the bottom of the cooler. Line cooler with open garbage bag, place all the samples upright inside the garbage bag and tie.
4. Double bag and seal loose ice to prevent melting ice from soaking the packing material. Place the ice outside the garbage bags containing the samples.
5. Pack shipping containers with packing material (closed-cell foam, vermiculite, or bubble wrap). Place this packing material around the sample bottles or metal cans to avoid breakage during shipment.
6. A temperature blank (provided by laboratory) will be included in each shipping container to monitor the internal temperature. Samples should be cooled to 4 degrees C on ice immediately after sampling.

7. Enclose all sample documentation (i.e., Field Parameter Forms, Chain-of-Custody forms) in a waterproof plastic bag and tape the bag to the underside of the cooler lid. If more than one cooler is being used, each cooler will have its own documentation. Add the total number of shipping containers included in each shipment on the chain-of-custody form.
8. Seal the coolers with signed and dated custody seals so that if the cooler were opened, the custody seal would be broken. Place clear tape over the custody seal to prevent damage to the seal.
9. Tape the cooler shut with packing tape over the hinges and place tape over the cooler drain.
10. Ship all samples via overnight delivery on the same day they are collected if possible.

4.0 MAINTENANCE

Not applicable.

5.0 PRECAUTIONS

5.1 PERMISSIBLE PACKAGING MATERIALS

- Non-absorbent
  — Bubble wrap; and
  — Closed cell foam packing sheets.
- Absorbent
  — Vermiculite.

5.2 NON-PERMISSIBLE PACKAGING MATERIALS

- Paper;
- Wood shavings (excelsior); and
- Cornstarch "peanuts".

6.0 REFERENCES

USEPA. 1990. *Sampler's Guide to the Contract Laboratory Program.* EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C., December 1990.

USEPA. 1991. *User's Guide to the Contract Laboratory Program.* EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response. January 1991.

USEPA. 1998. *EPA Requirements for Quality Assurance Project Plans.* EPA/600/R-98/018, QA/R5, Final, Office of Research and Development, Washington, D.C

STANDARD OPERATING PROCEDURE 70.1
INVESTIGATION-DERIVED MATERIAL

1.0 SCOPE AND APPLICATION

Management of investigation-derived material (IDM) minimizes the potential for the spread of waste material onsite or offsite through investigation activities. The purpose of this standard operating procedure (SOP) is to provide general guidelines for appropriate management of potentially contaminated materials derived from the field investigations. Specific procedures related to the transportation and disposal of hazardous waste are beyond the scope of this SOP.

2.0 INTRODUCTION

Investigation derived material (IDM) consists of waste materials that are known or suspected to be contaminated with waste substances through the actions of sample collection or personnel and equipment decontamination. These materials include decontamination solutions, disposable equipment, drill cuttings and fluids, and water from groundwater monitoring well development and purging. To the extent possible, the site manager will attempt to minimize the generation of these materials through careful design of decontamination schemes and groundwater sampling programs. Testing conducted on soil and water investigation-derived material will show if they are also hazardous wastes as defined by RCRA. This will determine the proper handling and ultimate disposal requirements.

The criteria for designating a substance as hazardous waste according to RCRA is provided in 40 CFR 261.3. If IDM meet these criteria, RCRA requirements will be followed for packaging, labeling, transporting, storing, and record keeping as described in 40 CFR 262.34. Those materials that are judged potentially to meet the criteria for a regulated solid or hazardous waste will be placed in DOT-approved 55-gallon steel drums or another type of DOT approved container; based on waste characteristics and volume.
Investigation-derived material will be appropriately placed in containers, labeled, and tested to determine disposal options in accordance with RCRA regulations and Virginia Hazardous Waste Management Regulations.

3.0 INVESTIGATION-DERIVED MATERIAL MANAGEMENT

Procedures that minimize potential for the spread of waste material include minimizing the volume of material generated, material segregation, appropriate storage, and disposal according to RCRA requirements.

3.1 WASTE MINIMIZATION

In the development of work plan addenda, each aspect of the investigation will be reviewed to identify areas where excess waste generation can be eliminated. General procedures that will eliminate waste include avoidance of unnecessary exposure of materials to hazardous material and coordination of sampling schedules to avoid repetitious purging of wells and use of sampling equipment.

3.2 WASTE SEGREGATION

Waste accumulation and management procedures to be used depend upon the type of material generated. For this reason, IDM described below are segregated into separate 55-gallon storage drums or other appropriate DOT containers. Waste materials that are known to be free of potential hazardous waste contamination (such as broken sample bottles or equipment containers and wrappings) must be collected separately for disposal to municipal systems. Large plastic garbage or "lawn and leaf" bags are useful for collecting this trash. Even "clean" sample bottles or Tyvek should be disposed of with care. Although they are not legally a problem, if they are discovered by the public they may cause concern. Therefore, items that are known to be free from contamination but are also known to represent "hazardous or toxic waste" to the public must not be disposed of in any public trash receptacle, such as found at your hotel or park.

3.2.1 Decontamination Solutions

Solutions considered investigation-derived materials range from detergents, organic solvents, and acids used to decontaminate small hand samplers to steam-cleaning rinsate used to wash drill rigs and other large equipment. These solutions are to be placed in 55-gallon drums with bolt-sealed lids or other appropriate DOT approved containers. Residual liquid IDM from decontamination pads will be removed and appropriately placed in container(s) at the end of each field day.

3.2.2 Soil Cuttings and Drilling Muds

Soil cuttings are solid to semi-solid soils generated during trenching activities or drilling for the collection of subsurface soil samples or the installation of monitoring wells. Depending on the type of drilling, drilling fluids known as "muds" may be used to remove soil cuttings. Drilling fluids flushed from the borehole must be directed into a settling section of a mud pit. This allows reuse of the decanted fluids after removal of the settled sediments. Drill cuttings, whether generated with or without drilling fluids, are to be removed with a flat-bottomed shovel and placed in 55-gallon drums with bolt-sealed lids or other appropriate DOT containers, as conditions or volume of IDM dictate.

3.2.3 Well Development and Purge Water

Well development and purge water is removed from monitoring wells to repair damage to the aquifer following well installation, obtain characteristic aquifer groundwater samples, or measure aquifer hydraulic properties. The volume of groundwater to be generated will determine the appropriate container to be used for accumulation of IDM.

For well development and purging, 55-gallon drums are typically an efficient container for accumulation. When larger volumes of water are removed from wells, such as when pumping tests are conducted, the use of large-volume portable tanks such as "Baker Tanks" should be considered for IDM accumulation.

Analytical data for groundwater samples associated with the well development and purge water will be used to assist in characterizing IDM and evaluating disposal options.

3.2.4 Personal Protective Equipment and Disposable Sampling Equipment

Personal protective equipment and clothing (PPE) may include such items as Tyvek coveralls, gloves, booties, and APR cartridges. Disposable sampling equipment may include such items as plastic sheeting, bailers, disposable filters, disposable tubing and paper towels. PPE and disposable sampling equipment that have or may have contacted contaminated media (soil, water, etc.) will be segregated and placed in 55-gallon drums separate from soil and water IDM. Disposition of this type of IDM will be determined by the results of IDM testing of the media in which the PPE and sampling equipment contacted.

3.3 MATERIAL ACCUMULATION

The IDM in containers must be placed in an appropriate designated RCRA container accumulation area at RFAAP, where it is permissible to accumulate such waste. IDM placed into a designated 90-day accumulation area will be properly sealed, labeled and covered. All drums will be placed on pallets.

A secure and controlled waste staging area will be designated by the installation prior the commencement of field sampling activities. Per the facility's requirements as a RCRA large quantity generator, waste accumulation cannot exceed 90 days for materials presumed or shown to be RCRA-designated hazardous wastes; waste which is known not to be RCRA-designated waste should be promptly disposed to municipal waste systems or appropriate facility.

3.3.1 IDM Accumulation Containers

Containers will be DOT-approved (DOT 17H 18/16GA OH unlined) open-head steel drums or other DOT approved container, as appropriate.

Container lids should lift completely off be secured by a bolt ring (for drum). Order enough containers to accumulate all streams of expected IDM including soil, PPE and disposable sampling equipment, decontamination water, purge water, etc.

Solid and liquid waste streams will not be mixed in a container. PPE and expendable sampling equipment will be segregated from other IDM and placed in different containers than soil. Containers inside containers are not permitted. PPE must be placed directly in a drum not in a plastic bag.

Pallets are often required to allow transport of filled drums to the staging area with a forklift. Normal pallets are 3×4 ft and will hold two to three 55-gallon drums depending on the filled weight. If pallets are required for drum transport or storage, field personnel are responsible for ensuring that the empty drums are placed on pallets before they are filled and that the lids are sealed on with the bolt-tighten ring after the drums are filled. Because the weight of one drum can exceed 500 lbs, under no circumstances should personnel attempt to move the drums by hand.

3.3.2 Container Labeling

Each container that is used to accumulate IDM will be appropriately labeled at the time of accumulation and assigned a unique identification number for tracking purposes. The following information will be written in permanent marker on a drum label affixed on the exterior side at a location at least two-thirds of the way up from the bottom of the drum.

- Facility name.
- Accumulation start date and completion date.
- Site identifier information (SWMU, boring, well, etc.).
- Description of IDM.
- Drum ID No.

4.0 MATERIAL CHARACTERIZATION AND DISPOSAL

IDM will be characterized and tested to determine whether it is a hazardous waste as defined by 40 CFR Part 261 and to determine what disposal options exist in accordance with RCRA regulations and the Virginia Hazardous Waste Management Regulations (VHWMR).

In general, IDM will be considered a hazardous waste if it contains a listed hazardous waste or if the IDM exhibits a characteristic of hazardous waste.

Work plan addenda will identify the appropriate characterization and testing program for IDM based on the following:

- Site-specific conditions related to chemicals of concern, etc.
- The nature and quantity of expected IDM to be generated during site-specific investigations.
- Applicable Federal, State, and local regulations, such as RCRA, VHWMR regulations and policies and procedures, and Army Regulation 200-1.
- RFAAP specific requirements and policies for IDM characterization and disposal at the time of the investigation.

In general, appropriate USEPA SW 846 Test Methods for Evaluating Solid Waste will be used for testing IDM and will be specified in work plan addenda. Other appropriate test methods may be specified by RFAAP in addition to SW 846 Methods that are specific to installation operations, the site of interest (percent explosive content, reactivity, etc.), or requirements for disposal at RFAAP water treatment facilities or publicly owned treatment works.

Responsibility for the final disposal of IDM will be determined before field activities are begun and will be described in work plan addenda. Off-site disposal of IDM will be coordinated with RFAAP (generator) to ensure appropriate disposition. The contractor will coordinate IDM transportation and disposal activities for RFAAP (generator).

At the direction of RFAAP, appropriate waste manifests will be prepared by the USACE contractor or Alliant Techsystems subcontractor for transportation and disposal. Alliant Techsystems or other appropriate RFAAP entity will be listed as the generator and an appointed representative from RFAAP will review and sign the manifest for offsite disposal.

RFAAP will make the final decision on the selection of the transporter, storage, and disposal facility (TSDFs) or recycling facility. RFAAP will provide the contractor a listing of previously used TSDFs for priority consideration. Proposed facilities that are not included on the listing are required to provide a copy of the TSDFs most recent state or federal inspection to the installation. Waste characterization and testing results will be submitted to RFAAP (generator) for review and approval before final disposition of the material.

Hazardous waste: Prior to final disposition, a hazardous waste manifest will be furnished by the TSDF to accompany transport to the disposal facility. Following final disposition, a certificate of disposal will be furnished by the disposal facility. Copies of the manifests and certificates of disposal are to be provided to RFAAP and retained on file by the contractor or subcontractor.

4.0 PRECAUTIONS

- Because the weight of one drum can exceed 500 lbs, under no circumstances should personnel attempt to move drums by hand.
- Refer to the site-specific health and safety plan when managing IDM.

5.0 REFERENCES

Safety Rules for Contractors and Subcontractors, 1995. Alliant Techsystems, Incorporated, Radford Army Ammunition Plant.

STANDARD OPERATING PROCEDURE 80.1
DECONTAMINATION

1.0 SCOPE AND APPLICATION

Before leaving the site, all personnel or equipment involved in intrusive sampling or having entered a hazardous waste site during intrusive sampling must be thoroughly decontaminated to prevent adverse health effects and minimize the spread of contamination. Equipment must be decontaminated between sites to preclude cross-contamination. Decontamination water will be free of contaminants as evidenced through either chemical analyses or certificates of analysis. This standard operating procedure (SOP) describes general decontamination requirements for site personnel and sampling equipment. Decontamination procedures for contaminants requiring a more stringent procedure, e.g., dioxins/furans, will be included in site-specific addenda.

2.0 MATERIALS

- Plastic sheeting, buckets or tubs, pressure sprayer, rinse bottles, and brushes;
- U.S. Army Corps of Engineers or installation approved decontamination water source;
- Deionized ultra-filtered, HPLC-grade organic free water (DIUF);
- Non-phosphate laboratory detergent;
- Nitric Acid, 0.1 Normal (N) solution;
- Pesticide-grade solvent, Methanol;
- Aluminum foil;
- Paper towels;
- Plastic garbage bags; and
- Appropriate containers for management of investigation-derived material (IDM).

3.0 PROCEDURE

3.1 SAMPLE BOTTLES

At the completion of each sampling activity the exterior surfaces of the sample bottles must be decontaminated as follows:

- Be sure that the bottle lids are on tight.
- Wipe the outside of the bottle with a paper towel to remove gross contamination.

3.2 PERSONNEL DECONTAMINATION

Review the site-specific health and safety plan for the appropriate decontamination procedures.

3.3 EQUIPMENT DECONTAMINATION

3.3.1 Drilling Rigs

Drilling rigs and associated equipment, such as augers, drill casing, rods, samplers, tools, recirculation tank, and water tank (inside and out), will be decontaminated before site entry, after over-the-road mobilization and immediately upon departure from a site after drilling a hole. Supplementary cleaning will be performed before site entry. There is a likelihood that contamination has accumulated on tires and as spatter or dust en route from one site to the next.

1. Place contaminated equipment in an enclosure designed to contain all decontamination residues (water, sludge, etc.).
2. Steam-clean equipment until all dirt, mud, grease, asphaltic, bituminous, or other encrusting coating materials (with the exception of manufacturer-applied paint) has been removed.
3. Water used will be taken from an approved source.
4. When cross-contamination from metals is a concern, rinse sampling components such as split spoons, geo-punch stems, and augers with nitric acid, 0.1N.
5. Rinse with DIUF water.
6. When semi-volatile and non-volatile organics may be present, rinse the sampling components with pesticide-grade solvent methanol.
7. Double rinse the sampling components with DIUF water.
8. Decontamination residues and fluids will be appropriately managed as IDM per work plan addenda and SOP 80.1.

3.3.2 Well Casing and Screen

Prior to use, well casing and screen materials will be decontaminated. This activity will be performed in the leak proof, decontamination pad, which will be constructed prior to commencement of the field investigation. The decontamination process will include:

- Steam cleaning with approved source water.
- Rinse with DUIF water.
- Air-dry on plastic sheeting.
- Wrap in plastic sheeting to prevent contamination during storage/transit.

3.3.3 Non Dedicated Submersible Pumps Used for Purging and Sampling

1. Scrub the exterior of the pump to remove gross (visible) contamination using appropriate brushes, approved water, and non-phosphate detergent (steam cleaning may be substituted for detergent scrub).
2. Pump an appropriate amount of laboratory detergent solution (minimum 10 gallons) to purge and clean the interior of the pump.
3. Rinse by pumping no less than 10 gallons of approved water to rinse.
4. Rinse the pump exterior with approved decontamination water.
5. When cross-contamination from metals is a concern, rinse the pump exterior with approved nitric acid 0.1N solution.
6. Rinse the pump exterior with DIUF water.

7. When semi-volatile and non-volatile organics may be present, rinse the pump exterior with pesticide-grade solvent methanol.
8. Double rinse the pump exterior with DIUF water.
9. Air-dry on aluminum foil or clean plastic sheeting.
10. Wrap pump in aluminum foil or clean plastic sheeting, or store in a clean, dedicated PVC or PTFE storage container.
11. Solutions and residuals generated from decontamination activities will be managed appropriately as IDM per work plan addenda and SOP 80.1.

3.3.4 Sample Equipment and Measuring Water Level Devices

1. Scrub the equipment to remove gross (visible) contamination using appropriate brush (es), approved water, and non-phosphate detergent.
2. Rinse with approved source water.
3. When cross-contamination from metals is a concern, rinse the sampling equipment with approved nitric acid 0.1N solution.
4. Rinse equipment with DIUF water.
5. When semi-volatile and non-volatile organics may be present, rinse the sampling equipment with pesticide-grade solvent methanol.
6. Double rinse the sampling equipment with DIUF water.
7. Air-dry on aluminum foil or clean plastic sheeting.
8. Wrap in aluminum foil, clean plastic sheeting, or zip top bag or store in a clean, dedicated PVC or PTFE storage container.
9. Solutions and residuals generated from decontamination activities will be managed appropriately as IDM per work plan addenda and SOP 80.1.

3.3.5 Other Sampling and Measurement Probes

Temperature, pH, conductivity, Redox, and dissolved oxygen probes will be decontaminated according to manufacturer's specifications. If no such specifications exist, remove gross contamination and triple-rinse probe with DIUF water.

4.0 PRECAUTIONS

- Manage IDM appropriately according to the requirements specified in work plan addenda.
- Follow appropriate procedures as specified in the site-specific health and safety plan.

5.0 REFERENCES

USACE. 2001. Requirements for the Preparation of Sampling and Analysis Plans. EM 200-1-3. 1 February.

STANDARD OPERATING PROCEDURE 90.1
PHOTOIONIZATION DETECTOR (HNu Model PI–101 and HW–101)

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for field operations with a photoionization detector (HNu Systems Model PI–101 or HW–101). The photoionization detector (PID) detects total ionizables; hence it is used to monitor both organic and inorganic vapors and gases to determine relative concentrations of air contaminants. This information is used to establish level of protection and other control measures such as action levels. The PID cannot effectively detect compounds having ionization potentials above the photon energy level of the lamp used; therefore, methane, which has an ionization potential of 12.98 eV, is undetectable by PIDs because the lamps produce 9.5, 10.2, or 11.7 eV.

Use of brand names in this SOP is in not intended as an endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc., are acceptable. If alternate equipment is to be used, the contractor shall provide applicable and comparable SOPs for its maintenance and calibration.

2.0 MATERIALS

- HNu Systems Model PI–101 or HW–101 survey probe with 9.5, 10.2, or 11.7 eV lamp;
- Lead-acid gel-cell battery;
- Calibration gas (e.g., isobutylene, 101 ppm) with regulator;
- Tygon tubing;
- Tedlar bag (optional);
- Instrument logbook; and
- Field logbook.

3.0 PROCEDURE

These procedures are to be followed when using the HNu in the field.

3.1 STARTUP

1. Before attaching the probe, check the function switch on the control panel to ensure that it is in the off position. Attach the probe by plugging it into the interface on the top of the readout module.
2. Turn the function switch to the battery check position. The needle on the meter should read within or above the green battery arc on the scale; if not, recharge the battery. If the red indicator light comes on, the battery needs recharging or service may be indicated.
3. Turn the function switch to any range setting. Listen for the hum of the fan motor. Check meter function by holding a solvent-based marker pen near the sample intake. If there is no needle deflection, look briefly into the end of the probe (no more than 1 or 2 sec) to see if the lamp is on; if it is on, it will give a purple glow. Do not stare into the probe any longer than 2 sec. Long-term exposure to UV light can damage the eyes. (See further information in Section 5.)

4. To zero the instrument, turn the function switch to the standby position and rotate the zero adjustment until the meter reads zero. A calibration gas is not needed since this is an electronic zero adjustment. If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted if necessary. Allow the instrument to warm up for 3–5 min to ensure that the zero reading is stable. If necessary, readjust the zero.

3.2 OPERATIONAL CHECK

Follow the startup procedure in Section 3.1.
With the instrument set on the 0–20 range, hold a solvent-based marker near the probe tip. If the meter deflects upscale, the instrument is working.

3.3 FIELD CALIBRATION PROCEDURE

1. Follow the startup procedures in Section 3.1 and the operational check in Section 3.2.
2. Set the function switch to the range setting for the concentration of the calibration gas.
3. Attach a regulator HNu P/N 101-351 or equivalent (flow = 200 to 300 ml/min) to a disposable cylinder of isobutylene (HNu 101-351 or equivalent). Connect the regulator to the probe of the HNu with a piece of clean Tygon tubing. Turn on the valve of the regulator.
4. After 5 sec, adjust the span dial until the meter reading equals the benzene concentration of the calibration gas used, corrected to its equivalence, which should be marked on the canister (Isobutylene ~0.7X benzene).
5. Record in the field log the instrument ID No., serial No., initial and final span settings, date, time, location, concentration and type of calibration gas used, and the signature of the person who calibrated the instrument.
6. If the HNu does not function or calibrate properly, the project equipment manager is to be notified as soon as possible. Under no circumstances is work requiring monitoring with a PI–101 or HW–101 to be done with a malfunctioning instrument.

3.4 CALIBRATION TO A GAS OTHER THAN ISOBUTYLENE

The HNu may be calibrated to any certified calibration gas. However, after calibration, all subsequent instrument readings will be relative to the calibration gas used. General procedures include the following:

1. Calibrate according to procedure 3.3.
2. Partially fill and flush one-to-two times a gas bag (Tedlar recommended) with the certified National Institute of Standards and Technology (NIST) (formerly NBS) traceable calibration gas. Then fill the bag with 1–3 L of the calibration gas. If the gas is toxic, this must be done in a fume hood.
3. Feed the calibration gas into the probe with the range set for the value of the gas. After 5 sec, adjust the span control until the meter reads the value of the calibration gas.
4. Record the results of the calibration on the calibration/maintenance log and attach a new calibration sticker (if available) or correct the existing sticker to reflect the new calibration data. All subsequent readings will be relative to the new calibration gas.

3.5 OPERATION

1. Follow the startup procedure, operational check, and calibration check (refer to Section 3.1).

2. Set the function switch to the appropriate range. If the concentration of gas vapors is unknown, set the function switch to 0-20 ppm range. Adjust if necessary.
3. Prevent exposing the HNu to excessive moisture, dirt, or contaminant while monitoring the work activity as specified in the Site Health and Safety Plan.
4. When the activity is completed, or at the end of the day, carefully clean the outside of the HNu with a damp disposable towel to remove all visible dirt. Return the HNu to a secure area and place on charge. Charge after each use; the lead acid batteries cannot be ruined by over charging.
5. With the exception of the probe's inlet and exhaust, the HNu can be wrapped in clear plastic to prevent it from becoming contaminated and to prevent water from getting inside in the event of precipitation. If the instrument becomes contaminated, make sure to take necessary steps to decontaminate it. Call the Equipment Administrator if necessary; under no circumstances should an instrument be returned from the field in a contaminated condition.

4.0 MAINTENANCE

Calibration/maintenance logs are to be filled in completely whenever a PI-101 or HW-101 receives servicing. This is true of both contractor-owned and rental instruments.

The equipment manager should be called to arrange for a fresh instrument when necessary. The contractor's equipment facility is responsible for arranging all repairs that cannot be performed by the project equipment manager.

4.1 ROUTINE SERVICE

The PID's performance is affected by a number of factors. These include but are not limited to the decay of the UV lamp output over time and the accumulation of dust and other particulate material and contaminates on the lamp and in the ion chamber. Because of these factors, the PID should not be left in the field for a period of more than 2 weeks before being replaced with a fresh instrument. If a site is going to be inactive for a period of more than a week, all monitoring instruments are to be returned to the project equipment manager or his trained designee for servicing and/or reassignment. The following procedures are to be performed at the designated intervals for routine service.

Procedure	Frequency
Operational check	Before use and at instrument return
Field calibration	Before use and at instrument return
Full calibration	Bi-weekly (return instrument to equipment manager for replacement with a fresh unit)
Clean UV lamp and	Bi-weekly or as needed ion chamber
Replace UV Lamp	As needed

4.1.1 UV Lamp and Ion Chamber Cleaning

During periods of analyzer operation, dust and other foreign materials are drawn into the probe forming deposits on the surface of the UV lamp and in the ion chamber. This condition is indicated by meter readings that are low, erratic, unstable, non-repeatable, or drifting and show apparent moisture sensitivity. These

deposits interfere with the ionization process and cause erroneous readings. Check for this condition regularly to ensure that the HNu is functioning properly. If the instrument is malfunctioning, call your equipment manager to arrange to have a fresh replacement.

4.1.2 Lamp eV Change

If different applications for the analyzer would require different eV lamps, separate probes, each with its own eV lamp, must be used. A single readout assembly will serve for any of the probes (9.5, 10.2, and 11.7 eV). A change in probe will require resetting of the zero control and recalibrating the instrument. The 11.7 eV lamp will detect more compounds than either of the two lower eV lamps. However, the 11.7 eV probe needs more frequent calibration; it burns out much faster than the lower eV lamps.

5.0 PRECAUTIONS

- The HNu PI–101 and HW–101 are designed to sample air or vapors only. *Do not allow any liquids or low boiling vapors to get into the probe or meter assembly.*
- High concentrations of any gas can cause erroneous readings. High humidity can also cause the instrument readings to vary significantly from the actual concentration of gases or vapors present. This is true even through the HNu cannot react to water vapor.
- High humidity, dust, and exposure to concentrations of low boiling vapors will contaminate the ion chamber, causing a steady decrease in sensitivity.
- Continued exposure to ultraviolet light generated by the light source can be harmful to eyesight. If a visual check of the UV lamp is performed *do not look at the light source from a distance closer than 6 inches with unprotected eyes.* Use eye protection (UV-blocking sunglasses or safety glasses). Only look briefly—never more than about 2 sec.

  Place the instrument on charge after each use; the lead batteries cannot be ruined by over charging.
- If at any time the instrument does not check out or calibrate properly in the field, the equipment manager is to be notified immediately and a replacement obtained for the malfunctioning instrument. Under no circumstances should fieldwork requiring continuous air monitoring for organic vapors and/or gases be done with a malfunctioning Hnu or without a HNu or an approved comparable instrument.

6.0 REFERENCES

Manufacturer's Equipment Manual.

ATTACHMENT B

Laboratory QC Limits
(on CD)

6010-Solid

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Aluminum	7429-90-5	REG	mg/kg	10	20	80	120	20
Zirconium	7440-67-7	REG	mg/kg	12.5	25	80	120	20
Arsenic	7440-38-2	REG	mg/kg	0.5	5	80	120	20
Barium	7440-39-3	REG	mg/kg	0.1	0.5	80	120	20
Beryllium	7440-41-7	REG	mg/kg	0.0125	0.5	80	120	20
Boron	7440-42-8	REG	mg/kg	2.5	5	80	120	20
Cadmium	7440-43-9	REG	mg/kg	0.05	0.5	80	120	20
Calcium	7440-70-2	REG	mg/kg	5	10	80	120	20
Chromium	7440-47-3	REG	mg/kg	0.125	1	80	120	20
Cobalt	7440-48-4	REG	mg/kg	0.125	1	80	120	20
Copper	7440-50-8	REG	mg/kg	0.5	1	80	120	20
Iron	7439-89-6	REG	mg/kg	1	3	80	120	20
Lead	7439-92-1	REG	mg/kg	0.5	5	80	120	20
Lithium	7439-93-2	REG	mg/kg	2.5	5	80	120	20
Magnesium	7439-95-4	REG	mg/kg	12	25	80	120	20
Manganese	7439-96-5	REG	mg/kg	0.1	0.5	80	120	20
Molybdenum	7439-98-7	REG	mg/kg	1.5	5	80	120	20
Nickel	7440-02-0	REG	mg/kg	0.5	2	80	120	20
Potassium	7440-09-7	REG	mg/kg	25	50	80	120	20
Selenium	7782-49-2	REG	mg/kg	0.5	5	80	120	20
Silver	7440-22-4	REG	mg/kg	0.25	2	80	120	20
Sodium	7440-23-5	REG	mg/kg	5	25	80	120	20
Strontium	7440-24-6	REG	mg/kg	0.25	0.5	80	120	20
Thallium	7440-28-0	REG	mg/kg	1	25	80	120	20
Tin	7440-31-5	REG	mg/kg	5	25	80	120	20
Titanium	7440-32-6	REG	mg/kg	0.5	2	80	120	20
Vanadium	7440-62-2	REG	mg/kg	0.25	0.5	80	120	20
Zinc	7440-66-6	REG	mg/kg	0.5	1	80	120	20
Phosphorus	7723-14-0	REG	mg/kg	25	50	80	120	20
Antimony	7440-36-0	REG	mg/kg	0.5	10	80	120	20

6010-Water

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Calcium	7440-70-2	REG	mg/L	0.1	0.2	85	115	20
Vanadium	7440-62-2	REG	mg/L	0.005	0.01	85	115	20
Cobalt	7440-48-4	REG	mg/L	0.0025	0.02	85	115	20
Copper	7440-50-8	REG	mg/L	0.005	0.02	85	115	20
Iron	7439-89-6	REG	mg/L	0.025	0.1	85	115	20
Lead	7439-92-1	REG	mg/L	0.01	0.1	85	115	20
Lithium	7439-93-2	REG	mg/L	0.05	0.1	85	115	20
Magnesium	7439-95-4	REG	mg/L	0.25	0.5	85	115	20
Manganese	7439-96-5	REG	mg/L	0.005	0.01	85	115	20
Molybdenum	7439-98-7	REG	mg/L	0.005	0.1	85	115	20
Nickel	7440-02-0	REG	mg/L	0.005	0.04	85	115	20
Potassium	7440-09-7	REG	mg/L	0.25	1	85	115	20
Selenium	7782-49-2	REG	mg/L	0.04	0.08	85	115	20
Silicon	7440-21-3	REG	mg/L	0.25	1	85	115	20
Silver	7440-22-4	REG	mg/L	0.005	0.01	85	115	20
Sodium	7440-23-5	REG	mg/L	0.25	0.5	85	115	20
Strontium	7440-24-6	REG	mg/L	0.005	0.01	85	115	20
Thallium	7440-28-0	REG	mg/L	0.1	1	85	115	20
Zinc	7440-66-6	REG	mg/L	0.005	0.02	85	115	20
Aluminum	7429-90-5	REG	mg/L	0.05	0.1	85	115	20
Antimony	7440-36-0	REG	mg/L	0.05	0.2	85	115	20
Arsenic	7440-38-2	REG	mg/L	0.01	0.1	85	115	20
Barium	7440-39-3	REG	mg/L	0.0025	0.01	85	115	20
Beryllium	7440-41-7	REG	mg/L	0.0005	0.01	85	115	20
Boron	7440-42-8	REG	mg/L	0.05	0.1	85	115	20
Cadmium	7440-43-9	REG	mg/L	0.0025	0.01	85	115	20
Silica, Calculated as SiO2		REG	mg/L	0.25	1	85	115	20
Phosphorus	7723-14-0	REG	mg/L	0.5	1	85	115	20
Zirconium	7440-67-7	REG	mg/L	0.25	0.5	85	115	20
Tin	7440-31-5	REG	mg/L	0.05	0.5	85	115	20
Titanium	7440-32-6	REG	mg/L	0.005	0.03	85	115	20
Chromium	7440-47-3	REG	mg/L	0.0025	0.02	85	115	20

6020-Solid

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Arsenic, Total	7440-38-2	REG	mg/kg	0.075	0.3	80	120	20
Uranium, Total	7440-61-1	REG	mg/kg	0.1	0.4	80	120	20
Antimony, Total	7440-36-0	REG	mg/kg	0.05	0.1	80	120	20
Selenium, Total	7782-49-2	REG	mg/kg	0.1	0.2	80	120	20
Thallium, Total	7440-28-0	REG	mg/kg	0.01	0.02	80	120	20
Silver, Total	7440-22-4	REG	mg/kg	0.05	0.2	80	120	20
Barium, Total	7440-39-3	REG	mg/kg	0.075	0.3	80	120	20
Cadmium, Total	7440-43-9	REG	mg/kg	0.025	0.1	80	120	20
Cobalt, Total	7440-48-4	REG	mg/kg	0.125	0.5	80	120	20
Chromium, Total	7440-47-3	REG	mg/kg	0.1	0.4	80	120	20
Copper, Total	7440-50-8	REG	mg/kg	0.15	0.6	80	120	20
Manganese, Total	7439-96-5	REG	mg/kg	0.05	0.2	80	120	20
Nickel, Total	7440-02-0	REG	mg/kg	0.2	0.8	80	120	20
Vanadium, Total	7440-62-2	REG	mg/kg	0.125	0.5	80	120	20
Zinc, Total	7440-66-6	REG	mg/kg	0.625	2.5	80	120	20
Lead, Total	7439-92-1	REG	mg/kg	0.1	0.2	80	120	20

6020-Water

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Arsenic	7440-38-2	REG	mg/L	0.00025	0.001	80	120	20
Uranium	7440-61-1	REG	mg/L	0.00025	0.001	80	120	20
Selenium	7782-49-2	REG	mg/L	0.0005	0.001	80	120	20
Silver	7440-22-4	REG	mg/L	0.00025	0.001	80	120	20
Thallium	7440-28-0	REG	mg/L	0.00005	0.0002	80	120	20
Barium	7440-39-3	REG	mg/L	0.0005	0.003	80	120	20
Cadmium	7440-43-9	REG	mg/L	0.000125	0.0005	80	120	20
Cobalt	7440-48-4	REG	mg/L	0.00025	0.001	80	120	20
Chromium	7440-47-3	REG	mg/L	0.0005	0.002	80	120	20
Copper	7440-50-8	REG	mg/L	0.0005	0.002	80	120	20
Manganese	7439-96-5	REG	mg/L	0.0005	0.002	80	120	20
Nickel	7440-02-0	REG	mg/L	0.001	0.004	80	120	20
Vanadium	7440-62-2	REG	mg/L	0.00025	0.001	80	120	20
Zinc	7440-66-6	REG	mg/L	0.005	0.025	80	120	20
Antimony	7440-36-0	REG	mg/L	0.00025	0.001	80	120	20
Lead	7439-92-1	REG	mg/L	0.00025	0.001	80	120	20

Microbac Laboratories Inc.
List Definitions Report

AUGUST 23, 2010 12:08

List Type: 6850
List Function: ALL
List Matrix Class: WATER
List Join ID: LJ56434
Methodref: 6850
PKey: STD Description: STD

Compound	CAS #	Parm Type	Units	MDL	RL	QCKEY	L_LCS	U_LCS
Perchlorate	14797-73-0	REG	ug/L	.1	.2	STD	80	120
O18LP		STD	ug/L			STD	0	0

Microbac Laboratories Inc.
List Definitions Report

AUGUST 27, 2010 13:24

List Type: 8081
List Function: ALL
List Matrix Class: SOLID
List Join ID: LJ65199
Methodref: 8081
PKey: 2415.034 Description:

Compound	CAS #	Parm Type	Units	MDL	RL	QCKEY	L_LCS	U_LCS
4,4'-DDD	72-54-8	REG	ug/kg	.33	1.65	DOD4	30	135
4,4'-DDE	72-55-9	REG	ug/kg	.33	1.65	DOD4	70	125
4,4'-DDT	50-29-3	REG	ug/kg	.33	1.65	DOD4	45	140
Aldrin	309-00-2	REG	ug/kg	.33	1.65	DOD4	45	140
alpha Chlordane	5103-71-9	REG	ug/kg	.33	1.65	DOD4	65	120
alpha-BHC	319-84-6	REG	ug/kg	.33	1.65	DOD4	60	125
beta-BHC	319-85-7	REG	ug/kg	.33	1.65	DOD4	60	125
delta-BHC	319-86-8	REG	ug/kg	.33	1.65	DOD4	55	130
Dieldrin	60-57-1	REG	ug/kg	.33	1.65	DOD4	65	125
Endosulfan I	959-98-8	REG	ug/kg	.33	1.65	DOD4	15	135
Endosulfan II	33213-65-9	REG	ug/kg	.33	1.65	DOD4	35	140
Endosulfan sulfate	1031-07-8	REG	ug/kg	.33	1.65	DOD4	60	135
Endrin	72-20-8	REG	ug/kg	.33	1.65	DOD4	60	135
Endrin aldehyde	7421-93-4	REG	ug/kg	.33	1.65	DOD4	35	145
Endrin ketone	53494-70-5	REG	ug/kg	.33	1.65	DOD4	65	135
gamma Chlordane	5103-74-2	REG	ug/kg	.33	1.65	DOD4	65	125
gamma-BHC (Lindane)	58-89-9	REG	ug/kg	.33	1.65	DOD4	60	125
Heptachlor	76-44-8	REG	ug/kg	.33	1.65	DOD4	50	140
Heptachlor epoxide	1024-57-3	REG	ug/kg	.33	1.65	DOD4	65	130
Methoxychlor	72-43-5	REG	ug/kg	.33	1.65	DOD4	55	145
Toxaphene	8001-35-2	REG	ug/kg	16.7	33	DOD4	25	138
2,4,5,6-Tetrachloro-m-xylene	877-09-8	SURR	% Recovery			DOD4	70	125
Decachlorobiphenyl	2051-24-3	SURR	% Recovery			DOD4	55	130

Microbac Laboratories Inc.
List Definitions Report

AUGUST 27, 2010 13:25

List Type: 8081
List Function: ALL
List Matrix Class: WATER
List Join ID: LJ65200
Methodref: 8081
PKey: 2415.034 Description:

Compound	CAS #	Parm Type	Units	MDL	RL	QCKEY	L_LCS	U_LCS
4,4'-DDD	72-54-8	REG	ug/L	.01	.05	DOD4	25	150
4,4'-DDE	72-55-9	REG	ug/L	.01	.05	DOD4	35	140
4,4'-DDT	50-29-3	REG	ug/L	.01	.05	DOD4	45	140
Aldrin	309-00-2	REG	ug/L	.01	.05	DOD4	25	140
alpha Chlordane	5103-71-9	REG	ug/L	.01	.05	DOD4	65	125
alpha-BHC	319-84-6	REG	ug/L	.01	.05	DOD4	60	130
beta-BHC	319-85-7	REG	ug/L	.01	.05	DOD4	65	125
delta-BHC	319-86-8	REG	ug/L	.01	.05	DOD4	45	135
Dieldrin	60-57-1	REG	ug/L	.01	.05	DOD4	60	130
Endosulfan I	959-98-8	REG	ug/L	.01	.05	DOD4	50	110
Endosulfan II	33213-65-9	REG	ug/L	.01	.05	DOD4	30	130
Endosulfan sulfate	1031-07-8	REG	ug/L	.01	.05	DOD4	55	135
Endrin	72-20-8	REG	ug/L	.01	.05	DOD4	55	135
Endrin aldehyde	7421-93-4	REG	ug/L	.01	.05	DOD4	55	135
Endrin ketone	53494-70-5	REG	ug/L	.01	.05	DOD4	75	125
gamma Chlordane	5103-74-2	REG	ug/L	.01	.05	DOD4	60	125
gamma-BHC (Lindane)	58-89-9	REG	ug/L	.01	.05	DOD4	25	135
Heptachlor	76-44-8	REG	ug/L	.01	.05	DOD4	40	130
Heptachlor epoxide	1024-57-3	REG	ug/L	.01	.05	DOD4	60	130
Methoxychlor	72-43-5	REG	ug/L	.01	.05	DOD4	55	150
Toxaphene	8001-35-2	REG	ug/L	.3	1	DOD4	41	126
2,4,5,6-Tetrachloro-m-xylene	877-09-8	SURR	% Recovery			DOD4	25	140
Decachlorobiphenyl	2051-24-3	SURR	% Recovery			DOD4	30	135

8082-Solid

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Aroclor-1254	11097-69-1	REG	ug/kg	8.25	16.5	60	130	40
Aroclor-1260	11096-82-5	REG	ug/kg	8.25	16.5	60	130	40
Aroclor-1016	12674-11-2	REG	ug/kg	8.25	16.5	40	140	40
Aroclor-1242	53469-21-9	REG	ug/kg	8.25	16.5	64	136	40
Aroclor-1248	12672-29-6	REG	ug/kg	8.25	16.5	64	136	40
Aroclor-1221	11104-28-2	REG	ug/kg	8.25	16.5	64	136	40
Aroclor-1232	11141-16-5	REG	ug/kg	8.25	16.5	64	136	40
2,4,5,6-Tetrachloro-m-Xylene	877-09-8	SURR	% Recovery			29	133	
Decachlorobiphenyl	2051-24-3	SURR	% Recovery			30	173	

8260-Solid

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
1,1,1,2-Tetrachloroethane	630-20-6	REG	ug/kg	0.5	5	71	137	30
1,1,1-Trichloroethane	71-55-6	REG	ug/kg	0.5	5	70	135	30
1,1,2,2-Tetrachloroethane	79-34-5	REG	ug/kg	0.5	5	55	130	30
1,1,2-Trichloroethane	79-00-5	REG	ug/kg	0.5	5	60	125	30
1,1-Dichloroethane	75-34-3	REG	ug/kg	1	5	75	125	30
1,1-Dichloroethene	75-35-4	REG	ug/kg	0.5	5	65	135	30
1,1-Dichloropropene	563-58-6	REG	ug/kg	0.5	5	57	138	30
1,2,3-Trichlorobenzene	87-61-6	REG	ug/kg	0.5	5	60	135	30
1,2,3-Trichloropropane	96-18-4	REG	ug/kg	1	5	65	130	30
1,2,4-Trichlorobenzene	120-82-1	REG	ug/kg	0.5	5	65	130	30
1,2,4-Trimethylbenzene	95-63-6	REG	ug/kg	0.5	5	75	132	30
1,2-Dibromo-3-chloropropane	96-12-8	REG	ug/kg	2	5	40	135	30
1,2-Dibromoethane	106-93-4	REG	ug/kg	0.5	5	69	130	30
1,2-Dichlorobenzene	95-50-1	REG	ug/kg	0.5	5	70	130	30
1,2-Dichloroethane	107-06-2	REG	ug/kg	0.5	5	63	133	30
1,2-Dichloropropane	78-87-5	REG	ug/kg	0.5	5	72	130	30
1,3,5-Trimethylbenzene	108-67-8	REG	ug/kg	0.5	5	74	133	30
1,3-Dichlorobenzene	541-73-1	REG	ug/kg	0.5	5	70	130	30
1,3-Dichloropropane	142-28-9	REG	ug/kg	0.5	5	65	128	30
1,4-Dichlorobenzene	106-46-7	REG	ug/kg	0.5	5	70	130	30
2,2-Dichloropropane	594-20-7	REG	ug/kg	0.5	5	66	135	30
2-Butanone	78-93-3	REG	ug/kg	2.5	10	37	180	30
2-Chloroethyl vinyl ether	110-75-8	REG	ug/kg	2	10	35	154	30
2-Chlorotoluene	95-49-8	REG	ug/kg	0.5	5	63	147	30
2-Hexanone	591-78-6	REG	ug/kg	2.5	10	45	145	30
4-Chlorotoluene	106-43-4	REG	ug/kg	0.5	5	70	138	30
4-Methyl-2-pentanone	108-10-1	REG	ug/kg	2.5	10	47	146	30
Acetone	67-64-1	REG	ug/kg	5	10	20	160	30
Benzene	71-43-2	REG	ug/kg	0.5	5	70	130	30
Bromobenzene	108-86-1	REG	ug/kg	0.5	5	72	131	30
Bromochloromethane	74-97-5	REG	ug/kg	0.5	5	70	130	30
Bromodichloromethane	75-27-4	REG	ug/kg	0.5	5	72	137	30
Bromoform	75-25-2	REG	ug/kg	0.5	5	49	136	30
Bromomethane	74-83-9	REG	ug/kg	1	10	37	143	30
Carbon disulfide	75-15-0	REG	ug/kg	0.5	5	39	139	30
Carbon tetrachloride	56-23-5	REG	ug/kg	0.5	5	59	136	30
Chlorobenzene	108-90-7	REG	ug/kg	0.5	5	70	130	30
Chloroethane	75-00-3	REG	ug/kg	1	10	52	135	30
Chloroform	67-66-3	REG	ug/kg	0.5	5	74	129	30
Chloromethane	74-87-3	REG	ug/kg	2	10	30	131	30
cis-1,2-Dichloroethene	156-59-2	REG	ug/kg	0.5	5	70	130	30
cis-1,3-Dichloropropene	10061-01-5	REG	ug/kg	0.5	5	70	142	30
Chlorodibromomethane	124-48-1	REG	ug/kg	0.5	5	59	136	30
Dibromomethane	74-95-3	REG	ug/kg	0.5	5	69	130	30
Dichlorodifluoromethane	75-71-8	REG	ug/kg	1	10	25	130	30
Ethylbenzene	100-41-4	REG	ug/kg	0.5	5	70	130	30
Hexachlorobutadiene	87-68-3	REG	ug/kg	0.5	5	65	135	30
Isopropylbenzene	98-82-8	REG	ug/kg	0.5	5	68	129	30

m-,p-Xylene	179601-23-1	REG	ug/kg	0.5	5	70	130	30
Methylene chloride	75-09-2	REG	ug/kg	1	5	74	128	30
n-Butylbenzene	104-51-8	REG	ug/kg	0.5	5	70	136	30
n-Propylbenzene	103-65-1	REG	ug/kg	0.5	5	72	136	30
Naphthalene	91-20-3	REG	ug/kg	0.5	10	50	146	30
o-Xylene	95-47-6	REG	ug/kg	0.5	5	70	130	30
p-Isopropyltoluene	99-87-6	REG	ug/kg	0.5	5	72	128	30
sec-Butylbenzene	135-98-8	REG	ug/kg	0.5	5	71	132	30
Styrene	100-42-5	REG	ug/kg	0.5	5	74	130	30
tert-Butylbenzene	98-06-6	REG	ug/kg	0.5	5	72	130	30
Tetrachloroethene	127-18-4	REG	ug/kg	0.5	5	72	130	30
Toluene	108-88-3	REG	ug/kg	0.5	5	77	126	30
trans-1,2-Dichloroethene	156-60-5	REG	ug/kg	0.5	5	72	127	30
trans-1,3-Dichloropropene	10061-02-6	REG	ug/kg	0.5	5	65	139	30
Trichloroethene	79-01-6	REG	ug/kg	0.5	5	72	126	30
Trichlorofluoromethane	75-69-4	REG	ug/kg	1	10	48	154	30
Vinyl acetate	108-05-4	REG	ug/kg	1	10	10	150	30
Vinyl chloride	75-01-4	REG	ug/kg	1	10	45	140	30
Chlorobenzene-d5	3114-55-4	STD	ug/kg			0	0	0
Fluorobenzene	462-06-6	STD	ug/kg			0	0	0
1,4-Dichlorobenzene-d4	3855-82-1	STD	ug/kg			0	0	0
4-Bromofluorobenzene	460-00-4	SURR	% Recovery			74	121	
Toluene-d8	2037-26-5	SURR	% Recovery			81	117	
1,2-Dichloroethane-d4	17060-07-0	SURR	% Recovery			80	120	
Dibromofluoromethane	1868-53-7	SURR	% Recovery			80	120	

8260 Water

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
1,1,1,2-Tetrachloroethane	630-20-6	REG	ug/L	0.25	1	80	130	20
1,1,1-Trichloroethane	71-55-6	REG	ug/L	0.25	1	80	134	20
1,1,2,2-Tetrachloroethane	79-34-5	REG	ug/L	0.2	1	79	125	20
1,1,2-Trichloroethane	79-00-5	REG	ug/L	0.25	1	80	125	20
1,1-Dichloroethane	75-34-3	REG	ug/L	0.125	1	80	125	20
1,1-Dichloroethene	75-35-4	REG	ug/L	0.5	1	80	132	20
1,1-Dichloropropene	563-58-6	REG	ug/L	0.25	1	75	130	20
1,2,3-Trichlorobenzene	87-61-6	REG	ug/L	0.15	1	55	140	20
1,2,3-Trichloropropane	96-18-4	REG	ug/L	0.5	1	75	125	20
1,2,4-Trichlorobenzene	120-82-1	REG	ug/L	0.2	1	65	135	20
1,2,4-Trimethylbenzene	95-63-6	REG	ug/L	0.25	1	80	125	20
1,2-Dibromo-3-chloropropane	96-12-8	REG	ug/L	1	5	50	130	20
1,2-Dibromoethane	106-93-4	REG	ug/L	0.25	1	80	129	20
1,2-Dichlorobenzene	95-50-1	REG	ug/L	0.125	1	80	125	20
1,2-Dichloroethane	107-06-2	REG	ug/L	0.25	1	80	129	20
1,2-Dichloropropane	78-87-5	REG	ug/L	0.2	1	80	120	20
1,3,5-Trimethylbenzene	108-67-8	REG	ug/L	0.25	1	80	127	20
1,3-Dichlorobenzene	541-73-1	REG	ug/L	0.25	1	80	120	20
1,3-Dichloropropane	142-28-9	REG	ug/L	0.2	1	80	120	20
1,4-Dichlorobenzene	106-46-7	REG	ug/L	0.125	1	80	120	20
2,2-Dichloropropane	594-20-7	REG	ug/L	0.25	1	80	133	20
2-Butanone	78-93-3	REG	ug/L	2.5	10	10	170	20
2-Chloroethyl vinyl ether	110-75-8	REG	ug/L	2	10	45	160	20
2-Chlorotoluene	95-49-8	REG	ug/L	0.125	1	80	127	20
2-Hexanone	591-78-6	REG	ug/L	2.5	10	55	130	20
4-Chlorotoluene	106-43-4	REG	ug/L	0.25	1	80	126	20
4-Methyl-2-pentanone	108-10-1	REG	ug/L	2.5	10	64	140	20
Acetone	67-64-1	REG	ug/L	2.5	10	40	180	20
Benzene	71-43-2	REG	ug/L	0.125	1	80	121	20
Bromobenzene	108-86-1	REG	ug/L	0.125	1	80	120	20
Bromochloromethane	74-97-5	REG	ug/L	0.2	1	65	130	20
Bromodichloromethane	75-27-4	REG	ug/L	0.25	1	80	131	20
Bromoform	75-25-2	REG	ug/L	0.5	1	70	130	20
Bromomethane	74-83-9	REG	ug/L	0.5	1	30	145	20
Carbon disulfide	75-15-0	REG	ug/L	0.5	1	58	128	20
Carbon tetrachloride	56-23-5	REG	ug/L	0.25	1	65	140	20
Chlorobenzene	108-90-7	REG	ug/L	0.125	1	80	120	20
Chloroethane	75-00-3	REG	ug/L	0.5	1	60	135	20
Chloroform	67-66-3	REG	ug/L	0.125	1	80	125	20
Chloromethane	74-87-3	REG	ug/L	0.5	1	40	125	20
cis-1,2-Dichloroethene	156-59-2	REG	ug/L	0.25	1	70	125	20
cis-1,3-Dichloropropene	10061-01-5	REG	ug/L	0.25	1	70	130	20
Chlorodibromomethane	124-48-1	REG	ug/L	0.25	1	60	135	20
Dibromomethane	74-95-3	REG	ug/L	0.25	1	75	125	20
Dichlorodifluoromethane	75-71-8	REG	ug/L	0.25	1	40	160	20
Ethylbenzene	100-41-4	REG	ug/L	0.25	1	80	122	20
Hexachlorobutadiene	87-68-3	REG	ug/L	0.25	1	72	132	20
Isopropylbenzene	98-82-8	REG	ug/L	0.25	1	80	122	20

8260 Water

m-,p-Xylene	179601-23-1	REG	ug/L	0.5	1	80	122	20
Methylene chloride	75-09-2	REG	ug/L	0.25	5	80	123	20
n-Butylbenzene	104-51-8	REG	ug/L	0.25	1	80	131	20
n-Propylbenzene	103-65-1	REG	ug/L	0.125	1	80	129	20
Naphthalene	91-20-3	REG	ug/L	0.2	1	59	149	20
o-Xylene	95-47-6	REG	ug/L	0.25	1	80	122	20
p-Isopropyltoluene	99-87-6	REG	ug/L	0.25	1	80	122	20
sec-Butylbenzene	135-98-8	REG	ug/L	0.25	1	80	127	20
Styrene	100-42-5	REG	ug/L	0.125	1	80	123	20
tert-Butylbenzene	98-06-6	REG	ug/L	0.25	1	80	126	20
Tetrachloroethene	127-18-4	REG	ug/L	0.25	1	80	124	20
Toluene	108-88-3	REG	ug/L	0.25	1	80	124	20
trans-1,2-Dichloroethene	156-60-5	REG	ug/L	0.25	1	80	127	20
trans-1,3-Dichloropropene	10061-02-6	REG	ug/L	0.5	1	80	130	20
Trichloroethene	79-01-6	REG	ug/L	0.25	1	80	122	20
Trichlorofluoromethane	75-69-4	REG	ug/L	0.25	1	62	151	20
Vinyl acetate	108-05-4	REG	ug/L	2.5	10	10	190	20
Vinyl chloride	75-01-4	REG	ug/L	0.25	1	50	170	20
Chlorobenzene-d5	3114-55-4	STD				0	0	0
Fluorobenzene	462-06-6	STD				0	0	0
1,4-Dichlorobenzene-d4	3855-82-1	STD				0	0	0
4-Bromofluorobenzene	460-00-4	SURR	% Recovery			86	115	
Toluene-d8	2037-26-5	SURR	% Recovery			88	110	
1,2-Dichloroethane-d4	17060-07-0	SURR	% Recovery			80	120	
Dibromofluoromethane	1868-53-7	SURR	% Recovery			86	118	

8270-Solid

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
1,2,4-Trichlorobenzene	120-82-1	REG	ug/kg	82.5	165	35	100	40
1,2-Dichlorobenzene	95-50-1	REG	ug/kg	82.5	165	35	95	40
1,3-Dichlorobenzene	541-73-1	REG	ug/kg	82.5	165	35	100	40
1,4-Dichlorobenzene	106-46-7	REG	ug/kg	82.5	165	35	105	40
2,4,5-Trichlorophenol	95-95-4	REG	ug/kg	82.5	165	40	110	40
2,4,6-Trichlorophenol	88-06-2	REG	ug/kg	82.5	165	40	110	40
2,4-Dichlorophenol	120-83-2	REG	ug/kg	82.5	165	35	110	40
2,4-Dimethylphenol	105-67-9	REG	ug/kg	82.5	165	30	105	40
2,4-Dinitrophenol	51-28-5	REG	ug/kg	330	825	40	130	40
2,4-Dinitrotoluene	121-14-2	REG	ug/kg	82.5	165	50	130	40
2,6-Dinitrotoluene	606-20-2	REG	ug/kg	82.5	165	50	125	40
2-Chloronaphthalene	91-58-7	REG	ug/kg	82.5	165	40	105	40
2-Chlorophenol	95-57-8	REG	ug/kg	82.5	165	35	105	40
2-Methylnaphthalene	91-57-6	REG	ug/kg	82.5	165	35	115	40
Phenol	108-95-2	REG	ug/kg	82.5	165	35	100	40
Pyrene	129-00-0	REG	ug/kg	82.5	165	35	140	40
2-Methylphenol	95-48-7	REG	ug/kg	82.5	165	35	100	40
2-Nitroaniline	88-74-4	REG	ug/kg	330	825	45	120	40
2-Nitrophenol	88-75-5	REG	ug/kg	82.5	165	35	100	40
3,3'-Dichlorobenzidine	91-94-1	REG	ug/kg	165	330	40	140	40
3-,4-Methylphenol	106-44-5	REG	ug/kg	82.5	165	35	105	40
3-Nitroaniline	99-09-2	REG	ug/kg	330	825	50	130	40
4,6-Dinitro-2-methylphenol	534-52-1	REG	ug/kg	330	825	45	130	40
4-Bromophenyl-phenylether	101-55-3	REG	ug/kg	82.5	165	40	115	40
4-Chloro-3-methylphenol	59-50-7	REG	ug/kg	82.5	165	40	100	40
4-Chloroaniline	106-47-8	REG	ug/kg	82.5	165	35	100	40
4-Chlorophenyl-phenyl ether	7005-72-3	REG	ug/kg	82.5	165	40	110	40
4-Nitroaniline	100-01-6	REG	ug/kg	330	825	35	140	40
4-Nitrophenol	100-02-7	REG	ug/kg	330	825	45	140	40
Acenaphthene	83-32-9	REG	ug/kg	82.5	165	40	110	40
Acenaphthylene	208-96-8	REG	ug/kg	82.5	165	40	110	40
Anthracene	120-12-7	REG	ug/kg	82.5	165	55	130	40
Benzo(a)anthracene	56-55-3	REG	ug/kg	82.5	165	50	130	40
Benzo(a)pyrene	50-32-8	REG	ug/kg	82.5	165	50	130	40
Benzo(b)fluoranthene	205-99-2	REG	ug/kg	82.5	165	45	125	40
Benzo(g,h,i)Perylene	191-24-2	REG	ug/kg	82.5	165	40	140	40
Benzo(k)fluoranthene	207-08-9	REG	ug/kg	82.5	165	45	135	40
Benzoic acid	65-85-0	REG	ug/kg	330	5000	20	110	40
Benzyl alcohol	100-51-6	REG	ug/kg	82.5	165	30	100	40
Bis(2-Chloroethoxy)Methane	111-91-1	REG	ug/kg	82.5	165	30	100	40
Bis(2-Chloroethyl)ether	111-44-4	REG	ug/kg	82.5	165	30	100	40
bis(2-Chloroisopropyl)ether	108-60-1	REG	ug/kg	82.5	165	20	115	40
bis(2-Ethylhexyl)phthalate	117-81-7	REG	ug/kg	82.5	165	50	150	40
Butylbenzylphthalate	85-68-7	REG	ug/kg	82.5	165	50	150	40
Chrysene	218-01-9	REG	ug/kg	82.5	165	55	140	40
Di-N-Butylphthalate	84-74-2	REG	ug/kg	82.5	165	55	140	40
Di-n-octylphthalate	117-84-0	REG	ug/kg	82.5	165	40	145	40
Dibenzo(a,h)Anthracene	53-70-3	REG	ug/kg	82.5	165	40	140	40

8270-Solid

Dibenzofuran	132-64-9	REG	ug/kg	82.5	165	35	110	40
Diethylphthalate	84-66-2	REG	ug/kg	82.5	165	50	130	40
Dimethylphthalate	131-11-3	REG	ug/kg	82.5	165	45	115	40
Fluoranthene	206-44-0	REG	ug/kg	82.5	165	55	140	40
Fluorene	86-73-7	REG	ug/kg	82.5	165	45	115	40
Hexachlorobenzene	118-74-1	REG	ug/kg	82.5	165	45	120	40
Hexachlorobutadiene	87-68-3	REG	ug/kg	82.5	165	30	100	40
Hexachlorocyclopentadiene	77-47-4	REG	ug/kg	82.5	165	30	110	40
Hexachloroethane	67-72-1	REG	ug/kg	82.5	165	30	100	40
Indeno(1,2,3-cd)pyrene	193-39-5	REG	ug/kg	82.5	165	50	135	40
Isophorone	78-59-1	REG	ug/kg	82.5	165	35	100	40
N-Nitrosodiphenylamine	86-30-6	REG	ug/kg	82.5	165	50	130	40
N-Nitrosodipropylamine	621-64-7	REG	ug/kg	82.5	165	35	110	40
Naphthalene	91-20-3	REG	ug/kg	82.5	165	35	100	40
Nitrobenzene	98-95-3	REG	ug/kg	82.5	165	35	100	40
Pentachlorophenol	87-86-5	REG	ug/kg	330	825	50	150	40
Phenanthrene	85-01-8	REG	ug/kg	82.5	165	50	130	40
1,4-Dichlorobenzene-d4	3855-82-1	STD				0	0	0
Acenaphthene-d10	15067-26-2	STD				0	0	0
Perylene-d12	1520-96-3	STD				0	0	0
Naphthalene-D8	1146-65-2	STD				0	0	0
Chrysene-d12	1719-03-5	STD				0	0	0
Phenanthrene-d10	1517-22-2	STD				0	0	0
Nitrobenzene-d5	4165-60-0	SURR	% Recovery			23	120	
p-Terphenyl-d14	1718-51-0	SURR	% Recovery			18	137	
Phenol-d5	4165-62-2	SURR	% Recovery			24	113	
2-Fluorophenol	367-12-4	SURR	% Recovery			25	121	
2-Fluorobiphenyl	321-60-8	SURR	% Recovery			30	115	
2,4,6-Tribromophenol	118-79-6	SURR	% Recovery			19	122	

8270-Water

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
1,2,4-Trichlorobenzene	120-82-1	REG	ug/L	2.5	5	25	105	30
1,2-Dichlorobenzene	95-50-1	REG	ug/L	2.5	5	25	110	30
1,3-Dichlorobenzene	541-73-1	REG	ug/L	2.5	5	25	110	30
1,4-Dichlorobenzene	106-46-7	REG	ug/L	2.5	5	25	110	30
2,4,5-Trichlorophenol	95-95-4	REG	ug/L	2.5	5	35	120	30
2,4,6-Trichlorophenol	88-06-2	REG	ug/L	2.5	5	30	120	30
2,4-Dichlorophenol	120-83-2	REG	ug/L	2.5	5	20	110	30
2,4-Dimethylphenol	105-67-9	REG	ug/L	2.5	5	20	120	30
2,4-Dinitrophenol	51-28-5	REG	ug/L	12.5	25	20	140	30
2,4-Dinitrotoluene	121-14-2	REG	ug/L	2.5	5	50	139	30
2,6-Dinitrotoluene	606-20-2	REG	ug/L	2.5	5	50	120	30
2-Chloronaphthalene	91-58-7	REG	ug/L	2.5	5	25	120	30
2-Chlorophenol	95-57-8	REG	ug/L	2.5	5	25	110	30
2-Methylnaphthalene	91-57-6	REG	ug/L	2.5	5	25	120	30
2-Methylphenol	95-48-7	REG	ug/L	2.5	5	20	110	30
2-Nitroaniline	88-74-4	REG	ug/L	12.5	25	45	115	30
2-Nitrophenol	88-75-5	REG	ug/L	2.5	5	20	115	30
3,3'-Dichlorobenzidine	91-94-1	REG	ug/L	2.5	10	30	140	30
3-,4-Methylphenol	106-44-5	REG	ug/L	2.5	5	20	110	30
3-Nitroaniline	99-09-2	REG	ug/L	12.5	25	40	120	30
4,6-Dinitro-2-methylphenol	534-52-1	REG	ug/L	12.5	25	40	145	30
4-Bromophenyl-phenylether	101-55-3	REG	ug/L	2.5	5	40	115	30
4-Chloro-3-methylphenol	59-50-7	REG	ug/L	2.5	5	25	110	30
4-Chloroaniline	106-47-8	REG	ug/L	2.5	5	25	120	30
4-Chlorophenyl-phenyl ether	7005-72-3	REG	ug/L	2.5	5	35	120	30
4-Nitroaniline	100-01-6	REG	ug/L	12.5	25	53	135	30
4-Nitrophenol	100-02-7	REG	ug/L	12.5	25	10	132	30
Acenaphthene	83-32-9	REG	ug/L	2.5	5	30	120	30
Acenaphthylene	208-96-8	REG	ug/L	2.5	5	30	120	30
Anthracene	120-12-7	REG	ug/L	2.5	5	55	130	30
Benzo(a)anthracene	56-55-3	REG	ug/L	2.5	5	60	130	30
Benzo(a)pyrene	50-32-8	REG	ug/L	2.5	5	55	135	30
Benzo(b)fluoranthene	205-99-2	REG	ug/L	2.5	5	45	125	30
Benzo(g,h,i)Perylene	191-24-2	REG	ug/L	2.5	5	45	140	30
Benzo(k)fluoranthene	207-08-9	REG	ug/L	2.5	5	55	140	30
Benzoic acid	65-85-0	REG	ug/L	12.5	25	10	100	30
Benzyl alcohol	100-51-6	REG	ug/L	2.5	5	20	110	30
Bis(2-Chloroethoxy)Methane	111-91-1	REG	ug/L	2.5	5	20	105	30
Bis(2-Chloroethyl)ether	111-44-4	REG	ug/L	2.5	5	25	110	30
bis(2-Chloroisopropyl)ether	108-60-1	REG	ug/L	2.5	5	20	110	30
bis(2-Ethylhexyl)phthalate	117-81-7	REG	ug/L	3	10	50	150	30
Butylbenzylphthalate	85-68-7	REG	ug/L	2.5	5	55	150	30
Chrysene	218-01-9	REG	ug/L	2.5	5	55	130	30
Di-N-Butylphthalate	84-74-2	REG	ug/L	2.5	5	55	118	30
Di-n-octylphthalate	117-84-0	REG	ug/L	2.5	5	40	146	30
Dibenzo(a,h)Anthracene	53-70-3	REG	ug/L	2.5	5	45	125	30
Dibenzofuran	132-64-9	REG	ug/L	2.5	5	35	115	30
Diethylphthalate	84-66-2	REG	ug/L	2.5	5	45	120	30

8270-Water

Dimethylphthalate	131-11-3	REG	ug/L	2.5	5	25	112	30
Fluoranthene	206-44-0	REG	ug/L	2.5	5	50	137	30
Fluorene	86-73-7	REG	ug/L	2.5	5	40	120	30
Hexachlorobenzene	118-74-1	REG	ug/L	2.5	5	50	130	30
Hexachlorobutadiene	87-68-3	REG	ug/L	2.5	5	24	105	30
Hexachlorocyclopentadiene	77-47-4	REG	ug/L	2.5	5	20	143	30
Hexachloroethane	67-72-1	REG	ug/L	2.5	5	25	95	30
Indeno(1,2,3-cd)pyrene	193-39-5	REG	ug/L	2.5	5	50	135	30
Isophorone	78-59-1	REG	ug/L	2.5	5	30	110	30
N-Nitrosodiphenylamine	86-30-6	REG	ug/L	2.5	5	40	110	30
N-Nitrosodipropylamine	621-64-7	REG	ug/L	2.5	5	28	120	30
Naphthalene	91-20-3	REG	ug/L	2.5	5	25	110	30
Nitrobenzene	98-95-3	REG	ug/L	2.5	5	30	110	30
Pentachlorophenol	87-86-5	REG	ug/L	12.5	25	40	140	30
Phenanthrene	85-01-8	REG	ug/L	2.5	5	55	120	30
Phenol	108-95-2	REG	ug/L	2.5	5	10	120	30
Pyrene	129-00-0	REG	ug/L	2.5	5	55	130	30
1,4-Dichlorobenzene-d4	3855-82-1	STD						
Chrysene-d12	1719-03-5	STD						
Phenanthrene-d10	1517-22-2	STD						
Perylene-d12	1520-96-3	STD						
Naphthalene-D8	1146-65-2	STD						
Acenaphthene-d10	15067-26-2	STD						
p-Terphenyl-d14	1718-51-0	SURR	% Recovery			33	141	
Phenol-d5	4165-62-2	SURR	% Recovery			10	94	
Nitrobenzene-d5	4165-60-0	SURR	% Recovery			35	114	
2-Fluorophenol	367-12-4	SURR	% Recovery			21	100	
2-Fluorobiphenyl	321-60-8	SURR	% Recovery			43	116	
2,4,6-Tribromophenol	118-79-6	SURR	% Recovery			10	123	

827 PAHL-Solid

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Phenanthrene	85-01-8	REG	ug/kg	1.25	2.5	35	92	40
Indeno(1,2,3-cd)pyrene	193-39-5	REG	ug/kg	1.25	2.5	42	132	40
Naphthalene	91-20-3	REG	ug/kg	1.25	2.5	37	87	40
Acenaphthene	83-32-9	REG	ug/kg	1.25	2.5	30	87	40
1-Methylnaphthalene	90-12-0	REG	ug/kg	1.25	2.5	35	89	40
Acenaphthylene	208-96-8	REG	ug/kg	1.25	2.5	31	87	40
Anthracene	120-12-7	REG	ug/kg	1.25	2.5	37	94	40
Benzo(a)anthracene	56-55-3	REG	ug/kg	1.25	2.5	43	136	40
Benzo(a)pyrene	50-32-8	REG	ug/kg	1.25	2.5	45	136	40
Benzo(b)fluoranthene	205-99-2	REG	ug/kg	1.25	2.5	39	130	40
Benzo(g,h,i)perylene	191-24-2	REG	ug/kg	1.25	2.5	42	132	40
Benzo(k)fluoranthene	207-08-9	REG	ug/kg	1.25	2.5	40	133	40
Chrysene	218-01-9	REG	ug/kg	1.25	2.5	43	131	40
Pyrene	129-00-0	REG	ug/kg	1.25	2.5	47	118	40
Dibenzo(a,h)anthracene	53-70-3	REG	ug/kg	1.25	2.5	40	135	40
Fluoranthene	206-44-0	REG	ug/kg	1.25	2.5	41	123	40
Fluorene	86-73-7	REG	ug/kg	1.25	2.5	31	86	40
2-Methylnaphthalene	91-57-6	REG	ug/kg	1.25	2.5	35	92	40
Naphthalene-d8	1146-65-2	STD				0	0	0
Chrysene-d12	1719-03-5	STD				0	0	0
Acenaphthene-d10	15067-26-2	STD				0	0	0
Phenanthrene-d10	1517-22-2	STD				0	0	0
Perylene-d12	1520-96-3	STD				0	0	0
Nitrobenzene-d5	4165-60-0	SURR	% Recovery			23	120	
2-Fluorobiphenyl	321-60-8	SURR	% Recovery			30	115	
p-Terphenyl-d14	1718-51-0	SURR	% Recovery			18	137	

827-PAHL-Water

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Phenanthrene	85-01-8	REG	ug/L	0.025	0.05	33	122	40
Indeno(1,2,3-cd)pyrene	193-39-5	REG	ug/L	0.025	0.05	42	147	40
Naphthalene	91-20-3	REG	ug/L	0.025	0.05	33	112	40
Acenaphthene	83-32-9	REG	ug/L	0.025	0.05	22	116	40
1-Methylnaphthalene	90-12-0	REG	ug/L	0.025	0.05	31	111	40
Acenaphthylene	208-96-8	REG	ug/L	0.025	0.05	21	116	40
Anthracene	120-12-7	REG	ug/L	0.025	0.05	34	121	40
Benzo(a)anthracene	56-55-3	REG	ug/L	0.025	0.05	55	150	40
Benzo(a)pyrene	50-32-8	REG	ug/L	0.025	0.05	55	150	40
Benzo(b)fluoranthene	205-99-2	REG	ug/L	0.025	0.05	51	148	40
Benzo(g,h,i)perylene	191-24-2	REG	ug/L	0.025	0.05	29	149	40
Benzo(k)fluoranthene	207-08-9	REG	ug/L	0.025	0.05	42	157	40
Chrysene	218-01-9	REG	ug/L	0.025	0.05	50	155	40
Pyrene	129-00-0	REG	ug/L	0.025	0.05	55	138	40
Dibenzo(a,h)anthracene	53-70-3	REG	ug/L	0.025	0.05	23	150	40
Fluoranthene	206-44-0	REG	ug/L	0.025	0.05	51	141	40
Fluorene	86-73-7	REG	ug/L	0.025	0.05	23	118	40
2-Methylnaphthalene	91-57-6	REG	ug/L	0.025	0.05	32	115	40
Naphthalene-d8	1146-65-2	STD				0	0	0
Chrysene-d12	1719-03-5	STD				0	0	0
Acenaphthene-d10	15067-26-2	STD				0	0	0
Phenanthrene-d10	1517-22-2	STD				0	0	0
Perylene-d12	1520-96-3	STD				0	0	0
Nitrobenzene-d5	4165-60-0	SURR	% Recovery			35	114	
2-Fluorobiphenyl	321-60-8	SURR	% Recovery			43	116	
p-Terphenyl-d14	1718-51-0	SURR	% Recovery			33	141	

CAS/HOUSTON DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	EDL	MRL	DOD LOD	DOD LOQ	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)	Accuracy (Sample %Rec.)
1613B	2378-TCDD	1746-01-6	Solid	0.0664	1	NA	NA	ng/Kg	67-158	67-158	50	NA	NA	NA
1613B	12378-PeCDD	40321-76-4	Solid	0.0656	5	NA	NA	ng/Kg	70-142	70-142	50	NA	NA	NA
1613B	123478-HxCDD	57653-85-7	Solid	0.0500	5	NA	NA	ng/Kg	70-164	70-164	50	NA	NA	NA
1613B	123678-HxCDD	39227-28-6	Solid	0.0616	5	NA	NA	ng/Kg	76-134	76-134	50	NA	NA	NA
1613B	123789-HxCDD	19408-74-3	Solid	0.0525	5	NA	NA	ng/Kg	64-162	64-162	50	NA	NA	NA
1613B	1234678-HpCDD	35822-46-9	Solid	0.0539	5	NA	NA	ng/Kg	70-140	70-140	50	NA	NA	NA
1613B	OCDD	3268-87-9	Solid	0.0990	10	NA	NA	ng/Kg	78-144	78-144	50	NA	NA	NA
1613B	2378-TCDF	51207-31-9	Solid	0.0726	1	NA	NA	ng/Kg	75-158	75-158	50	NA	NA	NA
1613B	12378-PeCDF	57117-41-6	Solid	0.0501	5	NA	NA	ng/Kg	80-134	80-134	50	NA	NA	NA
1613B	23478-PeCDF	57117-31-4	Solid	0.0444	5	NA	NA	ng/Kg	68-160	68-160	50	NA	NA	NA
1613B	123478-HxCDF	57117-44-9	Solid	0.0489	5	NA	NA	ng/Kg	72-134	72-134	50	NA	NA	NA
1613B	123678-HxCDF	72918-21-9	Solid	0.0521	5	NA	NA	ng/Kg	84-130	84-130	50	NA	NA	NA
1613B	123789-HxCDF	70648-26-9	Solid	0.0688	5	NA	NA	ng/Kg	78-130	78-130	50	NA	NA	NA
1613B	234678-HxCDF	60851-34-5	Solid	0.0490	5	NA	NA	ng/Kg	70-156	70-156	50	NA	NA	NA
1613B	1234678-HpCDF	67562-39-4	Solid	0.0482	5	NA	NA	ng/Kg	82-132	82-132	50	NA	NA	NA
1613B	1234789-HpCDF	55673-89-7	Solid	0.0561	5	NA	NA	ng/Kg	78-138	78-138	50	NA	NA	NA
1613B	OCDF	39001-02-0	Solid	0.0782	10	NA	NA	ng/Kg	63-170	63-170	50	NA	NA	NA
1613B	Total TCDD	41903-57-5	Solid	NA	1	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total PeCDD	36088-22-9	Solid	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total HxCDD	34465-46-8	Solid	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total HpCDD	37871-00-4	Solid	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total TCDF	55722-27-5	Solid	NA	1	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total PeCDF	30402-15-4	Solid	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total HxCDF	55684-94-1	Solid	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total HpCDF	38998-75-3	Solid	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	13C-2378-TCDD		Solid	NA	NA	NA	NA	Percent	20-175	20-175	NA	NA	NA	25-164
1613B	13C-12378-PeCDD		Solid	NA	NA	NA	NA	Percent	21-227	21-227	NA	NA	NA	25-181
1613B	13C-123478-HxCDD		Solid	NA	NA	NA	NA	Percent	21-193	21-193	NA	NA	NA	32-141
1613B	13C-123678-HxCDD		Solid	NA	NA	NA	NA	Percent	25-163	25-163	NA	NA	NA	26-123
1613B	13C-1234678-HpCDD		Solid	NA	NA	NA	NA	Percent	26-166	26-166	NA	NA	NA	23-140
1613B	13C-OCDD		Solid	NA	NA	NA	NA	Percent	13-199	13-199	NA	NA	NA	17-157
1613B	13C-2378-TCDF		Solid	NA	NA	NA	NA	Percent	22-152	22-152	NA	NA	NA	24-169
1613B	13C-12378-PeCDF		Solid	NA	NA	NA	NA	Percent	21-192	21-192	NA	NA	NA	24-185
1613B	13C-23478-PeCDF		Solid	NA	NA	NA	NA	Percent	13-328	13-328	NA	NA	NA	21-178
1613B	13C-123478-HxCDF		Solid	NA	NA	NA	NA	Percent	19-202	19-202	NA	NA	NA	26-152
1613B	13C-123678-HxCDF		Solid	NA	NA	NA	NA	Percent	21-159	21-159	NA	NA	NA	26-123
1613B	13C-123789-HxCDF		Solid	NA	NA	NA	NA	Percent	17-205	17-205	NA	NA	NA	29-147
1613B	13C-234678-HxCDF		Solid	NA	NA	NA	NA	Percent	22-176	22-176	NA	NA	NA	28-136
1613B	13C-1234678-HpCDF		Solid	NA	NA	NA	NA	Percent	21-158	21-158	NA	NA	NA	28-143
1613B	13C-1234789-HpCDF		Solid	NA	NA	NA	NA	Percent	20-186	20-186	NA	NA	NA	26-138
1613B	37Cl-2378-TCDD		Solid	NA	NA	NA	NA	Percent	31-191	31-191	NA	NA	NA	35-197
1613B	2378-TCDD	1746-01-6	Tissue	0.0664	1	NA	NA	ng/Kg	67-158	67-158	50	NA	NA	NA
1613B	12378-PeCDD	40321-76-4	Tissue	0.0656	5	NA	NA	ng/Kg	70-142	70-142	50	NA	NA	NA
1613B	123478-HxCDD	57653-85-7	Tissue	0.0500	5	NA	NA	ng/Kg	70-164	70-164	50	NA	NA	NA
1613B	123678-HxCDD	39227-28-6	Tissue	0.0616	5	NA	NA	ng/Kg	76-134	76-134	50	NA	NA	NA
1613B	123789-HxCDD	19408-74-3	Tissue	0.0525	5	NA	NA	ng/Kg	64-162	64-162	50	NA	NA	NA
1613B	1234678-HpCDD	35822-46-9	Tissue	0.0539	5	NA	NA	ng/Kg	70-140	70-140	50	NA	NA	NA
1613B	OCDD	3268-87-9	Tissue	0.0990	10	NA	NA	ng/Kg	78-144	78-144	50	NA	NA	NA
1613B	2378-TCDF	51207-31-9	Tissue	0.0726	1	NA	NA	ng/Kg	75-158	75-158	50	NA	NA	NA
1613B	12378-PeCDF	57117-41-6	Tissue	0.0501	5	NA	NA	ng/Kg	80-134	80-134	50	NA	NA	NA
1613B	23478-PeCDF	57117-31-4	Tissue	0.0444	5	NA	NA	ng/Kg	68-160	68-160	50	NA	NA	NA
1613B	123478-HxCDF	57117-44-9	Tissue	0.0489	5	NA	NA	ng/Kg	72-134	72-134	50	NA	NA	NA
1613B	123678-HxCDF	72918-21-9	Tissue	0.0521	5	NA	NA	ng/Kg	84-130	84-130	50	NA	NA	NA

CAS/HOUSTON DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	EDL	MRL	DOD LOD	DOD LOQ	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)	Accuracy (Sample %Rec.)
1613B	123789-HxCDF	70648-26-9	Tissue	0.0688	5	NA	NA	ng/Kg	78-130	78-130	50	NA	NA	NA
1613B	234678-HxCDF	60851-34-5	Tissue	0.0490	5	NA	NA	ng/Kg	70-156	70-156	50	NA	NA	NA
1613B	1234678-HpCDF	67562-39-4	Tissue	0.0482	5	NA	NA	ng/Kg	82-132	82-132	50	NA	NA	NA
1613B	1234789-HpCDF	55673-89-7	Tissue	0.0561	5	NA	NA	ng/Kg	78-138	78-138	50	NA	NA	NA
1613B	OCDF	39001-02-0	Tissue	0.0782	10	NA	NA	ng/Kg	63-170	63-170	50	NA	NA	NA
1613B	Total TCDD	41903-57-5	Tissue	NA	1	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total PeCDD	36088-22-9	Tissue	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total HxCDD	34465-46-8	Tissue	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total HpCDD	37871-00-4	Tissue	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total TCDF	55722-27-5	Tissue	NA	1	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total PeCDF	30402-15-4	Tissue	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total HxCDF	55684-94-1	Tissue	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	Total HpCDF	38998-75-3	Tissue	NA	5	NA	NA	ng/Kg	NA	NA	NA	NA	NA	NA
1613B	13C-2378-TCDD		Tissue	NA	NA	NA	NA	Percent	20-175	20-175	NA	NA	NA	25-164
1613B	13C-12378-PeCDD		Tissue	NA	NA	NA	NA	Percent	21-227	21-227	NA	NA	NA	25-181
1613B	13C-123478-HxCDD		Tissue	NA	NA	NA	NA	Percent	21-193	21-193	NA	NA	NA	32-141
1613B	13C-123678-HxCDD		Tissue	NA	NA	NA	NA	Percent	25-163	25-163	NA	NA	NA	26-123
1613B	13C-1234678-HpCDD		Tissue	NA	NA	NA	NA	Percent	26-166	26-166	NA	NA	NA	23-140
1613B	13C-OCDD		Tissue	NA	NA	NA	NA	Percent	13-199	13-199	NA	NA	NA	17-157
1613B	13C-2378-TCDF		Tissue	NA	NA	NA	NA	Percent	22-152	22-152	NA	NA	NA	24-169
1613B	13C-12378-PeCDF		Tissue	NA	NA	NA	NA	Percent	21-192	21-192	NA	NA	NA	24-185
1613B	13C-23478-PeCDF		Tissue	NA	NA	NA	NA	Percent	13-328	13-328	NA	NA	NA	21-178
1613B	13C-123478-HxCDF		Tissue	NA	NA	NA	NA	Percent	19-202	19-202	NA	NA	NA	26-152
1613B	13C-123678-HxCDF		Tissue	NA	NA	NA	NA	Percent	21-159	21-159	NA	NA	NA	26-123
1613B	13C-123789-HxCDF		Tissue	NA	NA	NA	NA	Percent	17-205	17-205	NA	NA	NA	29-147
1613B	13C-234678-HxCDF		Tissue	NA	NA	NA	NA	Percent	22-176	22-176	NA	NA	NA	28-136
1613B	13C-1234678-HpCDF		Tissue	NA	NA	NA	NA	Percent	21-158	21-158	NA	NA	NA	28-143
1613B	13C-1234789-HpCDF		Tissue	NA	NA	NA	NA	Percent	20-186	20-186	NA	NA	NA	26-138
1613B	37Cl-2378-TCDD		Tissue	NA	NA	NA	NA	Percent	31-191	31-191	NA	NA	NA	35-197
1613B	2378-TCDD	1746-01-6	Aqueous	0.370	10	NA	NA	pg/L	67-158	67-158	50	NA	NA	NA
1613B	12378-PeCDD	40321-76-4	Aqueous	0.475	50	NA	NA	pg/L	70-142	70-142	50	NA	NA	NA
1613B	123478-HxCDD	57653-85-7	Aqueous	0.348	50	NA	NA	pg/L	70-164	70-164	50	NA	NA	NA
1613B	123678-HxCDD	39227-28-6	Aqueous	0.448	50	NA	NA	pg/L	76-134	76-134	50	NA	NA	NA
1613B	123789-HxCDD	19408-74-3	Aqueous	0.377	50	NA	NA	pg/L	64-162	64-162	50	NA	NA	NA
1613B	1234678-HpCDD	35822-46-9	Aqueous	0.716	50	NA	NA	pg/L	70-140	70-140	50	NA	NA	NA
1613B	OCDD	3268-87-9	Aqueous	0.653	100	NA	NA	pg/L	78-144	78-144	50	NA	NA	NA
1613B	2378-TCDF	51207-31-9	Aqueous	0.555	10	NA	NA	pg/L	75-158	75-158	50	NA	NA	NA
1613B	12378-PeCDF	57117-41-6	Aqueous	0.361	50	NA	NA	pg/L	80-134	80-134	50	NA	NA	NA
1613B	23478-PeCDF	57117-31-4	Aqueous	0.331	50	NA	NA	pg/L	68-160	68-160	50	NA	NA	NA
1613B	123478-HxCDF	57117-44-9	Aqueous	0.313	50	NA	NA	pg/L	72-134	72-134	50	NA	NA	NA
1613B	123678-HxCDF	72918-21-9	Aqueous	0.336	50	NA	NA	pg/L	84-130	84-130	50	NA	NA	NA
1613B	123789-HxCDF	70648-26-9	Aqueous	0.387	50	NA	NA	pg/L	78-130	78-130	50	NA	NA	NA
1613B	234678-HxCDF	60851-34-5	Aqueous	0.325	50	NA	NA	pg/L	70-156	70-156	50	NA	NA	NA
1613B	1234678-HpCDF	67562-39-4	Aqueous	0.402	50	NA	NA	pg/L	82-132	82-132	50	NA	NA	NA
1613B	1234789-HpCDF	55673-89-7	Aqueous	0.443	50	NA	NA	pg/L	78-138	78-138	50	NA	NA	NA
1613B	OCDF	39001-02-0	Aqueous	0.596	100	NA	NA	pg/L	63-170	63-170	50	NA	NA	NA
1613B	Total TCDD	41903-57-5	Aqueous	NA	10	NA	NA	pg/L	NA	NA	NA	NA	NA	NA
1613B	Total PeCDD	36088-22-9	Aqueous	NA	50	NA	NA	pg/L	NA	NA	NA	NA	NA	NA
1613B	Total HxCDD	34465-46-8	Aqueous	NA	50	NA	NA	pg/L	NA	NA	NA	NA	NA	NA
1613B	Total HpCDD	37871-00-4	Aqueous	NA	50	NA	NA	pg/L	NA	NA	NA	NA	NA	NA
1613B	Total TCDF	55722-27-5	Aqueous	NA	10	NA	NA	pg/L	NA	NA	NA	NA	NA	NA
1613B	Total PeCDF	30402-15-4	Aqueous	NA	50	NA	NA	pg/L	NA	NA	NA	NA	NA	NA
1613B	Total HxCDF	55684-94-1	Aqueous	NA	50	NA	NA	pg/L	NA	NA	NA	NA	NA	NA

CAS/HOUSTON DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	EDL	MRL	DOD LOD	DOD LOQ	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)	DOD QSM (LCS %Rec.)	DOD QSM (% RPD)	Accuracy (Sample %Rec.)
1613B	Total HpCDF	38998-75-3	Aqueous	NA	50	NA	NA	pg/L	NA	NA	NA	NA	NA	NA
1613B	13C-2378-TCDD		Aqueous	NA	NA	NA	NA	Percent	20-175	20-175	NA	NA	NA	25-164
1613B	13C-12378-PeCDD		Aqueous	NA	NA	NA	NA	Percent	21-227	21-227	NA	NA	NA	25-181
1613B	13C-123478-HxCDD		Aqueous	NA	NA	NA	NA	Percent	21-193	21-193	NA	NA	NA	32-141
1613B	13C-123678-HxCDD		Aqueous	NA	NA	NA	NA	Percent	25-163	25-163	NA	NA	NA	26-123
1613B	13C-1234678-HpCDD		Aqueous	NA	NA	NA	NA	Percent	26-166	26-166	NA	NA	NA	23-140
1613B	13C-OCDD		Aqueous	NA	NA	NA	NA	Percent	13-199	13-199	NA	NA	NA	17-157
1613B	13C-2378-TCDF		Aqueous	NA	NA	NA	NA	Percent	22-152	22-152	NA	NA	NA	24-169
1613B	13C-12378-PeCDF		Aqueous	NA	NA	NA	NA	Percent	21-192	21-192	NA	NA	NA	24-185
1613B	13C-23478-PeCDF		Aqueous	NA	NA	NA	NA	Percent	13-328	13-328	NA	NA	NA	21-178
1613B	13C-123478-HxCDF		Aqueous	NA	NA	NA	NA	Percent	19-202	19-202	NA	NA	NA	26-152
1613B	13C-123678-HxCDF		Aqueous	NA	NA	NA	NA	Percent	21-159	21-159	NA	NA	NA	26-123
1613B	13C-123789-HxCDF		Aqueous	NA	NA	NA	NA	Percent	17-205	17-205	NA	NA	NA	29-147
1613B	13C-234678-HxCDF		Aqueous	NA	NA	NA	NA	Percent	22-176	22-176	NA	NA	NA	28-136
1613B	13C-1234678-HpCDF		Aqueous	NA	NA	NA	NA	Percent	21-158	21-158	NA	NA	NA	28-143
1613B	13C-1234789-HpCDF		Aqueous	NA	NA	NA	NA	Percent	20-186	20-186	NA	NA	NA	26-138
1613B	37Cl-2378-TCDD		Aqueous	NA	NA	NA	NA	Percent	31-191	31-191	NA	NA	NA	35-197

CAS/HOUSTON DATA QUALITY OBJECTIVES														
						DOD	DOD		Accuracy	Matrix Spike	Precision	DOD QSM	DOD QSM	Accuracy
METHOD	ANALYTE	CAS No.	MATRIX	EDL	MRL	LOD	LOQ	UNITS	(LCS %Rec.)	(%Rec.)	(% RPD)	(LCS %Rec.)	(% RPD)	(Sample %Rec.)
1613B	2378-TCDD	1746-01-6	Solid	0.0664	1	NA	NA	ng/Kg	73-146	73-146	50	NA	NA	NA
1613B	2378-TCDF	51207-31-9	Solid	0.0726	1	NA	NA	ng/Kg	80-147	80-147	50	NA	NA	NA
1613B	13C-2378-TCDD	76523-40-5	Solid	NA	NA	NA	NA	Percent	25-141	25-141	NA	NA	NA	31-137
1613B	13C-2378-TCDF	89059-46-1	Solid	NA	NA	NA	NA	Percent	26-126	26-126	NA	NA	NA	29-140
1613B	37Cl-2378-TCDD	85508-50-5	Solid	NA	NA	NA	NA	Percent	37-158	37-158	NA	NA	NA	42-164
1613B	2378-TCDD	1746-01-6	Tissue	0.0664	1	NA	NA	ng/Kg	73-146	73-146	50	NA	NA	NA
1613B	2378-TCDF	51207-31-9	Tissue	0.0726	1	NA	NA	ng/Kg	80-147	80-147	50	NA	NA	NA
1613B	13C-2378-TCDD	76523-40-5	Tissue	NA	NA	NA	NA	Percent	25-141	25-141	NA	NA	NA	31-137
1613B	13C-2378-TCDF	89059-46-1	Tissue	NA	NA	NA	NA	Percent	26-126	26-126	NA	NA	NA	29-140
1613B	37Cl-2378-TCDD	85508-50-5	Tissue	NA	NA	NA	NA	Percent	37-158	37-158	NA	NA	NA	42-164
1613B	2378-TCDD	1746-01-6	Aqueous	0.370	10	NA	NA	ng/Kg	73-146	73-146	50	NA	NA	NA
1613B	2378-TCDF	51207-31-9	Aqueous	0.555	10	NA	NA	ng/Kg	80-147	80-147	50	NA	NA	NA
1613B	13C-2378-TCDD	76523-40-5	Aqueous	NA	NA	NA	NA	Percent	25-141	25-141	NA	NA	NA	31-137
1613B	13C-2378-TCDF	89059-46-1	Aqueous	NA	NA	NA	NA	Percent	26-126	26-126	NA	NA	NA	29-140
1613B	37Cl-2378-TCDD	85508-50-5	Aqueous	NA	NA	NA	NA	Percent	37-158	37-158	NA	NA	NA	42-164

HG-Solid

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Mercury, Total	7439-97-6	REG	mg/kg	0.01	0.25	80	120	25

HG-Water

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Mercury	7439-97-6	REG	mg/L	0.0001	0.0002	85	115	20

TOC-Solid

Compound	CAS Number	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Total Organic Carbon		REG	mg/kg	500	1000	70	140	50

Compound	CAS Numb	Parm Type	Units	MDL	RDL	LCL	UCL	RPD
Total Organic Carbon		REG	mg/L	0.5	1	85	115	15

ATTACHMENT C

SWMU-57 Laboratory Standard Operating Procedures (on CD)

6010 6020 Soils Digest SOP
6010 6020 Water Digest SOP
6010 SOP
6020 SOP
6850 SOP
8081 SOP
8082 Extraction SOP
8082 SOP
8260 SOP
8270 Extraction SOP
8270 PAHL SOP
8270 SOP
827-PAHL Extraction SOP
HG Soil SOP
HG Water SOP
SOP-45 Validation
TOC SOP
8081-8082 Extraction SOP
8081-8082 Soil Extraction SOP
TCLP Prep SOP

Microbac

Document Control #: - 10292

STANDARD OPERATING PROCEDURES FOR ORGANIC CARBON, TOTAL (OXIDATION) SOP K4151 EPA 415.1/SW846 9060A/SM5310C

Revision 12

Issue/Implementation Date: 15 August 2009

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Microbac Laboratories Inc.
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Approved by:

Deanna I. Hesson, Conventionals Supervisor — Date: 8/3/09

David L. Bumgarner, Technical Director/QAO — Date: 8/14/09

David E. Vandenberg, Managing Director — Date: 8/14/09

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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
	8/3/09

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TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the determination of total organic carbon (TOC) in drinking, surface and saline waters, domestic and industrial wastes. It also applicable for leachable organic carbon on a 1:10 soil to acid mixture. **It is not applicable for total organic carbon on a soil**. TOC is defined as all of the organic carbon present in the sample that can be determined by this method. All analyses are performed within the Shimadzu TOC-VWP analyzer.

1.2 TOC is determined by the difference of total carbon (TC) and inorganic carbon (TIC). For the TC analysis, gas flows at a controlled rate through the TC reactor, which is comprised of a UV lamp and heater. When sample is injected along with the oxidizing reagent (containing sodium persulfate and phosphoric acid) into the TC reactor which has been heated to 80°C, the TC in the sample is oxidized and decomposed to form carbon dioxide. This carbon dioxide is swept via the carrier gas from the reaction tube to a dehumidifier for cooling and dehydration. These products then pass through a halogen scrubber to reach the cell of a non-dispersive infrared detector (NDIR), where the carbon dioxide is detected. The analog detection signal of the NDIR forms a peak, and the area of this peak is measured by a data processor. The peak area is proportional to the TC concentration of the sample. Inorganic carbon (IC) refers to carbon contained in the carbon dioxide dissolved in water and that found in carbonates. By acidifying the sample with a small amount of phosphoric acid to obtain a pH less than 3, all the carbonates produce carbon dioxide (CO_2). The carbon dioxide and dissolved carbon dioxide in the sample are volatilized by bubbling (sparging) gas through the sample. Sparge gas consisting of tiny bubbles flows at a controlled rate through the IC reactor. When sample is injected along with phosphoric acid into the IC reactor, only the IC component of the sample is converted to carbon dioxide, which is subsequently detected by the NDIR. The IC concentration in the sample is then measured in the same way as in the TC measurement process.

1.3 This method references EPA Method 415.1, and SW846 Method 9060A, and Standard Method 5310C.

1.4 To comply with SW846 Method 9060A, each sample must be injected in quadruplicate.

1.4.1 The LIMS product TOC-14 is used when one sample is injected four times and one average result is reported (1 sample * 4 injections).

1.4.2 The LIMS product TOC-44 is used when the sample was sampled in the field in quadruplicate and each one is injected four times. This gives 16 results. The average of each bottle (four injections) is reported (4 samples * 4 injections).

1.4.3 The LIMS product TOC-1 is used to show that the sample gets one injection. The sample is reported as Method 9060 modified since 4 injections are not analyzed.

1.5 Method EPA 415.1 requires only one injection. SM5310C requires duplicate injections (average reported).

1.5.1 If samples are logged as a matrix 2, SM5310C must be followed. All other liquid matrices may follow Method 415.1.

1.5.2 The LIMS product TOC is used for both methods. One result is reported.

1.5.3 The LIMS product TOC-4 is used when one sample is sampled in the field in quadruplicate. Four results are reported.

2.0 SAFETY PRECAUTIONS

2.1 Standard laboratory safety procedures should be followed when working with unknown samples. Lab coats and safety glasses with sideshields are required.

2.2 When doing maintenance on the TOC instrument, the electricity should be turned off to prevent electrocution.

2.3 The following chemicals have the potential to be toxic or hazardous. Consult the MSDS for further information.

2.3.1 Sodium persulfate: This is a strong oxidizing agent and should not be stored near strong reducing agents.

2.3.2 Phosphoric acid: This is a strong and corrosive reagent.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Samples should be collected in a glass container and preserved with sulfuric acid (pH < 2). **NOTE**: HCL should not be used as preservative, because chloride is an interference and the result could be biased.

3.2 Samples should be kept refrigerated at 4° C until time of analysis, which is not to exceed 28 days from the time of collection. After analysis, samples are stored in a designated area in the archive room. See Section 16 for sample disposal.

3.3 Samples should be kept from sunlight and atmospheric oxygen.

3.4 A minimum of 40 ml is required for one analysis. More volume is required for reanalysis and QC requirements.

3.5 If less than the recommended volume is available the analysis will be performed on a dilution, and the reporting limit will be elevated proportionately.

4.0 METHOD PERFORMANCE

4.1 This method uses a reporting limit of 1 mg/L for an undiluted liquid sample. Upon request, a reporting limit of 0.5 mg/L can be reported. For leachable organic carbon, the reporting limit is approximately10 mg/kg. Reporting limits (RL) are nominal laboratory values, but project RLs may vary.

4.2 The laboratory verified MDL is 0.5 mg/L. The statistical MDL is determined annually or when necessary. Verification consists of analyzing a fortified blank (MDL check standard) spiked at a concentration neat the concentration of the statistically determine MDL. This MDL check standard is used to verify that the laboratory MDL is routinely achievable over the course of time. Additional details on MDL studies may be found in SOP 45.

4.3 The linear range of the method depends on the range selected. The overall range is 1 mg/L to 50 mg/L total carbon. The upper limit may be extended by diluting the sample.

4.4 The current statistical limits are 85-115% for the LCS and MS. The duplicate RPD is 15%. However, for method 9060A, the method specified limits of 90-

110% are used. Precision and accuracy data were derived from laboratory control samples results from the previous year and verified annually.

4.5 Each analyst is required to perform an initial demonstration of precision and accuracy. Annually, each analyst is recertified by performing four LCS. See Microbac SOP 45, which describes the procedure in detail.

4.6 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QUAPP.

5.0 INTERFERENCES & CORRECTIVE MEASURES

5.1 The TOC value is determined by the difference in TC and IC analysis, which includes errors associated with both TC and IC analyses.

5.2 Large particles that cannot enter the sipper tube cannot be analyzed. This can result in low recovery.

5.3 Filtration can result in low recovery (removal of carbon-containing particles) or high recovery (carbon from filter paper).

5.4 Very high chloride content results in low recovery. Adding mercuric nitrate to the sample reduces this interference. Dilution also reduces the interference. The instrument can tolerate up to approximately 3000 mg/L CL.

6.0 EQUIPMENT AND SUPPLIES

6.1 TOC Analyzer: Shimadzu TOC-VWP with autosampler, computer, and printer

6.2 Several 40 mL VOA vials

6.3 Mechanical shaker

6.4 All glassware is washed with soap and water as described in MICROBAC SOP K0001.

7.0 STANDARDS AND REAGENTS

NOTE: See Section 16 for reagent disposal.

NOTE: All standards and reagents are prepared using class A volumetric glassware.

NOTE: 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 TOC Stock Standard Solution (1000 mg/L): purchased. Discard by manufacturer's printed expiration date.

7.2 TOC LCS Stock Solution (1000 mg/L): Dissolve 2.1254g KHP deionized water. Mix well. Preserve with 5 mL of 1:1 H_2SO_4. Dilute to 1 liter. Discard after 3 months.

7.3 TIC Calibration Stock Solution (1000 mg/L): Dissolve 4.41 g sodium carbonate and 3.5 g sodium bicarbonate in deionized water and dilute to one liter.

7.4 TIC ICV Stock (1000 mg/L): Make exactly like calibration stock (7.3) except use different lots.

7.5 Sodium Persulfate Solution: Dissolve 120 g ultra pure sodium persulfate and 30 mL phosphoric acid in deionized water and dilute to one liter. This solution should sit for a day to allow oxidation of trace impurities. This reagent stable for at least a month when stored in a cool dark location.

7.6 Phosphoric Acid: Dilute 100 mL of concentrated phosphoric acid with deionized water to a finle volume of 500 mL with deionized water. Discard after 6 months.

7.7 Sulfuric Acid (20%): Add 200 ml concentrated sulfuric acid to 800 ml deionized water. Mix well.

7.8 Ultra pure Nitrogen gas.

8.0 CALIBRATION PROCEDURES

8.1 The instrument is calibrated as needed (when CCV's fail or project defined). Two calibration curves are needed: total carbon (TC) and total inorganic carbon (TIC).

8.1.1 The following standards are analyzed to determine the calibration curves. The TC and TIC curves are prepared identically from respective stocks (7.1 and 7.3).

STANDARD	mL USED	STOCK	TOTAL VOLUME	CONCENTRATION mg/L
1	10	1000	200	50
2	5	1000	200	25
3	2	1000	200	10
4	1	1000	200	5
5	10	10	100	1
6	-	-	-	0

8.2 R must be > 0.995 for each curve.

8.3 TOC ICV(Second Source Check): (25 mg/L) Prepare a 25 mg/L standard using the LCS stock (7.2), and a dilute 5 ml of stock (7.2) to 200 ml deionized water to make a 25 mg/L standard. The result must be within ± 10% of true value, or the instrument will need recalibrated.

8.4 TIC ICV (second source check): (25 mg/L) Prepare a 25 mg/L standard using the LCS stock (7.4), and a dilute 5 ml of stock (7.4) to 200 ml deionized water to make a 25 mg/L standard. The result must be within ± 10% of true value, or the instrument will need recalibrated.

8.5 To analyze a calibration curve use the following procedure:

8.5.1 Choose NEW FILE

8.5.2 Calibration curve

8.5.3 Choose system (TOCVW ASI)

8.5.4 Edit points manually

8.5.5 Choose analysis (TC or IC): a curve will be ran for both.

8.5.6 Choose linear regression

8.5.7 UNCHOOSE zero shift

8.5.8 Type the name of the file (ex: TCCURVE-8-28-2003 or TICCURVE-8-28-2003)

8.5.9 Choose units, TWO injections, ONE wash then next page

8.5.10 Enter each calibration point by pressing ADD and typing concentration then OK. Enter from blank to highest concentration.

8.5.11 Create a run as in Section 11.

8.5.12 Analyze the curves

8.5.13 Analyze an ICV for both curves

8.5.14 Before analyzing samples, a method must be created using the new curves.

8.5.14.1 New file

8.5.14.2 Method

8.5.14.3 Pick system

8.5.14.4 Next

8.5.14.5 Choose analysis: TOC

8.5.14.6 Choose a name for method (ex: TOC-9-30-2003)

8.5.14.7 Next

8.5.14.8 Pick calibration curve for TC analysis

8.5.14.9 Next

8.5.14.10 Enter number of injections: (1 for 415.1, 4 for 9060A, 2 for SM5310C)

8.5.14.11 Enter washes: 1, UV oxid vol: 1.5

8.5.14.12 Next

8.5.14.13 pg. 5 Next

8.5.14.14 Repeat steps 8.5.14.8 - 8.5.14.13 for TIC analysis (wash: 1, acid add :10)

8.5.14.15 pg. 5 Next

8.5.14.16 Next

8.6 A CCV and CCB are analyzed every 10 samples. See section 13 for criteria.

9.0 SAMPLE PREPARATION

9.1 If the sample is a solid and leachable organic carbon is needed, a leachate of the solid will need prepared.

9.2 Add 10 ml of 20% sulfuric acid (7.5) to 10 g of soil. Mix well.

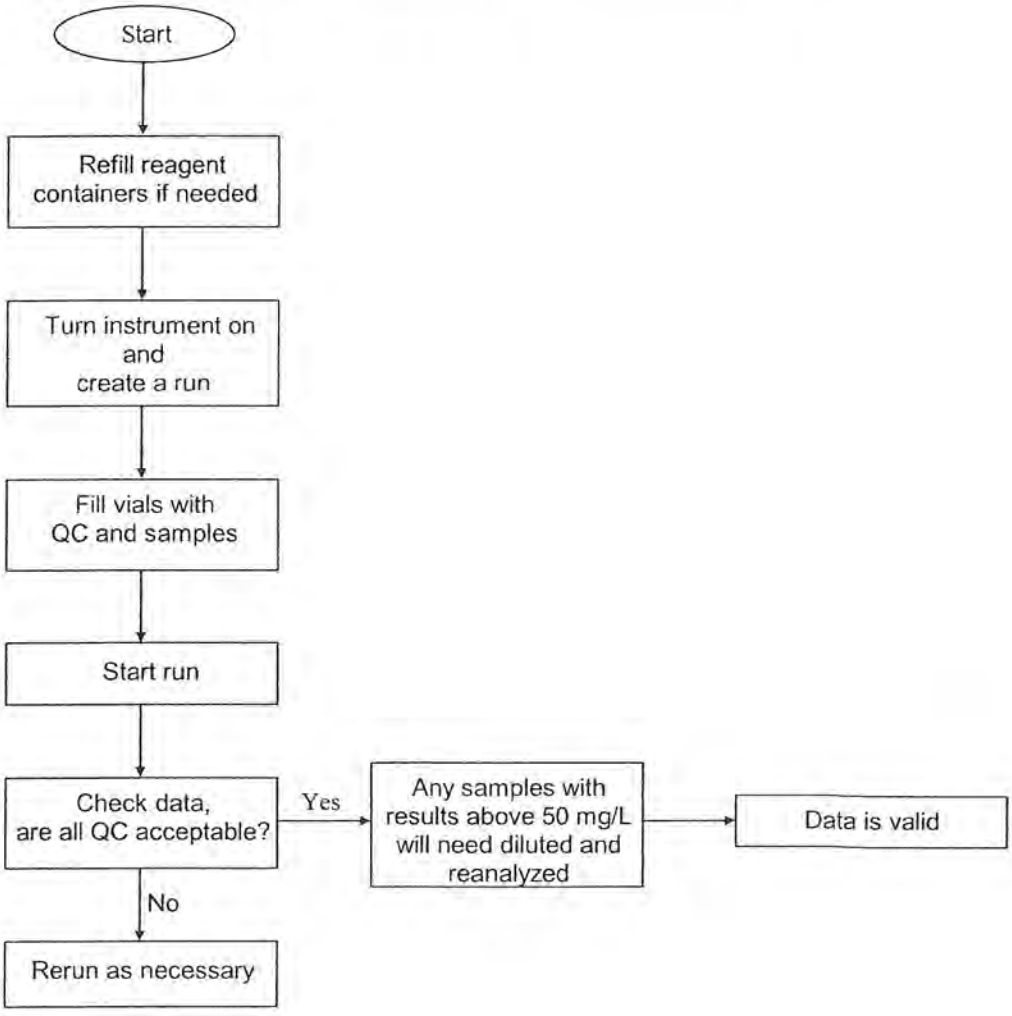
9.3 Add 90 ml of deionized water and shake on a mechanical shaker for 30 minutes.

9.4 Let the sample settle.

9.5 Decant the solution and analyze the solution for TOC. This is considered Leachable Organic Carbon.

9.6 If dissolved organic carbon is needed, filter the sample through a 0.45 um membrane filter (previously soaked in 1:1 nitric acid) and analyze as below. A filtered deionized water blank should also be analyzed.

10.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



11.0 ANALYTICAL PROCEDURES

11.1 Fill reagent containers with the persulfate (7.5) and acid reagents (7.6), and also the three deionized water containers.

11.2 Make sure gas is on. The gas is left on at the tank at all times. The instrument turns the gas on and off within the instrument. Turn the instrument on.

11.3 Create a run as follows:

11.3.1 TOC icon

11.3.2 Sample table editor

11.3.3 OK

11.3.4 New file

11.3.5 Sample run

11.3.6 System: TOCVW ASI

11.3.7 OK

11.3.8 Save with file name (ex: 9-30-2003-dih-TOC)

11.3.9 Save

11.3.10 At position 1 press "autogenerate" icon

11.3.11 Choose method from list (choose from three methods depending on how many injections are needed).

11.3.12 Next

11.3.13 Enter number of samples to be run

11.3.14 Enter start position

11.3.15 Next

11.3.16 pg. 3 Next

11.3.17 pg.4 Next

11.3.18 At sparge/acid window fix vial numbers if needed

11.3.19 OK

11.3.20 Continue with other methods if needed

11.3.21 Enter the sample ID and dilution in the sample ID box.

11.3.22 Press the "connect" icon

11.3.23 The instrument will "warm up and stabilize".

11.4 Fill vials with at least 20mL of samples (or 20mL of a diluted portion) and place in the appropriate position.

11.4.1 Dilutions are made using disposable 1mL, 5mL or 10mL pipets, and graduated cylinders.

11.4.2 If the deionized water to be used is greater than 15mL, the water is measured with a graduated cylinder, otherwise a pipet is used.

11.4.3 If less than 2mL of the sample is needed, a pipet is used, otherwise a graduated cylinder is used.

11.4.4 See examples in Table 2.

11.5 The run should be as follows:

11.5.1 High TOC CCV (50 mg/L)

11.5.2 Low TOC CCV (10 mg/L)

11.5.3 TIC CCV (25 mg/L)

11.5.4 CCB / Blank

11.5.5 TOC LCS

11.5.6 TOC LCS Duplicate

11.5.7 8 samples

11.5.8 CCV

11.5.9 CCB

11.5.10 Continue run with samples and all required QC.

11.5.11 Run a CCV and CCB every ten samples.

11.5.12 Finish the run with a CCV / CCB.

11.6 When the instrument is stable press the "START" icon.

11.7 Fix any vial numbers in the sparge window.

11.8 Uncheck the "ADD ACID" box

11.9 The run will begin

11.10 Any sample with a TC value over 50mg/L will need diluted and reanalyzed.

12.0 DETAILS OF CALCULATIONS

12.1 The instrument calculates and prints out TC, TIC, and TOC results in mg/L. These results do not take into account dilution factors.

12.2 To correct for the dilution, the following formula is used:

*(instrument readout) *(dilution) = mg/L TOC*

where:

readout = Answer obtained from instrument

dilution = Dilution of the sample (ex: *A 1/5 Dilution = 5*)

12.3 If leachable organic carbon is needed, the formula is:

$$\left[instrument\ readout/(dilution)\right]*(volume/weight) = mg/Kg\ \mathrm{LOC}$$

where:

weight: weight in mg of sample volume: volume in mL of water used to shake

volume: volume in mL of water used to leach the sample.

12.4 The following equation is used to calculate the LCS 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

12.5 The following equation is used to calculate the spike recovery:

$$\%R = \left[\frac{\left(C_{spk} - C_x\right)}{C_t}\right]100$$

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 The following equation is used to calculate the duplicate 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 If Method 9060A is required, the average of the four results is reported.

13.0 QUALITY CONTROL (QC) REQUIREMENTS

NOTE: See project QAPP's for more specific QC requirements.
NOTE: See Table I for QC summary
NOTE: Separate Blanks, LCS, Dup, MS are run for each method.

13.1 High CCV (50 mg/L): Dilute 10 ml of the standard stock (7.1) to 200 ml and analyze exactly like the samples. The result should be ± 15% of true value, or the affected samples will need reanalyzed and/or a calibration curve ran.

13.2 Low CCV (10 mg/L): Dilute 2 ml of the standard stock (7.1) standard to 200 ml. Criteria is the same as step 13.1.

13.3 TIC CCV (25 mg/L): Dilute 5 mL of TIC stock (7.3) to 200 mL. Criteria is the sample as step 13.1.

13.4 CCB / Method blank: Analyze deionized water exactly like the samples. Results will be evaluated down to one half of the reporting limit (RL) and corrective action will be project driven if the blank exceeds this level.

13.5 LCS/LCS duplicate (25 mg/L): Dilute 5 mL of TOC LCS stock (7.2) to 200 mL and analyze exactly like the samples in duplicate. The recovery must be within the control limits (see Table 1) or a corrective action will be written to determine if the samples will need to be reanalyzed.

13.6 Matrix Spike: Add 0.4 mL of TOC LCS stock (7.2) to 40 mL of sample and analyze. The recovery must be within the statistically determined limits (see section 4) or a case narrative will be written. No spike is analyzed for Method 9060A unless a client specified matrix spike container is received.

13.7 Duplicate: Analyze any sample in the batch in duplicate at the same dilution. The RPD should be less than the statistically determined RPD (see Table 1) or the run will be examined and reanalyzed as necessary. No duplicate is necessary for Method 9060A.

13.8 A batch (workgroup) is defined as a blank, LCS, LCS Duplicate, Spike, duplicate, and up to 20 samples. Several batches may be run in sequence on any particular day.

13.9 Procedures for handling out-of-control data:

When out-of-control situations are encountered, the laboratory will proceed as outline in Section 13, 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, and if these measures fail, may require contacting the client to inform them of the problem and to obtain their directions re 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 CARs 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.

13.10 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, and 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.

13.10.1 An LCS or surrogate recovery exceeds the upper control limit, but the corresponding samples results are non-detect.

13.10.2 A method blank exceeds the upper limit, but the corresponding samples results are non-detect.

13.10.3 A method blank exceeds the upper limit, but the corresponding samples results are greater than twenty (20) times the level in the blank.

13.11 All data is scrutinized by the analyst for method and project specific compliance. Check lists are utilized and accompany each data batch (Figure1). Out-of-control data will be documented with the preparation of a corrective action report (CAR) followed by one or more of the following alternatives.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data rejection (R-flagging)
- Re-sampling and re-analysis (client decision)

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 QA/QC data is entered onto special forms and reported to the client upon request. Data review uses the attached checklist (Figure 1). All data is calculated and reviewed by the analyst. The supervisor (or designated person) performs a second review before uploading into the LIMS system.

14.2 TOC is reported in mg/L TOC using a maximum of three significant figures. The analyst, date and time are reported.

15.0 PREVENTATIVE MAINTENANCE

15.1 Daily

15.1.1 Verifiy dilution water is sufficient

15.1.2 Verifiy sufficient reagents for analyses

15.1.3 Verify drain vessel is full

15.1.4 Verify waste container is not full

15.1.5 Verify there is sufficient gas

15.1.6 Check for leaks

15.2 Periodically (as required)

15.2.1 Replace CO_2 absorber

15.2.2 Replace Halogen scrubber

15.2.3 Replace Syringe plunger tip

15.2.4 Wash the TC reactor

15.2.5 Wash the IC reactor

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.

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, and Ohio VAP rules allow the suspension of our certification for failure to comply with these laws.

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.

16.3 All reagents and samples and instrument waste are disposed of down the sink (near or in a hood) after neutralization and flushed with a large amount of water.

16.4 Upon completion of the analysis, the samples are stored in a designated area of the archive storage room to await proper disposal by the waste management team.

17.0 REFERENCES

17.1 "Methods for Chemical Analysis of Water and Wastes", EPA/600/4-79/020, Method 415.1 (Combustion or Oxidation), 1983.

17.2 SW846 Method 9060A

17.3 Standard Methods 21st Ed. 5310C.

17.4 Shimadzu TOC-VWP user's manual

Table I
Quality Control Criteria
Total Organic Carbon (TOC)

CONTROL ITEM	FREQUENCY	ACCEPTABLE CRITERIA	CORRECTIVE ACTION
Initial Calibration Curve	When needed or project defined	$R \geq 0.995$	Reanalyze calibration curve
ICV	After calibration	± 10%	See Section 8.3 and 8.4
CCV	Every 10 analyses	± 15%	See Section 13.1, 13.2, and 13.3
CCB	After CCV	≤ ½ RL	See Section 13.4
Method Blank	One per batch (20 samples maximum per batch)	≤ ½ RL	See Section 13.4
Laboratory Control Samples (LCS/LCS DUP)	One per batch (20 samples maximum per batch)	85 – 115% (415.1) 90 – 110% (9060A) or project specified	See Section 13.5
Matrix Spike (MSD if required)	One per batch (20 samples maximum per batch)	Same as LCS criteria	See Section 13.6
Sample Duplicate	One per batch (20 samples maximum per batch)	RPD ≤ 15%	See Section 13.7

Table 2
Examples of Common Dilutions

Dilution	Total Volume Needed	mL Sample	mL Deionized water
1/2	20	10	10
1/2	100	50	50
1/3	30	10	20
1/4	20	5	15
1/4	100	25	75
1/10	20	2	18
1/10	100	10	90
1/50	50	1	49
1/100	100	1	99

*Dilutions over 1/100 will need to be made in a series of dilutions. Ex: 1/5000 would be made as a 1/50 on a 1/100.

Microbac

Figure 1

Checklist ID: 27469

Microbac Laboratories Inc.

Data Checklist

Date: 14-APR-2008
Analyst:
Analyst: NA
Method:
Instrument:
Curve Workgroup: NA
Runlog ID:
Analytical Workgroups:

Calibration/Linearity	
Second Source Check	
ICV/CCV (std)	
ICB/CCB	
Blank	
LCS/LCS Dup	
MS/MSD	
Duplicate	
Upload Results	
Client Forms	
QC Violation Sheet	
Case Narratives	
Signed Raw Data	
STD/LCS on benchsheet	
Check for compliance with method and project specific requirements	
Check the completeness of reported information	
Check the information for the report narrative	
Primary Reviewer	
Secondary Reviewer	DIH
Comments	

Primary Reviewer:
14-APR-2008

Secondary Reviewer:
14-APR-2008

Signature

Definitions

The following definitions may be used in the production of this SOP.

Analytical Batch:

The basic unit for analytical quality control. The analytical batch is defined as samples which are analyzed (or sampled together) with the same method sequence, the same lots of reagents, and with the same treatment common to all samples. The samples must have been analyzed (or collected) within the same specified time period or in continuous sequential time periods. Samples in each batch should be of similar composition.

Calibration:

Process by which the correlation between instrument response and actual value of a measured parameter is determined.

Calibration Curve:

A curve which plots the concentration of known analyte standards against the instrument response to the analyte. Also known as a Standard Curve.

Calibration Standard:

Solutions or dilutions of a substance or material with a verifiable accuracy, which are used to evaluate the sample property of an unknown sample. In analytical terms, these standards are used to establish a calibration curve or standard instrument response factors.

Continuing Calibration Standard:

Standards that are analyzed during an analytical set to verify the accuracy of the calibration curve.

Internal Standard:

A compound having similar chemical characteristics to the compounds of interest, but which is not normally found in the environment or does not interfere with the compounds of interest. A known and specified concentration of the standard is added to each sample prior to analysis.

The concentration in the sample is based on the response of the internal standard relative to that of the calibration standard and the compound in the standard.

Method Detection Limit (MDL):

The smallest concentration of an analyte of interest that can be measured and reported with 99 percent confidence that the concentration is greater than zero. The MDL's are determined from the analysis of a sample in a given matrix containing the analyte at a specified level. Determination of MDL's must be done by procedures determined in Appendix B of 40 CFR, Part 136. Equivalent procedures to determine MDL's must be approved by DER.

Reporting Limit (RL):

The concentration of analyte, below which all quantitation are considered to be estimated. Instrument calibrations have a low standard at this level.

Quality Control:

The overall system of activities whose purpose is to document and control the quality of environmental data so that it meets the needs of the users.

Method Blank:

A blank of an analyte-free matrix that is processed (digested, extracted, etc.) and analyzed with a specified sample set designed to monitor the introduction of contamination into the system.

LCS (Laboratory Control Sample):

Samples of an analyte-free matrix (deionized water, sand, soil, etc.) that are fortified to a known and validated concentration of analyte(s) before sample preparation.

Matrix Spikes:

Environmental sample selected from a set (not blanks) that is fortified to a known and validated concentration of analyte(s) before sample preparation. The concentration of each analyte in the spiking solution should be approximately 3-5 times the level expected in the sample.

Surrogate Spikes:

A compound having similar chemical characteristics to the compounds of interest, but which is not normally found in environmental samples. Known concentrations of these compounds are added to all samples in the set before sample preparation.

Duplicate samples:

Samples that have been collected at the same time from the same source or aliquots of the sample that are prepared and analyzed at the same time. The analytical results from replicates are used to determine the precision of a system.

Microbac

Document Control #: 10281

STANDARD OPERATING PROCEDURE
MERCURY (7470A, 245.1)
SOP ME404

Revision 12

Issue/Implementation Date: 15 July 2009

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Microbac Laboratories, Inc.
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Approved By:

Robert E. Kyer, Digestion Group Leader — Date: 7/13/09

Maren M. Beery, Metal Supervisor — Date: 07/13/2009

David L. Bumgarner, Technical Director/QAO — Date: 7/14/09

David E. Vandenberg, Managing Director — Date: 7/14/09

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
Kim H. Rhoden	7-6-09
Lisa Mann	07-06-09
Sheri L. Hatzigeorgiou	7/7/09
Cathy Artz	7/7/09
Brenda R. Gregory	7/7/09
Vicki Collins	7/7/09

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure provides sample preparation methods and details the operation of a Leeman HYDRA AA Auto analyzer for the analysis of mercury (CAS # 7439-97-6) in drinking, surface and saline waters, and domestic and industrial wastes consistent with protocols for SW 7470A and/or EPA 245.1.

1.2 Mercury analysis is based on a cold-vapor atomic absorption technique. Mercury is reduced to the elemental state and purged with argon to form elemental mercury vapor. The mixture flows through a drying tube for water vapor removal and then enters one path of a double path optical cell. A mercury source, powered by a constant current power supply, delivers a stable source of emission at 254 nm. Absorbance by the mercury cold vapor is measured using a solid state detector with a wide dynamic range. The resulting signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions.

1.3 In addition to inorganic forms of mercury, organic mercury compounds may also be present. The compounds will not respond to the cold vapor technique unless they are broken down into mercuric ions. Potassium permanganate oxidizes most, but not all, of these compounds. Potassium persulfate has been found to oxidize the other organic compounds to give approximately 100% recovery. Therefore, a persulfate oxidation step following the permanganate oxidation has been added to insure complete breakdown of any organo-mercury compounds.

2.0 SAFETY PRECAUTIONS

2.1 **CAUTION** - Acids used in this method for the determination of mercury can burn skin and cause blindness.

2.2 **WARNING** - Voltages as high as 117 or 220 VAC exist inside the instrument so always disconnect the instrument from the wall before opening the chassis.

2.3 Always wear a lab coat, safety glasses, and latex gloves to reduce possible health hazards to the lowest possible level.

2.4 The laboratory maintains MSDS sheets for handling and exposure information.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 All samples must be collected by the use of techniques that prevent contamination and cross-contamination between samples.

3.2 All sample containers must be pre-cleaned. Glass or plastic are both acceptable.

3.3 Aqueous samples must be preserved to pH less than 2 with nitric acid. For determinations of dissolved and suspended metals, the sample must be filtered before preservation on site. See Table 3-1. Upon receipt, the sample pH is determined and recorded in the pH logbook. The final pH is also recorded if adjustment was necessary.

3.4 If a 50 mL volume of sample is not available for digestion, it is permissible to perform the digestion with a reduced volume as outlined in Section 9.4.

Table 3-1

Measurement	Digestion Vol./Wt. Req.	Collection Vol./Wt.	Preservative/ Holding Time *
Total recoverable	50 mL	600 mL	HNO_3 to pH <2 4° C / 28 days
Dissolved	50 mL	600 mL	Filter on site; HNO_3 to pH <2 4° C / 28 days
Suspended	50 mL	600 mL	Filter on site 4° C / 28 days

* Storage time allowed between sample collection and analysis when properly preserved and stored.

4.0 METHOD PERFORMANCE

4.1 Mercury in water is analyzed at a wavelength of 253.7 nm with a reporting limit of 0.0002 mg/L and upper limit of 0.010 mg/L.

4.2 The verified MDL for waters is 0.10 ug/L. Additional details on MDL studies may be found in Microbac SOP 45.

4.3 Refer to Table 4-1 for precision and accuracy targets.

4.4 Refer to Microbac SOP 45 for details of method validation.

MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR INORGANIC METALS ANALYSES OF GROUNDWATER

PARAMETER	CAS #	EPA/SW-846 METHOD*	ACCURACY (% Recovery)*	PRECISION (% RPD)*	VERIFIED MDL WATER (ug/L)	REPORTING LIMITS WATER (ug/L)
Mercury (Hg) - VAPOR	7439-97-6	245.1/7470A	85-115	0-20	0.10	0.20

5.0 INTERFERENCES & CORRECTIVE MEASURES

5.1 Possible interference from sulfides is eliminated by addition of potassium permanganate.

5.2 Copper has been found to interfere; however concentrations as high as 10 mg/L had no effect on spike recovery.

5.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate. Care must be taken that free chlorine is absent before the mercury is reduced. This can be accomplished by using an excess of hydroxylamine hydrochloride reagent.

6.0 EQUIPMENT AND SUPPLIES

6.1 Major Instrumentation

6.1.1 Leeman HYDRA AA Automated Mercury Analyzer

6.2 Apparatus or equipment

6.2.1 Heated water bath capable of maintaining a temperature of 95° C ± 5° C.

6.2.2 Adjustable volume pipettor, 100 - 1000 uL

6.2.3 Adjustable volume pipettor, 1 - 10 mL

6.2.4 Argon gas supply

6.2.5 36 or 54 Well Hot Block Digestor

6.2.6 Polypropylane digestion tubes (50mL)

6.3 Glassware

6.3.1 100 mL graduated cylinder, Nalgene

6.3.2 100 mL volumetric flask, Kimax or Pyrex

6.3.3 See Microbac SOP ME201 for glassware cleaning procedures.

6.3.4 Class A pipets (various sizes)

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 and RGT number.

Calibration Solutions

7.2 Calibration Stock Mercury Solution - a purchased certified standard of 1000 ug/mL Hg-Specpure.

7.3 Intermediate Calibration Mercury Solution – Prepared by diluting 5.0 mL of the calibration stock solution (7.2) to a total volume of 500 mL with 5% HNO_3 deionized water. The concentration is 10.0 ug/mL Hg. The shelf life is 6 months or the same as the stock solution whichever is sooner.

7.4 Working Calibration Mercury Solution: Prepare fresh daily by diluting 2.0 mL of the intermediate calibration mercury solution (7.3) to 500 mL total volume with 5% HNO_3 deionized water. Concentration is 0.04 ug/mL Hg.

7.5 Calibration Standard preparation: Transfer 0.2, 1.0, 2.0, 5.0 and 10.0 mL aliquouts of the 0.04 ug/mL mercury working standard (7.4) to a series of 50 mL polypropylene tubes. Add enough deionized water to each tube to make a total volume of 40 mL. Add 1.0 mL of concentrated Nitric Acid, 2.0 mL of concentrated sulfuric acid, 6 mL's of 5% Potassium permanganate, and 3.2 mL's fo 5% Potassium persulfate. Final calibration standard concentrations are 0.2, 1.0, 2.0, 5.0 and 10.0 ug/L. These standards should be made fresh each day an analysis is prepared. A calibration blank is prepared by adding the same reagents to 40 mL of deionized water.

Quality Control Stock Mercury Solution

7.6 Quality Control Stock Mercury Solution – a purchased standard of 1000 ug/mL. This standard is purchased from Inorganic Ventures, a vendor independent of the calibration stock mercury solution.

7.7 Intermediate Quality Control Mercury Solution: Dilute 5.00 mL of the quality control stock mercury solution (7.6) to 500 mL total volume with 5% HNO_3 DI Water. Concentration is 10.0 ug/mL Hg. The shelf life is 6 months or the same as the stock solution, whichever is sooner.

7.8 0.0400 ug/mL Hg spike solution – Prepare fresh daily by diluting 2.0 mL of the intermediate quality control mercury solution (7.7) to 500 mL with 5% HNO_3 DI Water Concentration is 0.0400 ug/mL Hg.

7.9 ICV – An ICV solution is prepared by diluting 2.0 mL of the 0.04 ug/mL Hg spike solution (7.8) to a final volume of 40 mL. Add the same volumes of acids and reagents that are listed in (7.5) for the initial calibration standards. The concentration of the ICV solution is 2.0 ug/L.

7.10 Sulfuric Acid (H_2SO_4), Concentrated: Baker Instra Analyzed grade or better.

7.11 Nitric Acid (HNO_3), Concentrated: Baker Instra Analyzed grade or better.

7.12 Hydrochloric Acid (HCl), Concentrated: Baker Instra Analyzed grade or better

7.13 Stannous Chloride solution: Dissolve 50 g of Stannous Chloride in 50 mL concentrated HCl and dilute to 500 mL with acidified deionized water.

7.14 Hydroxylamine Hydrochloride solution: Dissolve 120 g of Hydroxylamine hydrochloride and 120 g sodium chloride NaCl in deionized water and dilute to 1000 mL.

7.15 Potassium Permanganate: 5% solution w/v. Dissolve 5 g of potassium permanganate in 100 mL of deionized water.

7.16 Potassium Persulfate: 5% solution w/v. Dissolve 5 g of potassium persulfate in 100 mL of deionized water.

7.17 Deionized Water (ASTM Type I)

8.0 CALIBRATION PROCEDURES

8.1 Initial calibration – The instrument is calibrated before analysis of any samples with a blank and five calibration standards (7.5). A linear curve type must be selected. The first standard run must be the blank followed by standards of increasing concentration in order to minimize cross contamination and carry over. The low calibration standard must contain mercury at a concentration at or below the reporting limit. See Tables 13-1 and 13-2 for acceptance criteria and corrective action for the initial calibration.

8.2 Initial Calibration Verification (ICV) Analysis – ICV analysis (7.9) must be performed immediately after calibration standards to verify calibration. See tables 13-1 and 13-2 for acceptance criteria and corrective actions.

8.3 Continuing calibration verification (CCV) Analysis - The CCV solution is the 2.0 ug/L standard from section 7.5. The CCV is required to be analyzed prior to sample analysis, after every ten analytical samples and at the end of the analysis. See Tables 13-1 and 13-2 for acceptance criteria and corrective actions.

8.4 Initial and continuing calibration blank (ICB/CCB) Anaylsis – The solution used is the calibration blank from section 7.5. See tables 13-1 and 13-2 for acceptance criteria and corrective actions.

8.5 See figure 8-1 for analytical sequence.

FIGURE 8-1
Analytical Sequence

Calibration (See Section 11.4)
ICV
ICB
CCV
CCB
10 or less samples
CCV
CCB
Repeat CCV/CCB every 10 samples
Always end run with CCV/CCB

9.0 SAMPLE PREPARATION

9.1 Upon receipt, the pH of the sample is taken and recorded. If the pH is greater than 2, additional nitric acid must be added and the final pH brought to less than 2 and recorded. The sample is allowed to equilibrate for 24 hours prior to digestion. A note should be made to the client in the final report.

9.2 Sample due dates are recorded on the caps and in the preliminary sample log.

9.3 Samples are organized into similar matrix batches of 20.

9.4 Hot Block Method:

9.4.1 Measure out 40 mL's of a well mixed sample into a 50 mL polypropylene tube that is labeled. For ground water, waste water and SPLP leachates use 40 mL. For TCLP leachates use 4 mL, diluted to 40 mL. For liquid wastes use 1 mL diluted to 40 mL. If a reduced volume of sample is used to perform the digestion then, the volume of reagents used must be adjusted accordingly.

9.4.2 Add 1.0 mL of concentrated Nitric Acid to each sample and quality control sample.

9.4.3 Add 2.0 mL of concentrated Sulfuric Acid to each sample and quality control sample.

9.4.4 Mix thoroughly and add 6 mL of 5% ($KMNO_4$) Potassium permanganate. Let stand for 15 minutes and if sample does not maintain purple or brown color, add an additional 1 mL of $KMNO_4$ solution.

9.4.5 Add 3.2 mL of 5% Potassium persulfate.

9.4.6 Place lid on tube and turn back one turn to allow for venting.

9.4.7 Digest the sample for 2 hours at 95°C ± 5°C.

9.4.8 After digest has cooled, make sure final volume is the same volume as standards and adjust if needed with DI water.

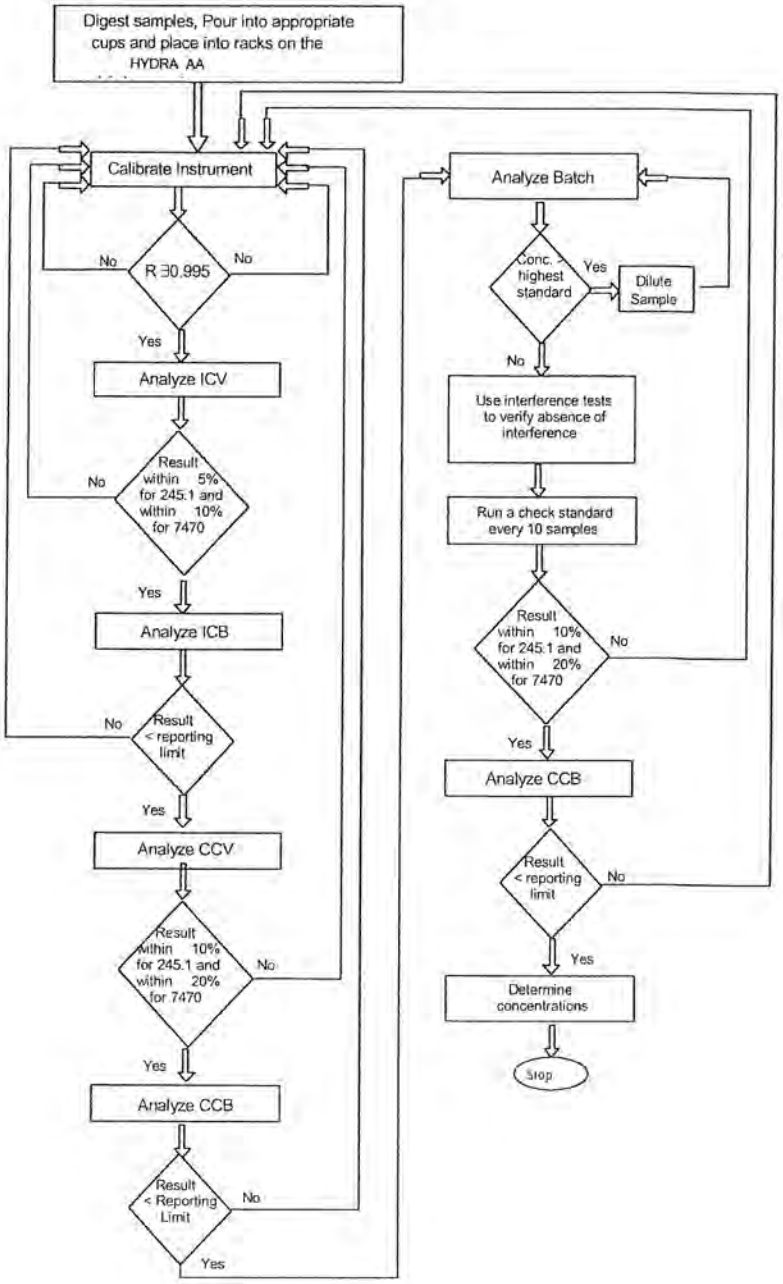
9.4.9 After final volume is determined, add 3.0 mL of 12% hydroxylamine hydrochloride.

9.5 A preparation blank (PB) is digested with each batch of water samples. Use 40 ml of polished deionized water (7.17). Treat as sample in Section 9.4.

9.6 The LCS (LCS DUP) is prepared by diluting 4 mL of the 0.04 ug/mL Hg spike solution (7.8) to a 40 mL final volume using deionized water. Treat as sample in Section 9.4. The final concentration of the LCS is 4.0 ug/L Hg. An LCS Dup may also be prepared with the batch.

9.7 A matrix spike (MS) and matrix spike duplicate (MSD) are prepared with each batch of water samples. Add 4 mL of spike solution (7.8) to the digestion vessel and fill to the 40 mL mark. Treat as sample in Section 9.4. The concentration of the spike is 4.0 ug/L, based on the 40 mL final volume.

10.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



11.0 ANALYTICAL PROCEDURES

11.1 HYDRA AA autoanalyzer Instrument Startup

11.1.1 Turn on the power strip, computer and gas supply.

11.1.2 Log into system and click on WinHG icon. This opens up WinHG Runner 1.4 window.

11.1.3 Click on Control and turn Hg lamp on by clicking the On Button

11.1.4 Check pump tubing for wear and replace if necessary

11.1.5 Check Stannous chloride and rinse solution volumes and refill if needed

11.2 Retrieve the proper protocol, standard. Main menu, protocol and click on down arrow then standard.

11.3 Create a new data set by clicking on file from the main menu, new dataset then enter date, then OK. Batch Id will appear, enter date, then ok.

11.4 Calibrate the instrument using the calibration standard solutions .

11.4.1 Transfer the standards and check standards to the appropriate cups in the calibration rack. Load the standard rack into its proper position on the HYDRA AA auto analyzer.

11.4.2 Click on Standard then on the buttons labeled S1-S6 then click on Stnd Auto button to begin calibration.

11.4.3 To review the calibration results click on the DB (database) button then on Cal Curve. If results are ≥0.995 on accept. This stores the calibration coefficients.

11.4.4 Print the calibration results by clicking on the printer icon located at the top of the database window.

11.5 Analyze ICV, ICB, CCV and CCB solutions. Load ICV in position #9, ICB in position #10, CCV in position #11 and CCB in position #12. To analyze each check standard click on buttons labeled C2-C5 then on Chk Std Auto.

11.6 Load samples into cups in sample rack making appropriate dilutions and spikes where necessary. Filter samples containing particulates prior to filling cups. Load the rack in the proper position in the HYDRA AA Auto analyzer. From the main menu click on the button resembling autosampler rack. Enter sample IDs and C4 space C5 in the last column. This tells the instrument to analyze check standards. Click on file, save as, enter date and close window.

11.7 From the main menu click on Sample, enter Rack Name. Enter Start and End cup numbers.

11.8 To start analysis click on the Run Auto button and monitor run for CCV and CCB acceptance. See Tables 13-1 and 13-2 for acceptance criteria.

11.9 After analyses are complete, place stannous and rinse tubing in DI water and rinse for 4-5 minutes. Pull tubing out and raise sample probe and allow all tubing to pump dry. Shut off lamp, pump, and gas. Exit out of software and shut down computer. Turn off power strip. Release tension on pump tubing.

11.10 Upload results into LIMS. Review batch QA/QC data while creating PDF format.

12.0 DETAILS OF CALCULATION

After the calibration is complete, the software performs a linear regression with a calculated intercept which is not forced through zero. The regression equation is given by $I = BX + C$

where:

I = mean intensity of standard

B = slope of ICAL

X = concentration of standard

C = intercept of ICAL

The coefficient of correlation (COC) is calculated to verify the linearity of the calibration curve. The COC is required to be ≥ 0.995 in order for the curve to be valid.

$$COC = \frac{\Sigma xy - \frac{\Sigma x \Sigma y}{n}}{\sqrt{\left(\Sigma x^2 - \frac{(\Sigma x)^2}{n}\right)\left(\Sigma y^2 - \frac{(\Sigma y)^2}{n}\right)}}$$

where:

X = standard concentration
Y = mean intensity
N = 6 = number of standards

12.1 Results are calculated from the calibration curve by the system software utilizing linear regression. Dilution factors and preparation information are applied by the LIMS system during data upload.

Water samples:

$$\text{Concentration (ug/L)} = \frac{I - C}{BD}$$

where:

B = slope from instrument ICAL
C = intercept from instrument ICAL
I = mean intensity for sample
D =decimal dilution factor (eg - 10X = 0.1)

All concentrations are based on 40mL volume for samples and standards.

12.2 LCS (LCS DUP) % recovery is calculated as follows:

$$\left(\frac{C_x}{C_T}\right)100 = \text{LCS \% recovery}$$

where:

C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.

12.3 Relative Percent Difference (RPD) results are calculated as follows:

$$\left[\frac{|C_1 - C_2|}{(C_1 + C_2)}\right]100 = \text{RPD}$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.4 See figure 12.1 for a calculation summary.

12.5 Matrix Spike recovery is calculated as follows:

$$\left[\frac{(C_{spk} - C_x)}{C_t}\right]100 = \%\ \text{recovery}$$

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.

12.6 Post digestion spike recovery is calculated as follows:

$$\frac{C_{spk} - [C_x * 0.9]}{C_t} * 100 = \%\,\text{Recovery}$$

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.

12.7 Serial dilution percent difference is calculated as follows:

$$\frac{C_x - [C_{DL} * 5]}{C_x} * 100 = \%\,Difference$$

C_x = the concentration of the analyte in the reference (parent) sample
C_{DL} = the concentration of the analyte in the five-fold dilution.

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). 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 corrective action measures are outlined in Tables 13-1 and 13-2.

13.2 With each batch of twenty (20) samples or less the following quality control samples will be analyzed and must meet the criteria listed in Tables 13-1 or 13-2. The quality control samples are processed through the digestion and analysis in the same manner as environmental samples.

- Method blank - an aliquot of deionized water that is digested with the sample batch.
- Laboratory Control Sample (LCS) - a spiked deionized water that is digested with the sample batch. A Laboratory Control Sample Duplicate (LCS DUP) may also be included.
- 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 4.0 mL of the 0.04 mg/L Hg spike solution (7.16) for each 36 mL of sample. The final concentration of spike in two spiked samples is 4.0 ug/L Hg.
- Sample duplicate - a sample prepared in duplicate, both carried through the batch digestion. (By client request only)

13.3 Interference Tests

To help verify the absence of matrix or chemical interference one interference test must be performed per batch or per matrix at a minimum. Depending upon the results of this test decisions will be made to report batch results or conduct further interference tests on some or all samples in the batch. Interference tests utilized could include the recovery test, serial dilution or method of standard additions.

13.3.1 Recovery Test

A post digestion spike (or single addition) is performed by pouring 10 ml of sample into first tube and 9 ml into a second tube. To the second is added a known volume of standard. The percent recovery is calculated (See Section 12.6) and compared to a control limit of 85-115%.

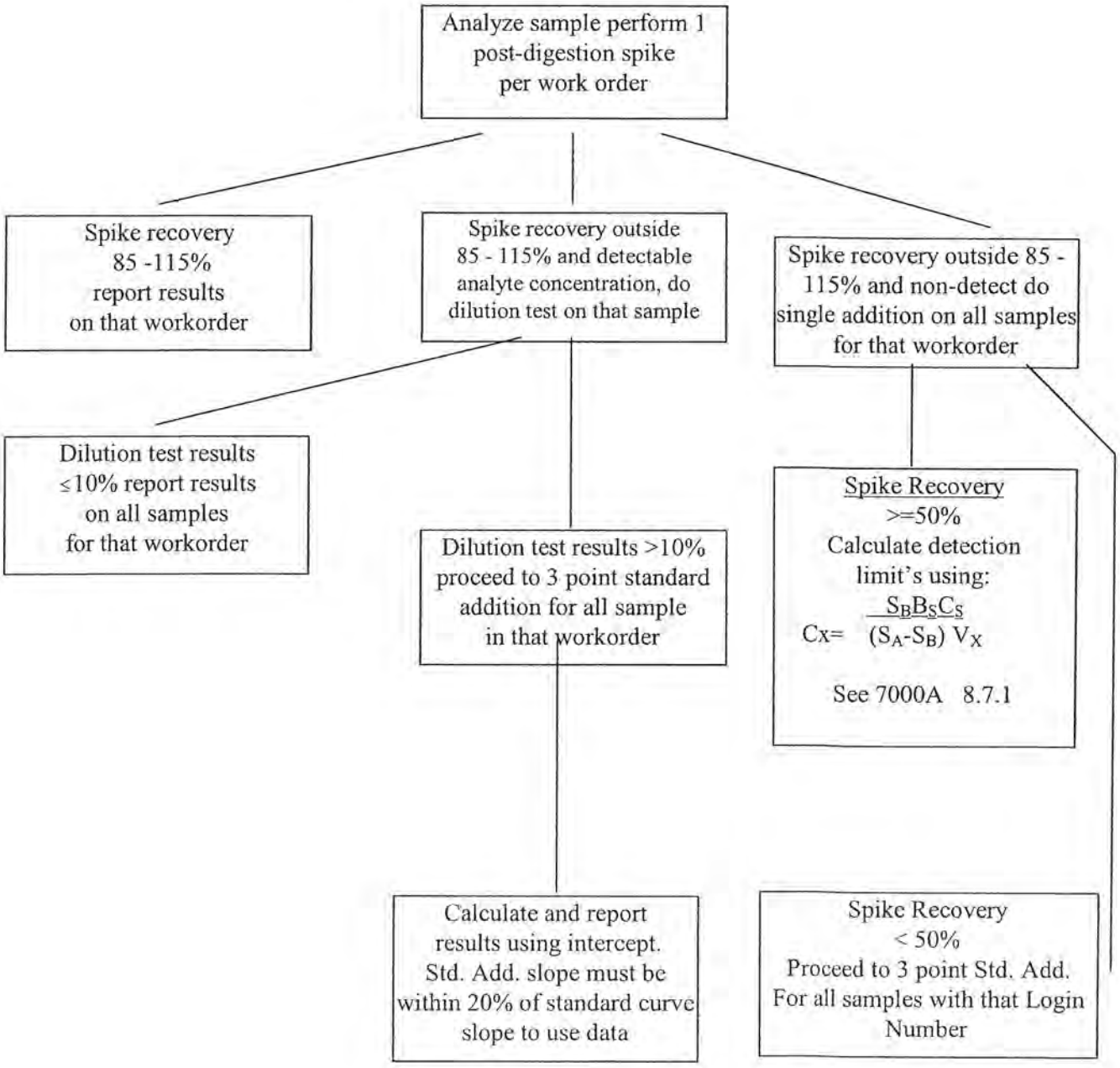
13.3.2 Serial Dilution

If the analyte concentration of a sample exceeds the MDL by a factor of 25 a serial dilution must be employed. This test is performed by diluting a sample five fold with deionized water. This accomplished by adding one mL of sample to four mL of deionized water. The results of the undiluted and diluted samples must agree within 10% when calculated as in section 12.7.

13.3.3 Method of Standard Additions

The method of standard additions is performed when post digestion spike recovery is below 50%; or when samples have detectable concentration (>25 x MDL), a post digestion spike fails and a serial dilution yields a result > 10% difference; or when TCLP analytes fall within 20% of the regulatory limit. Improved results can be obtained by employing a series of standard additions. 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% of the expected absorbance from the indigenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected indigenous sample absorbance. The absorbance of each solution is determined then plotted on the vertical axis of a graph, with the concentration of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the indigenous 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.

MERCURY INTERFERENCE CHECK FLOW CHART - WATERS



13.4 All batch quality control samples are subjected to exactly the same digestion as those used on actual samples in the digestion batch.

13.5 Contingencies for handling out-of-control or unacceptable data

13.5.1 Out-of-control situations may be addressed by one or more of the following approaches:

A. Internal documentation and communication (CAR)

B. Client notification

C. Re-digestion – in the event that sample redigestion is necessary due to quality control failure, new standards and quality control samples must be digested with the sample(s).

13.5.2 Out-of-control situations are documented on the corrective Action Request Forms (CAR) which are reviewed by the department manager and QA/QC supervisor. The project manager is copied on all corrective action forms so that they may inform the client affected and have the client assist with the decision of corrective action. These forms are kept on file and are available for review.

13.5.3 Any item not addressed here can be found in Section 13.0 Microbac Laboratories, Inc.'s Laboratory Quality Assurance Plan.

Table 13-1
Quality Control Criteria
Metals - Mercury
SW-846 Method 7470

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily (6 stds & Blank)	COC ≥ 0.995	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	80 - 120%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	≤ RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	Minimum every 10 samples	≤ RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	5%, or minimum of 1 per batch	≤ RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still > limit, redigest batch (required by Ohio VAP) or qualify data and address in narrative.
Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCS DUP)	5%, or minimum of 1 per batch	Control Limits 85 -115%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch (required by Ohio VAP) or qualify data and address in narrative
Matrix Spike/ Matrix Spike Duplicate	5% or minimum of 1 each per batch	85 - 115% Recovery RPD ≤ 20%	Perform post digestion spike or serial dilution. Qualify data and address in narrative if client specified.
Duplicate (Client Specified only)	5%, or minimum of 1 per batch	RPD ≤ 20%	Qualify data and address in narrative if client specified.
Interference Tests	per batch or per matrix as required by method	Post digestion spike 85 - 115% Serial dilution %D ≤ 10%	Data qualified as per method or perform method of standard additions

(1) Acceptance criteria are project specific, consult QAPP

Table 13-2

Quality Control Criteria
Metals - Mercury
EPA Method 245.1

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily (6stds & Blank)	COC $\geq$ 0.995	Recalibrate
Initial Calibration Verification (ICV)	After Calibration	95 - 105%	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
Initial Calibration Blank (ICB)	After ICV	$\leq$ RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	Minimum every 10 samples	$\leq$ RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	5%, or minimum of 1 per batch	$\leq$ RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still > limit, redigest batch (required for Ohio VAP or qualify data and address in narrative.
Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCS DUP)	5%, or minimum of 1 per batch	Control Limits 85 –115%	Stop analysis, investigate, reanalyze. If still outside limits redigest batch (required by Ohio VAP) or qualify data and address in narrative
Matrix Spike/ Matrix Spike Duplicate	5% or minimum of 1 each per batch	85 - 115% Recovery RPD $\leq$ 20%	Perform post digestion spike or serial dilution. Qualify data and address in narrative if client specified.
Duplicate (Client Specified only)	5%, or minimum of 1 per batch	RPD $\leq$ 20%	Qualify data and address in narrative if client specified.
Interference Tests	per batch or per matrix as required by method	Post digestion spike 85 - 115% Serial dilution %D $\leq$ 10% Method of Standard additions.	Data qualified as per method or perform method of standard additions.

(1) Acceptance criteria are project specific, consult QAPP

14.0 DATA REPORTING REQUIREMENTS

14.1 Data Review

14.1.1 Prior to data entry into the LIMS, all data must undergo thorough review in the department. This review will consist of 100% review by the primary analyst. This must be followed by a 100% review of each data package by another qualified analyst. The section supervisor or qualified designee must review each data package for completeness and adherence to Microbac analytical policies. The supervisor or qualified designee may conduct the 100% review of the data packages while reviewing for completeness and adherence to Microbac analytical policies provided s/he signs off on the review document in all applicable locations. The reporting requirements depend upon the need of the client. Microbac offers four levels of data reporting which are described in some detail below

14.1.2 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.

14.1.3 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.

14.1.4 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.

14.1.5 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 All data is calculated from the calibration curve and reported in units of mg/L or ug/L.

14.3 All data is reported using three significant figures.

14.4 See Figure 14.1 for Data Review Checklist.

15.0 PREVENTATIVE MAINTENANCE

15.1 Daily Maintenance

15.1.1 Check argon (>200 psi)

15.1.2 Perform lamp adjustment if needed.

15.1.3 Check 10% HCl rinse and refill as needed.

15.1.4 Check stannous chloride and refill as needed.

15.2 Weekly Maintenance

15.2.1 Pump tubing on the HYDRA AA analyzer must be changed when flat spots and loss of pliability are noticed. Tubing needs changed approximately weekly.

15.2.2 The autosampler rails on the analyzer should be lubricated weekly. Periodically clean with methanol before lubricating.

15.3 Yearly or as needed

15.3.1 Disassemble and clean optical cell and lenses.

15.3.2 Replace dehydrator tube.

15.4 Daily and Weekly preventative maintenance must be recorded on a preventative maintenance checklist.

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.

16.2 Each laboratory generated 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 containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside 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.

16.3 The following are waste streams in the sample digestion area.

16.3.1 Non-Halogentated solvents: Acetone

16.3.2 mercury: mercury waste

16.3.3 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

NOTE: 16.3.1, 2 and 3 are all kept in satellite containers in each lab and are combined into the proper 55 gallon waste drums in our explosion proof waste building located outside of the Microbac laboratory facility by a waste disposal technician. These drums are labeled and a manifest is created for each drum. 16.3.4 is neutralized and disposed in the municipal sewer system as per agreement with the city.

16.4 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 SOP's 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 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. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-70-020 (Rev. 3/1994), Method 245.1.

17.2 U.S. Environmental Protection Agency, Test Method for Evaluating Solid Waste, SW-846, September, 1994, Third Edition, Method 7470A..

17.3 U.S. Environmental Protection Agency, Contract Laboratory Program (CLP) Statement of Work (SOW ILMO4.0)

Figure 12.1

Example Cold Vapor Mercury Calculations
Hydra AA Mercury Analyzer

1.0 Initial Calibration (ICAL) Parameters
The system performs linear regression from data consisting of a blank and five standards

2.0 Calculating the concentration (C) of an element in water using data from 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 = Diluted to Volume (mL)	40
Vi = Aliquot Volume (mL)	40
D = Manual dilution factor, if required (10X = 10)	1
Cx = Concentration of element in ppb (ug/L)	0.1

3.0 Calculating the concentration (C) of an element in soil using data from prep log and quantitation report (note: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Ws} \times D$$

Where:	Example:
Cs = Concentration computed by the data system (ug/L)	0.1
Vf = Diluted to volume (mL)	40
Ws = Aliquot weight (g)	0.6
D = Manual dilution factor	1
Cx = Concentration of element in ug/kg	6.67

4.0 Adjusting the concentration to dry weight:

$$Cdry = \frac{Cx \times 100}{Px}$$

1 Cx = Concentration calculated as received (wet basis)	6.67
Px = Percent solids of sample (%wt)	80
$Cdry$ = Concentration calculated as dry weight (ug/kg)	8.33

8.33 ug/kg = 0.00833 mg/kg

Figure 14.1

Checklist ID: 2662

Microbac Laboratories Inc.

Data Checklist

Date: 13-MAY-2005
Analyst: KHR
Analyst: NA
Method: 6010B
Instrument: PE-ICP
Curve Workgroup: NA
Runlog ID:
Analytical Workgroups:

Run Log ID:	
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	X
Case Narrative	
Client Forms	X
Level X	
Level 3	
Level 4	
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	LSB
Comments	

Primary Reviewer: Kym H. Rhoden

Secondary Reviewer: Leslie Bucina

Appendix 1 - 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.

Calibration:

Process by which the correlation between instrument response and actual value of a measured parameter is determined.

Calibration Curve

A curve which plots the concentration of known analyte standards against the instrument response to the analyte. Also known as a Standard Curve.

Calibration Standard

Solutions or dilutions of a substance or material with a verifiable accuracy, which are used to evaluate the sample property of an unknown sample. In analytical terms, these standards are used to establish a calibration curve or standard instrument response factors.

Continuing Calibration Standard:

Standards that are analyzed during an analytical set to verify the accuracy of the calibration curve.

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 asses the effectiveness of equipment decontamination.

Extraction:

The process which releases or removes specific target analytes for subsequent analyses.

FID:

Flame Ionizaton 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 an 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.

Internal Standard

A compound having similar chemical characteristics to the compounds of interest, but which is not normally found in the environment or does not interfere with the compounds of interest. A known and specified concentration of the standard is added to each sample prior to analysis. The concentration in the sample is based on the response of the internal standard relative to that of the calibration standard and the compound in the standard.

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.

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.

Reporting Limit

The concentration of analyte, below which all quantitation are considered to be estimated. Instrument calibrations have a low standard at this level.

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.

Microbac

STANDARD OPERATING PROCEDURE

MERCURY
SW-846 Method 7471

Issue / Implementation Date: 15 December 2009

Last Review Date: 15 December 2009

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Robert E Kyer, Digestion Group Leader	12/15/09 Date
Mareh M. Beery, Metals Supervisor	12/15/09 Date
David L. Bumgarner, Technical Director/QAO	12/16/09 Date
David E. Vandenberg, Managing Director	12/14/09 Date

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure provides sample preparation methods and details the operation of a Leeman HYDRA AA Autoanalyzer for the analysis of mercury (CAS # 7439-97-6) in soil, sediment, sludges, oils, and bottom deposits, consistent with the protocol for SW-846 7471A and 7471B.

1.2 Mercury analysis is based on a cold-vapor atomic absorption technique. Mercury is reduced to the elemental state and purged with argon to form elemental mercury vapor. The mixture flows through a drying tube for water vapor removal and then enters one path of a double path optical cell. A mercury source, powered by a constant current power supply, delivers a stable source of emission at 253.7 nm. Absorbance by the mercury cold vapor is measured using a solid state detector with a wide dynamic range. The resulting signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions.

1.3 In addition to inorganic forms of mercury, organic mercury compounds may also be present. These compounds will not respond to the cold vapor technique unless they are broken down into mercuric ions. Potassium permanganate oxidizes most, but not all, of these compounds.

2.0 SAFETY PRECAUTIONS

2.1 **CAUTION** - Acids used in this method for the determination of mercury can burn skin and cause blindness. Contact must be avoided by using the appropriate personal protective equipment.

2.2 **WARNING** - Voltages as high as 220 VAC exist inside the instrument so always disconnect the instrument from the wall before opening the chassis. Wait 10 minutes after disconnecting the power to allow time for all capacitors to dissipate all energy.

2.3 Always wear a lab coat, safety glasses, and latex gloves to reduce possible health hazards to the lowest possible level.

2.4 The laboratory maintains MSDS sheets for handling and exposure information.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 All samples must be collected by the use of techniques that prevent contamination and cross-contamination between samples.

3.2 Sample containers are I-Chem 300 series glass jars with teflon lined lids. See Table 3-1 for collection and digestion volumes and hold times.

Table 3-1

Measurement	Digestion Weight Requirement	Collection Weight Requirement	Preservation	Holding Time *
Soil	0.6 grams	200 grams	4° C	28 Days

* Storage time allowed between sample collection and analysis when properly preserved and stored.

4.0 METHOD PERFORMANCE

4.1 Mercury in solid waste is analyzed at a wavelength of 253.7 nm with a reporting limit of 0.100 mg/kg with an upper limit of 0.667 mg/kg.

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 of detection (LOD), or verified MDL, is presented in Table 4-1 and was established using verification procedures outlined in SOP 45.

4.4 The limit of quantitation (LOQ) is the nominal laboratory reporting limit(s) (RL) and is established per SOP 45. Actual project reporting limits may be higher.

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 sample (LCS) to assess precision/accuracy and to annually evaluate the associated control limits.

Table 4-1
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR INORGANIC METALS ANALYSES SOIL AND SOLID WASTE

PARAMETER	CAS #	EPA SW-846 METHOD	ACCURACY (% Recovery)	PRECISION (% RPD)	VERIFIED MDL SOIL (mg/kg)	REPORTING LIMIT SOIL (mg/kg)
Mercury (Hg) - VAPOR	7439-97-6	7471	80-120	0-20	0.01	0.100

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Possible interference from sulfides is eliminated by the addition of potassium permanganate.

5.2 Copper has been found to interfere; however concentrations as high as 10 mg/kg had no effect on spike recovery.

5.3 Samples high in chlorides may require additional permanganate. Care must be taken that free chlorine is absent before the mercury is reduced. This can be accomplished by using an excess of hydroxylamine hydrochloride reagent.

5.4 Acetone also absorbs at 254 nm and gives a false positive signal.

6.0 EQUIPMENT AND SUPPLIES

6.1 Major Instrumentation

6.1.1 Leeman HYDRA AA Automated Mercury Analyzer.

6.2 Apparatus or equipment

6.2.1 Adjustable volume pipettor 100-1000 uL.

6.2.2 Adjustable volume pipettor 1-10 mL

6.2.3 Argon gas supply.

6.2.4 36 to 54 Well Hot Block Digestor

6.2.5 Polypropylene digestion tubes (50 mL)

6.2.6 Class A pipets (various sizes)

6.2.7 Digital Thermometer

6.2.8 Refer to Microbac SOP ME201 for glassware cleaning procedures.

7.0 STANDARDS AND REAGENTS

7.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. 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 and RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT Number.

Calibration Solutions

7.2 Calibration Stock Mercury Solution: A purchased certified standard of 1000 ug/mL mercury-Specpure.

7.3 Intermediate Calibration Mercury Solution – prepared by diluting 5.0 mL of the calibration stock mercury solution (7.2) to 500 mL with 10 mL HNO_3 and deionized water. The concentration is 10 ug/mL Hg. The shelf life is 6 months or the stock solution expiration whichever is sooner.

7.4 Working Calibration Mercury Solution: Prepare fresh daily by diluting 2.0 mL of the calibration intermediate mercury solution (7.3) to 500 mL with 10 mL HNO_3 and deionized water. The concentration of the working calibration solution is 0.040 ug/mL Hg.

7.5 Calibration Standard preparation: Transfer 0.0, 0.2, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the 0.04 ug/mL mercury working standard (Section 7.4) to a series of 50 mL graduated polypropylene tubes. Add enough deionized water to each tube to make a total volume of 10 mL. To all standards, add 1.25 mL HN03, 3.75 ml HCl and heat 2 minutes at 95 + 3°C. Allow the sample to cool. Add 15 mLs of KMn03. Dilute to a 40 mL total volume with deionized water. Process the standards following the same procedure as the samples. (See Section 9.0) Standard concentrations are 0.2, 1.0 2.0 5.0 and 10.0 ug/L. The sixth polypropylene tube initially containing only deionized water will serve as the calibration blank. Standards should be made fresh each day in which samples are digested and digested with the sample batch.

Initial Calibration Verification Solutions

7.6 Initial Calibration Verification (ICV) Mercury Solution: A purchased certified standard of 1000 ug/mL. This standard must be purchased from a vendor independent of the calibration stock mercury standard (7.2). – Inorganic Ventures

7.7 Intermediate ICV Mercury Solution: Dilute 5.00 mL of the ICV stock mercury solution (7.6) to 500 mL total volume with 10 mL HNO_3 and deionized water. The concentration of the intermediate ICV solution is 10.0 ug/mL mercury. The shelf life is 6 months or the same as the stock solution expiration, whichever is sooner.

7.8 0.0400 ug/mL Mercury Spike Solution: Prepare fresh daily by diluting 2.0 mL of the intermediate ICV mercury solution (7.7) to 500 mL with 10 mL HNO_3 and deionized water. Concentration is 0.0400 ug/mL mercury.

7.9 ICV Solution: Transfer 2.0 mL of the 0.0400 ug/ml mercury spike solution (7.8) to a 50 mL graduated polypropylene tube. Add a sufficient volume of deionized water to bring to a 10 mL total volume. Add all other reagents as discussed in Section 7.5 (calibration standard preparation) and digest with the sample batch. This yields a final concentration of 0.0020 ug/mL.

Additional Reagents

7.10 Teflon chips.

7.11 Nitric Acid (HNO3), Concentrated: Baker Instra Analyzed grade or better.

7.12 Hydrochloric Acid (HCl), Concentrated: Baker Instra Analyzed grade or better.

7.13 Stannous Chloride Solution: Dissolve 50 grams of Stannous Chloride in 50 mL concentrated HCl and dilute to 500 mL with acidified deionized water.

7.14 Hydroxylamine Hydrochloride Solution: Dissolve 120 grams of Hydroxylamine Hydrochloride Crystal and 120 grams of Sodium Chloride Crystal in deionized water and dilute to 1000 mL.

7.15 Deionized Water (ASTM Type II)

7.16 Potassium Permanganate Solution: ($KMnO_4$) Dissolve 100 grams of potassium permanganate in 2000 mL of deionized water.

8.0 CALIBRATION PROCEDURES

8.1 Initial calibration – The instrument is calibrated before analysis of any samples with a blank and five calibration standards (7.5). A linear curve must be selected. The first standard run must be the blank followed by standards of increasing concentration in order to minimize cross contamination and carry over. The low calibration standard must contain mercury at a concentration at or below the reporting limit. See Table 13-1 and for acceptance criteria and corrective action for the initial calibration.

8.2 Initial calibration verification (ICV) Analysis – ICV analysis (7.9) must be performed immediately after calibration standards to verify calibration. See Table 13-1 for acceptance criteria and corrective actions.

8.3 Continuing calibration verification (CCV) Analysis – The CCV solution is the 2.0 ug/L standard from section 7.5. The CCV is required to be analyzed prior to sample analysis, after every ten analytical samples and at the end of analysis. See Table 13-1 and for acceptance criteria and corrective actions.

8.4 Initial and continuing calibration blank (ICB/CCB) Analysis – The solution used is the calibration blank from section 7.5. See Table 13-1 for acceptance criteria and corrective actions.

8.5 See Figure 8-1 for analytical sequence.

Figure 8-1
Analytical Sequence

Calibration (See Section 11.4)
ICV
ICB
CCV
CCB
10 or less samples
CCV
CCB
Repeat CCV/CCB every 10 samples
Always end run with CCV/CCB

9.0 SAMPLE PREPARATION

9.1 Sample digestion

9.1.1 Weigh out 0.6 grams of representative sample into a clean labeled polypropylene tube.

9.1.2 Spike LCS and MS, MSD with 4.0 mL each of mercury spike solution (7.8).

9.1.3 Add 5 mL of water to all samples including Blank and LCS.

9.1.4 Add 3.75 mL of HCl and 1.25 mL of HN03.

9.1.5 Heat for two (2) minutes at 95° C ± 3°C.

9.1.6 Add 15 mL of 5% Potassium permanganate.

9.1.7 Add sufficient amount of DI water to make final volume 40 mL, let stand for 15 min, to assure samples maintain a purple color. If purple color fades add additional permanganate until color persists for 15 minutes.

9.1.8 Place a cap on the samples and turn back one (1) turn to allow for venting.

9.1.9 Digest in hot block for 30 minutes at 95°C ± 3°C. Cool and make sure the volume remains at 40 mL, if not add DI water. After final volume determination, add 6 mL of 12% hydroxylamine hydrochloride.

9.2 Method Blank - Weigh 0.6 g of Teflon chips into a digestion vessel. Treat the same as sample in Section 9.1.

9.3 Laboratory Control Sample (LCS/LCS Dup) – Weigh 0.6 g of Teflon chips into a digestion vessel and add 4 mL of spike solution (7.8). Treat as sample section 9.1. The concentration of the LCS is 267 ug/kg. An LCS Duplicate will be prepared if there is insufficient sample volume for MS/MSD analysis.

9.4 Matrix spikes – Prepare a matrix spike (MS) and matrix spike duplicate (MSD) by weighing two separate aliquots of one of the client samples. To each aliquot in the digestion vessel add 4 mL of spike solution (7.8). Treat as sample Section 9.1. The concentration of the spike solution is 267 ug/kg.

9.5 Laboratory Duplicate (By client request only) – weigh a second aliquot of one of the client samples. Treat as sample Section 9.1.

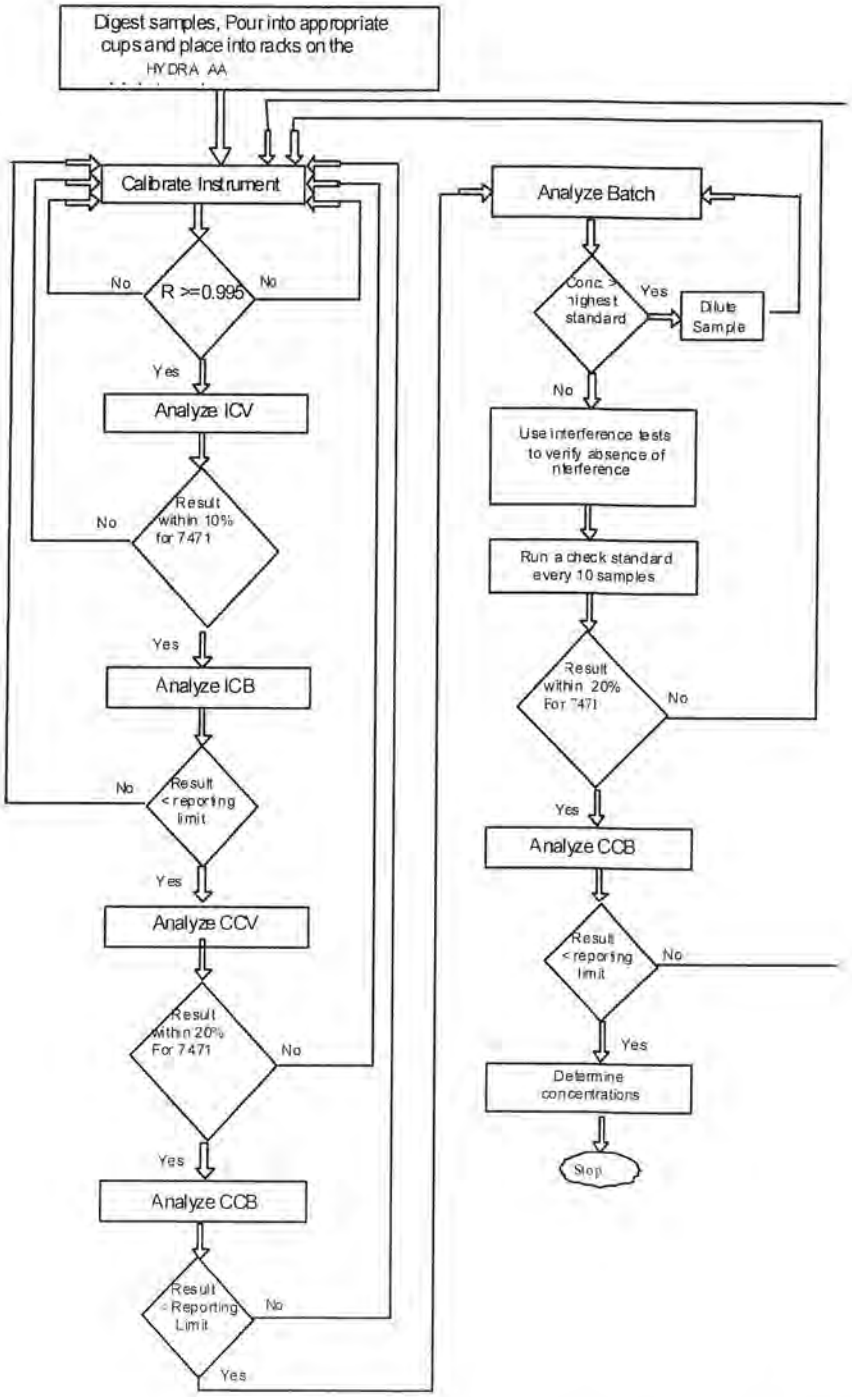
9.6 Hot Block Temperature Check

Each day they are to be used, the hot blocks are switched on and allowed to warm up. Each hot block is equipped with a digital thermometer suspended in DI water in a sealed digest tube. The tube is rotated daily to a new well position. Following the warmup interval, the well position, date and temperature are recorded in a notebook specific to each hot block. The temperature criteria is 95 ± 3° C.

Prior to beginning a sample digestion the temperature is again read and recorded on the digestion log as an initial temperature. At the conclusion of sample digestion, a final temperature is also recorded on the digestion log.

9.7 See Figure 9.1 for an example of the Metals Digestion Log.

10.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



11.0 STEP-BY-STEP ANALYTICAL PROCEDURES

11.1 HYDRA AA autoanalyzer Instrument Startup

11.1.1 Turn on the power strip, computer and gas supply (>200 lbs/in^2)

11.1.2 Log into system and click on WinHg icon. This opens up WinHg Runner 1.4 window.

11.1.3 Click on Control and turn Hg lamp on by clicking the On button.

11.1.4 Check pump tubing for wear and replace if necessary.

11.1.5 Check Stannous chloride and rinse solution volumes and refill if needed.

11.2 Retreive the proper protocol, standard. Main menu protocol, click on down arrow then on standard.

11.3 To create a new dataset, from the main menu click on file, new dataset, enter date, then ok. Batch ID will appear, enter date, then OK.

11.4 Calibrate the instrument using the calibration standard solutions.

11.4.1 Transfer the standards and check standards to the appropriate cups in the calibration rack. Load the standard rack into its proper position on the HYDRA AA autoanalyzer.

11.4.2 Click on Standard, then on S1-S6 and rep 1, then on Stnd Auto. Before calibrating make sure calibration setting is on linear setting. Calibration begins and Standards 1-6 are analyzed. The absorbance is determined from one replicate reading of the standards.

11.4.3 To review calibration results click on the down arrow at the top of main menu. This opens up WinHg Data base 1.4. Click on Cal Curve to display results. If the correlation coefficient is > or = 0.995 click on accept. This stores the new calibration coefficients.

11.4.4 Print the calibration curve and results by clicking on the button resembling a printer.

11.5 Analyze the ICV/ICB, CCV/CCB solutions, Load the ICV in position #9, ICB in position #10, CCV into position #11 and CCB into position #12. Click on buttons C2-C5 then CK STD Auto. This starts analysis of check standards.

11.6 Load samples into cups in sample rack making appropriate dilutions and spikes where necessary. Filter samples containing particulates prior to filling cups. Load the rack in the proper position in the HYDRAA AA Auto analyzer. From the main menu click on the button resembling a sample rack. Enter sample IDs and C4 space C5 every 10 samples and/or after the last sample in the right hand column. This tells the instrument to analyze check standards. Click on File, Save As, Choose a file name, then click on Save. Close Rack Editor window.

11.7 From the main menu click on Sample, enter Rack Name, enter Start Cup and End Cup.

11.8 Click on Run Auto button to start sample analysis. Monitor run for CCV and CCB acceptance. See Table 13-1 for acceptance criteria. The sample concentration is determined from one replicate reading of the sample.

11.9 After analyses are complete, place stannous tubing in a 10% HCl Solution and rinse for 4-5 minutes then place stannous and rinse tubing in DI water and rinse for 4-5 minutes. Pull tubing out and raise sample probe and allow all tubing to pump dry. Shut off lamp, pump and gas. Exit out of software and shut down computer. Turn off power strip. Release tension on pump tubing.

11.10 Upload data into LIMS. Review batch QA/QC forms while creating PDF format.

12.0 DETAILS OF CALCULATION

After the calibration is complete, the software performs a linear regression with a calculated intercept which is not forced through zero. The regression equation is given by $I = BX + C$

where:

I = mean intensity of standard

B = slope of ICAL

X = concentration of standard

C = intercept of ICAL

The coefficient of correlation (COC) is calculated to verify the linearity of the calibration curve. The COC is required to be ≥ 0.995 in order for the curve to be valid.

$$COC = \frac{\Sigma xy - \frac{\Sigma x \Sigma y}{n}}{\sqrt{\left(\Sigma x^2 - \frac{(\Sigma x)^2}{n}\right)\left(\Sigma y^2 - \frac{(\Sigma y)^2}{n}\right)}}$$

where:

X = standard concentration
Y = mean intensity
N = 6 = number of standards

12.1 Results are calculated from the calibration curve by the system software utilizing linear regression. Calibration standards and samples all undergo one replicate reading. Dilution factors and preparation information are applied by the LIMS system during data upload.

Soil samples:

$$\text{Concentration (ug/kg)} = \frac{(I-C)\,V}{BDW}$$

where:

B = slope from instrument ICAL
C = intercept from instrument ICAL
I = mean sample intensity
D = decimal dilution factor (eg - 10X = 0.1)
V = Final digestion volume (mL) (40mL= 0.040L)
W = initial weight of sample digested (g) (0.6g = 0.0006kg)

Nominal initial weight and final digestion volume are listed in (parentheses) for all samples.

12.2 LCS % recovery is calculated as follows:

$$\left(\frac{C_x}{C_T}\right)100 = \text{LCS \% recovery}$$

where:

C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.

12.3 Relative Percent Difference (RPD) results are 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

12.4 Matrix Spike recovery is calculated as follows:

$$\left[\frac{(C_{spk} - C_x)}{C_t}\right]100 = \text{\% recovery}$$

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.

12.5 Post digestion spike recovery is calculated as follows:

$$\left[\frac{(C_{spk} - C_x)}{C_t}\right] * 100 = \text{\% Recovery}$$

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.

12.6 Serial dilution percent difference is calculated as follows:

$$\frac{C_x - [C_{DL} * 5]}{C_x} * 100 = \%Difference$$

C_x = the concentration of the analyte in the reference (parent) sample
C_{dl} = the concentration of the analyte in the five-fold dilution.

12.7 See Figure 12.1 for a calculation summary.

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 and LCS DUP), matrix spikes (MS), matrix spike duplicates (MSD), laboratory duplicates (LD). 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 corrective action measures are outlined in Tables 13-1. See section 16.0 Microbac Laboratories, Inc. Laboratory Quality Assurance Plan for QC requirements specific to the VAP program.

13.2 With each batch of twenty (20) samples or less the following quality control samples will be analyzed and must meet the criteria listed in Table 13-1.

- Method blank - an aliquot of deionized water that is digested with the sample batch.
- Laboratory Control Sample (LCS). A Laboratory Control Sample Duplicate (LCS Dup) may also be included - (Section 9.3).
- Matrix spike and matrix spike duplicate - a sample that is spiked in duplicate and then digested with the sample batch - (Section 9.4).
- Laboratory duplicate - a sample prepared in duplicate, both carried through the batch digestion. (By client request only)

13.3 Interference Tests

To help verify the absence of matrix or chemical interference one interference test must be performed per workorder or per matrix at a minimum. Depending

upon the results of this test decisions will be made to report batch results or conduct further interference tests on some or all samples in the batch. Interference tests utilized could include the recovery test, serial dilution or method of standard additions.

13.3.1 Recovery Test

A post digestion spike (or single addition) is performed by taking one 10 ml aliquot and one 9 ml aliquot of sample. To the 9ml aliquot is added a 1ml volume of standard. The percent recovery is calculated (See Section 12.5) and compared to a control limit of 85-115%.

13.3.2 Serial Dilution

If the analyte concentration of a sample exceeds the MDL by a factor of 25 a serial dilution must be employed. This test is performed by diluting a sample five fold with deionized water. This is accomplished by adding one mL of sample to 4 mL deionized water. The results of the undiluted and diluted samples must agree within 10% as calculated in Section 12.6.

13.3.3 Method of Standard Additions

Method of Standard additions is performed when post spike recovery is below 50%; or if samples have detectable concentration (>25x MDL), a post digestion spike fails and a serial dilution test yields a result > 10% difference; or when TCLP analytes fall within 20% of the regulatory limit.

Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample, standard is added in a series of 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% of the expected absorbance from the indigenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected indigenous sample absorbance. The absorbance of each solution is determined then plotted on the vertical axis of a graph, with the concentration of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the indigenous 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.

13.4 All quality control samples are digested and analyzed identically as environmental samples.

13.5 Control of Nonconforming

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).

13.6 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 13-1 does not meet acceptance criteria. When nonconforming data occurs the employee initiates a Nonconformance Report (NCR) and proceeds with indicated corrections as per Table 13.1.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch Figure 14.1. 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 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-RAC, corrections, corrective action (s) and evidence of effectiveness.

13.8 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 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.

13.9 Items not addressed here can be found in Section 13.0 SOP Laboratory Quality Assurance Program.

Table 13-1
Quality Control Criteria
Metals -Graphite Furnace and Mercury
SW-846 Method 7471

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily (5 stds & Blk)	COC > or = 0.995	Recalibrate, if still outside limits redigest entire batch and associated standards.
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	80 - 120%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	≤ RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	Minimum every 10 samples	≤ RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Method Blank	5%, or minimum of 1 per batch	≤ RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still > limit, redigest batch (required for Ohio VAP) or qualify data and address in narrative.
Laboratory Control Sample (LCS) Laboratory Control Sample Duplicate (LCS Dup)	5%, or minimum of 1 per batch	Control Limits 80 -120%	Stop analysis, investigate, reanalyze. If still outside limits redigest batch (required for Ohio VAP) or qualify data and address in narrative
Matrix Spike/ Matrix Spike Duplicate	5% or minimum of 1 each per batch	80 - 120% Recovery RPD ≤ 20%	Perform post digestion spike or serial dilution. Qualify data and address in narrative if client specified.
Duplicate(Client Specific only)	5%, or minimum of 1 per batch	RPD ≤ 20%	Qualify data and address in narrative if client specified.
Interference Tests	per batch or per matrix as required by method	Post digestion spike 85 - 115% Serial dilution %D ≤ 10%	Data qualified as per method or perform method of standard additions analysis

14.0 DATA REPORTING REQUIREMENTS

14.1 Data Review

14.1.1 The reporting requirements depend upon the need of the client. MICROBAC offers four levels of data reporting which are described in some detail below. Prior to data entry into the LIMS, (either manual or automatic), all data must undergo thorough review in the department. This review will consist of 100% review by the primary analyst. This must be followed by a 100% review of each data package by another qualified analyst. The section supervisor or qualified designee must review each data package for completeness and adherence to Microbac analytical policies. The supervisor or qualified designee may conduct the 100% review of the data packages while reviewing for completeness and adherence to Microbac analytical policies provided she/he signs off on the review document in all applicable locations.

14.1.2 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.

14.1.3 Level 2 reporting provides the client with all of the information contained in a Level 1 report plus a summary of all of the batch QC analysis associated with the samples submitted by the client.

14.1.4 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.

14.1.5 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 All data is calculated from the calibration curve and reported in units of mg/kg or ug/Kg.

14.3 All data is reported using three significant figures.

14.4 See Figure 14-1 for data review checklist.

15.0 PREVENTIVE MAINTENANCE

15.1 Daily Maintenance

15.1.1 Check argon pressure (>200 psi)

15.1.2 Perform lamp adjustment if needed.

15.1.3 Check 10% HCl rinse and refill as needed.

15.1.4 Check stannous chloride and refill as needed.

15.2 Weekly Maintenance

15.2.1 Pump tubing on the HYDRA AA analyzer must be changed when flat spots and loss of pliability are noticed. Pump tubing needs replaced approximately weekly.

15.2.2 The autosampler rails must be lubricated weekly. Periodically clean with methanol before lubricating.

15.3 Yearly or as needed

15.3.1 Disassemble and clean optical cell and lenses.

15.3.2 Replace dehydrator tube

15.4 Daily and weekly preventive maintenance must be recorded on a preventive maintenance checklist.

16.0 WASTE MANAGEMENT

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste, which requires disposal or contributes to pollution of any type.

16.2 Each laboratory in the sample preparation department generates specific waste streams, which are segregated and collected in labeled satellite containers. Each analyst in the sample preparation department is responsible for proper disposal of spent samples and chemical waste in the specified satellite container daily or as needed.

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.

16.4 The following are waste streams in the sample preparation and analysis areas.

16.4.1 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

NOTE: 16.4.1 is kept in satellite containers in each lab and is combined into the proper 55 gallon waste drums in our explosion proof waste building located outside of the Microbac laboratory facility by a waste disposal technician. .

16.4.2 Mercury Digestions

16.4.2 is neutralized and disposed in the municipal sewer system as per agreement with the city.

17.0 REFERENCES

17.1 Mercury in Solid or Semi Solid Waste (Manual Cold Vapor Technique), US EPA SW-846 Method 7471A, Revision 1, September 1994, EPA publication SW-846.

17.2 Mercury in Solid or Semi Solid Waste (Manual Cold-Vapor Technique), US EPA SW-846 Method 7471B, Revision 2, February 2007, EPA publication SW-846.

17.3 Flame Atomic Absorption Spectrophotometry, US EPA SW-846 Method 7000B, Revision 2, February 2007, EPA publication SW-846

17.4 SOP 45 "Method Validation Procedures"

17.5 SOP LQAPP "Laboratory Quality Assurance Plan"

17.6 SOP ME 201 "Cleaning Procedure for Preparation of Glassware for the Analysis of Trace Metals"

17.7 SOP 33 "Laboratory Waste Management"

17.8 SOP GP-CAPA "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

17.9 SOP GP-RCA "Root Cause Anaysis"

Figure 9.1

Microbac Laboratories Inc.
Metals Digest Log

Workgroup:WG311556
Analyst:BRG
Spike Analyst:BRG
Method:7471A
Run Date:09/08/2009 08:28
Hotblock Start Temp:92.5 @ 09:05
Hotblock End Temp:92.4 @ 09:35

SOP:ME405 Revison 9
Spike Solution:STD35125
Spike Witness:ERP
KMnO4 1:1 Lot #:K0T14157
HNO3 Lot #:COA13945
HCL Lot #:COA14139
Digest tubes Lot #:COA14115
HG SOIL STD 10PPM Lot #:STD35132
HG SOILS ICV Lot #:STD35133

	SAMPLE #	Type	Matrix	Initial Amount	Final Volume	Spike Amount	Due Date
1	WG311556-03	BLANK	S	.6 g	40 mL		
2	WG311556-04	LCS	S	.6 g	40 mL	4 mL	
3	L09090126-01	SAMP	S	.614 g	40 mL		09/09/09
4	L09090127-02	SAMP	S	.574 g	40 mL		09/15/09
5	WG311556-01	PBP	S	.597 g	40 mL		
6	L09090127-03	MS01	S	.597 g	40 mL		09/15/09
7	WG311556-04	MS	S	.497 g	40 mL	1 mL	
8	L09090127-04	MS01	S	.497 g	40 mL	1 mL	09/15/09
9	WG311556-05	MSD	S	.497 g	40 mL	1 mL	
10	L09090127-05	ADD1	S	.497 g	40 mL	4 mL	09/15/09
11	L09090127-06	SAMP	S	.422 g	40 mL		09/15/09
12	L09090127-07	SAMP	S	.61 g	40 mL		09/15/09
13	L09090131-02	SAMP	SO	.538 g	40 mL		09/15/09

Analyst: Brad Gregory

Reviewer:

Figure 12.1

Example Cold Vapor Mercury Calculations
Hydra AA Mercury Analyzer

1.0 Initial Calibration (ICAL) Parameters
The system performs linear regression from data consisting of a blank and five standards.

2.0 Calculating the concentration (C) of an element in water using data from 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 = Diluted to Volume (mL)	40
Vi = Aliquot Volume (mL)	40
D = Manual dilution factor if required (10X = 10)	1
Cx = Concentration of element in ppb (ug/L)	0.1

3.0 Calculating the concentration (C) of an element in soil using data from prep log and quantitation report (note: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Ws} \times D$$

Where:	Example:
Cs = Concentration computed by the data system (ug/L)	0.1
Vf = Diluted to volume (mL)	40
Ws = Aliquot weight (g)	0.05
D = Manual dilution factor	1
Cx = Concentration of element in ug/kg	0.67

4.0 Adjusting the concentration to dry weight:

$$Cdry = \frac{Cx \times (\%S)}{1\%}$$

Cx = Concentration calculated as received (wet basis)	0.67
$\%S$ = Percent solids of sample (% wt)	80
$Cdry$ = Concentration calculated on dry weight (ug/kg)	8.33

8.33 ug/kg = 0.00833 mg/kg

Figure 14.1

Checklist ID: 2662

Microbac Laboratories Inc.

Data Checklist

Date: 13-MAY-2005
Analyst: KHR
Analyst: NA
Method: 6010B
Instrument: PE-ICP
Curve Workgroup: NA
Runlog ID:
Analytical Workgroups:

Run Log ID:	
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	X
Case Narrative	
Client Forms	X
Level X	
Level 3	
Level 4	
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	LSB

Comments

Primary Reviewer: Secondary Reviewer:

CHECKLIST1 - Modified 03/05/2008
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Microbac

Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method:

HCl – hydrochloric acid

$HN0_3$ – nitric Acid

COC – coefficient of correlation

AA – atomic absorption

For a more comprehensive list of common terms and definitions, consult Appendix A in SOP LQAP

Microbac

STANDARD OPERATING PROCEDURE SONICATION EXTRACTIONS FOR SEMIVOLATILES METHOD 3550B AND 3550C

Issue/Implementation Date: 15 January 2010

Last Review Date: 15 February 2010

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved By:

Chad E. Barnes, Extraction Supervisor — Date 2/9/10

David L. Bumgarner, Technical Director/QAO — Date 2/11/10

David E. Vandenberg, Managing Director — Date

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

This method describes a procedure for extracting and isolating semivolatiles from solid samples. The method also describes the concentration and procedure involved in preparing the extract for analysis by GC/MS. This method follows the procedures outlined in SW-846 Method 3500C and 3550B and 3550C.

2.0 SAFETY PRECAUTIONS

2.1 Proper gloves, lab coat, safety glasses, and ear protection must be worn during the extraction.

2.2 The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined. However, each chemical compound should be treated as a potential health hazard.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Samples should be collected in a widemouth glass container with teflon caps.

3.2 Sample weight needed for analysis is 30 grams but if sample weight is less than 30 grams, record the actual weight of sample and contact the TSR.

3.3 Sample preservation should be at, 4° C, and the maximum holding time from date of collection is 14 days.

4.0 INTERFERENCES AND CORRECTIVE MEASURES

4.1 Interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing.

4.2 Other interferences that may be encountered are discussed in Method 3500B and 3500C from SW846.

5.0 EQUIPMENT AND SUPPLIES

5.1 Major Instrumentation

5.1.1 Zymark Turbo Vap II concentration workstation

5.1.2 Ultrasonic cell disrupter: Misonix Model XL2020 sonicator or equivalent with a ¾" tapped disrupter horn.

5.1.3 Nitrogen Evaporator: Meyer N-Evap Analytical Evaporator or equivalent

5.2 Apparatus or equipment

5.2.1 Kuderna-Danish (K-D) apparatus:

5.2.1.1 Concentrator tube: 15 mL graduated (Supelco 6-4684M or equivalent)

5.2.1.2 Evaporation Flask: 500 mL attached to a concentrator tube with a clip or spring.

5.2.1.3 Snyder column: Three ball macro

5.2.2 Water bath: Heated, with concentric ring cover, capable of temperature control (± 5° C). The bath should be used in a hood.

5.3 Glassware

5.3.1 Beakers: 250 mL

5.3.2 Erlenmeyer flask: 250mL

5.3.3 Autosampler vials: 2 mL capacity

5.3.4 Zymark tubes 200 mL (with a 1.0 mL stem)

5.4 Other Supplies

5.4.1 Stainless steel funnel or glass funnel

5.4.2 Boiling chips: 10/40 mesh

5.4.3 Syringes – various sizes

5.4.4 Filter paper: Whatman #41 or equivalent

6.0 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.

6.1 Deionized water.

6.2 Acetone: Reagent grade or equivalent.

6.3 Sodium sulfate: Granular, anhydrous.

6.4 Sodium sulfate: Powdered, anhydrous.

6.5 Methylene chloride/Acetone mix (1:1): Pesticide grade or equivalent.

6.6 Sulfuric acid solution (1:1): Slowly add 50 mL of sulfuric acid to 50 mL of deionized water.

6.7 BNA surrogate: Methanol solution (NSI or equivalent) prepared at the working concentration:

2-fluorobiphenyl	100 ug/mL
Nitrobenzene-d5	100 ug/mL
p-terphenyl-d14	100 ug/mL
2-fluorophenol	200 ug/mL
2,4,6-tribromophenol	200 ug/mL
Phenol-d6	200 ug/mL

6.8 BNA Spike: Methanol solution containing 100 ug/mL - 71 Compounds:

Acenaphthene	2,4-Dinitrophenol
Acenaphthylene	2,4-Dinitrotoluene
Anthracene	2,6-Dinitrotoluene
Aniline	Diethylphthalate
Azobenzene	Dimethyl phthalate
Benzidine	Di-n-butyl phthalate
Benzyl alcohol	Di-n-octyl phthalate
Benzo(a)anthracene	bis(2-Ethylhexyl)phthalate
Benzo(b)fluoranthene	Fluoranthene
Benzo(k)fluoranthene	Fluorene
Benzo(g,h,i)perylene	Hexachlorobenzene
Benzo(a)pyrene	Hexachlorobutadiene
Benzoic acid	Hexachlorocyclopentadiene
4-Bromophenyl phenyl ether	Hexachloroethane
Butyl benzyl phthalate	Indeno(1,2,3-c,d)pyrene
Carbazole	Isophorone
4-Chloroaniline	2-Methylnapthalene
Chrysene	Naphthalene
bis(2-Chloroethoxy)methane	o-Nitroaniline
bis(2-Chloroethyl)ether	m-Nitroaniline
bis(2-Chloroisopropyl)ether	p-Nitroaniline
4-Chloro-3-methyl phenol	Nitrobenzene
2-Chloronaphthalene	2-Nitrophenol
2-Chlorophenol	4-Nitrophenol
4-Chlorophenyl phenyl ether	N-Nitrosodimethylamine
o-Cresol	N-Nitrosodiphenylamine
p-Cresol	N-Nitrosodi-n-proplamine
Dibenz(a,h)anthracene	Pentachlorophenol
Dibenzofuran	Phenanthrene
1,2-Dichlorobenzene	Phenol
1,3-Dichlorobenzene	Pyrene
1,4-Dichlorobenzene	Pyridine
3,3'-Dichlorobenzidine	1,2,4-Trichlorobenzene
2,4-Dichlorophenol	2,4,5-Trichlorophenol
2,4-Dimethylphenol	2,4,6-Trichlorophenol
4,6-Dinitro-2-methyl phenol	

NOTE: The spike contains the following twelve (12) compounds in addition to the 71 compounds previously listed. These are only calibrated when project QAPP's specify.

Acetophenone
n-Decane
2,3-Dichloroaniline
1-Methylnaphthalene
n-Octadecane
a-Terpineol
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
2,3,4,6-Tetrachlorophenol
2,3,5,6-Tetrachlorophenol
Bis(2-ehthylhexyl)adipate

7.0 CALIBRATION PROCEDURES

7.1 The probe or microtip should not be immersed in the liquid or come in contact with the work surface when tuning.

7.2 When operating with liquids at extreme temperature, immerse the probe in the liquid for a few minutes, remove from the liquid, then tune.

7.3 Secure the clamp to the converter housing only. Clamping the horn or the front driver will prevent ultrasonic sound waves from traveling through the horn.

7.4 Turn OUTPUT CONTROL knob counter-clockwise to zero.

7.5 Press POWER SWITCH to ON (up) position. The switch will illuminate.

7.6 When the prompt [for tuning procedure refer to manual] appears, press TUNE key. Screen will read: [TUNING --- PROBE ACTIVE].

7.7 Turn the Output Control Knob towards setting 3.

7.7.1 Note the position of the Bar Graph on the LCD Display Screen. Do **NOT** exceed 70%.

7.7.2 Rotate the Tuning Knob clockwise or counter-clockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.

7.8 Turn Output Control Knob towards setting 6.

7.8.1 Again, note the position of the Bar Graph and do not exceed 70%.

7.8.2 Rotate the tuning knob until you obtain a meter reading of 20% or below.

7.9 Repeat step 7.8 on power setting 10. Minimize the reading one last time to 20% or less.

7.10 Press the TUNE key to display prompt for programmed or continuous operation.

NOTE: If unit will not be used immediately, turn power switch to off.

8.0 SAMPLE PREPARATION

8.1 The samples are extracted by Method 3550C from SW-846.

8.2 Sample Homogenization

8.2.1 Remove the sample bottle contents and place in tray lined with aluminum foil.

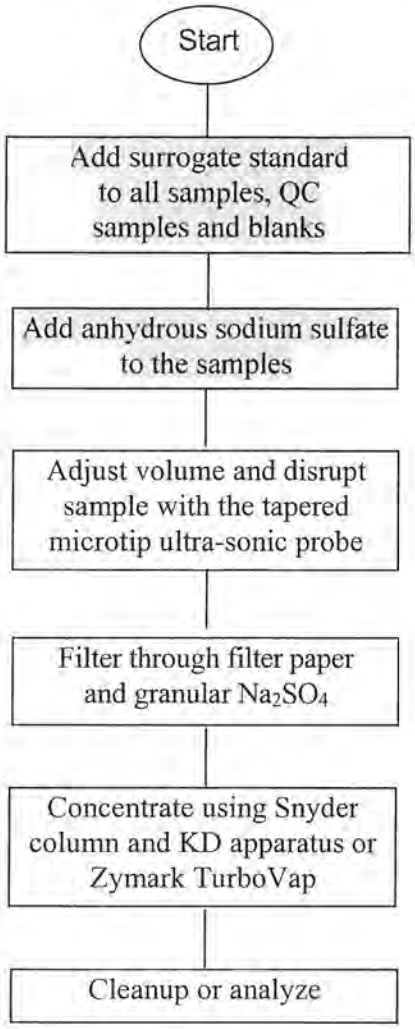
8.2.2 Mix sample with an inert rod or scoop and break up lump. Remove all large stones, sticks, leaves, etc. Do not overmix the sample.

8.2.3 Obtain representative sample either by random removal of 3-10 portions of the sample from the pan or by using a "standard" scoop designed to retrieve a linear cross-section of the pan contents.

8.2.4 The analyst will not attempt to target and exact weight once a method specified minimum amount is weighed.

8.2.5 The remaining sample will be returned to the sample container.

9.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



10.0 ANALYTICAL PROCEDURES

10.1 Rinse all extraction glassware with methylene chloride.

10.2 Weigh out 30 grams [±15%, do not target weight – see section 8.2] of sample into a 250 mL beaker and record to the nearest 0.1 g. Prepare a blank and laboratory control sample (LCS) by weighing up 30 grams of purified lab sand. The MS/MSD are prepared by weighing up one of the samples two (2) additional times. Add 500 ul of BNA surrogate to all samples, spikes and blanks. Add 500 ul of the BNA spike solution to the samples designated as matrix spikes. Add powdered sodium sulfate to the sample and mix until it is free flowing. Immediately add methylene chloride/acetone mix to the 100 mL mark on the beaker. Record surrogate and spike additions.

10.3 To sonicate, place the bottom of the tip of the horn below the surface of the liquid but above the sediment layer.

10.4 Sonicate for three (3) minutes with a 1 second pulse making sure the liquid is mixing thoroughly with the sample.

10.5 Decant the solvent through a funnel lined with filter paper and granular sodium sulfate into a 250 mL Erlenmeyer flask.

10.6 Repeat the extraction (10.3 - 10.5) two (2) more times adding methylene chloride/acetone mix to the 100 mL mark each time and combine the extracts to a 250 mL Erlenmeyer flask. Clean the horn between sonications with methylene chloride.

10.7 Concentration of extracts using the Kuderna-Danish apparatus.

10.7.1 Attach a 15 mL concentrator tube to a 500 mL Kuderna-Danish (K-D) flask with a spring and a clip.

10.7.2 Transfer the extract to the K-D flask and rinse the Erlenmeyer flask with 10 - 20 mL of methylene chloride and add it to the K-D flask.

10.7.3 Add a boiling chip to the K-D flask and attach the Snyder column to the top. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80° - 90° C) so that

the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. When the apparent volume reaches 1 mL, remove the K-D apparatus from the water bath and allow it to cool for at least 10 minutes.

10.7.4 Remove the concentrator tube from the K-D flask and place it on the N-evap.

10.7.5 To use the N-evap, turn on the heater to 35° C. Turn on the gas and lower the needle so that it is about a centimeter above the liquid. When the apparent volume of liquid reaches 0.5 mL, remove the concentrator tube from the N-evap and allow to cool. Adjust the final volume to 1 mL with methylene chloride. Record final volume.

10.7.6 Transfer the extract to an autosampler vial and seal, mark sample level and label it with sample ID, extraction date and test.

10.8 Concentration of extracts using the TurboVap II.

10.8.1 Rinse the concentration (200 mL) tubes with methylene chloride.

10.8.2 Transfer the extract into the tube, rinse the Erlenmeyer and add it also to the tube.

10.8.3 Place the tube into the TurboVap II, which is set at 45° C, and close the cover.

10.8.4 Press the sensor button on the Turbo Vap and start the concentration process for each cell by pushing the start/stop button for each cell.

10.8.5 Adjust the gas flow to get a nice helical flow that does not spit out the sample.

10.8.6 When the endpoint is reached, the light next to its start/stop button will blink and the beeper sounds briefly every thirty seconds.

10.8.7 Adjust the final volume to 1 mL with methylene chloride and transfer the extract to an autosampler vial marked BNA with the sample ID, extraction date, test, and fraction also on the vial. Seal the vial.

11.0 QUALITY CONTROL (QC) REQUIREMENTS

11.1 A reagent blank, laboratory control sample, matrix spike and matrix spike duplicate are extracted with each batch. The only exception is if the client does not send enough sample to perform a matrix spike and matrix spike duplicate. Then a

reagent blank, laboratory control sample and a laboratory control sample duplicate may be performed at the client's request.

11.2 All batch quality control samples are subjected to exactly the same extraction and clean-up procedures as those used on actual samples in the extraction batch.

11.3 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).

12.0 WASTE MANAGEMENT AND POLLUTION CONTROL

12.1 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.

12.2 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.

12.3 The following are waste streams in the sample preparation area.

12.3.1 Halogenated solvents: Methylene Chloride

12.3.2 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

13.0 REFERENCES

1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition Method 3500C, 3550B, 3550C, Updates I, II, IIA and III.

2. Microbac SOP 33, "Laboratory Waste Management"

3. Microbac SOP LQAP "Laboratory Quality Assurance Plan"

4. Microbac SOP GP-CAPA, "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

5. Microbac SOP GP-RCA, "Root Cause Analysis"

Microbac

STANDARD OPERATING PROCEDURE
ORGANIC ANALYTES
METHOD 8270

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TABLE OF CONTENTS

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.

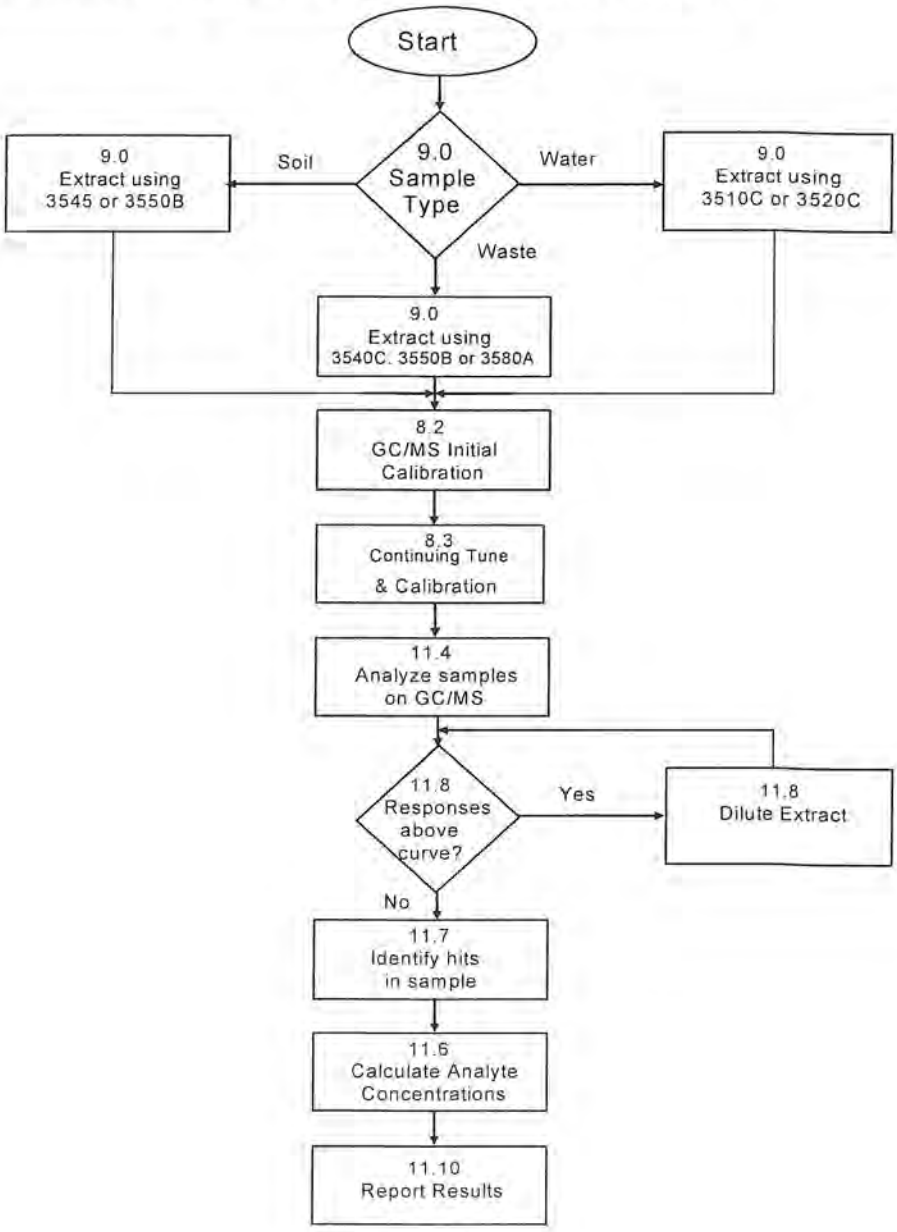
9.0 SAMPLE PREPARATION

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):

$$\frac{ug}{L} = \frac{(A_x)(I_s)(Vf)}{(A_{is})(\overline{RRF})(Vi)}(D)(1000)$$

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:

$$\frac{ug}{Kg} = \frac{(A_x)(I_s)(Vf)}{(A_{is})(\overline{RRF})(Wi)}(D)(1000)$$

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:

$$C_{ii} = \frac{(C_x)(100)}{(\%solids)}$$

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

$$RSD = \left(\frac{s}{x}\right)100$$

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:

$$\%R = \left(\frac{C_s}{C_T}\right)100$$

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	07/21/08 08:30
2	12M22746	WG277487-02 50PPM MEGAMIX STD	1	1	STD27564	07/21/08 08:52
3	12M22747	WG277556-01 FBLK 7/17	17	1		07/21/08 09:25
4	12M22748	WG277561-01 SPLP BLANK 7/17	16	1		07/21/08 09:52
5	12M22749	WG277469-01 BLANK V10009 PG25	17	1		07/21/08 10:33
6	12M22750	WG277469-02 LCS V10009 PG25	17	1		07/21/08 11:07
7	12M22751	WG277469-03 LCSDUP V10009 PG25	17	1		07/21/08 11:41
8	12M22752	L08070396-02 SOIL	7	1	SOIL	07/21/08 12:15
9	12M22753	L08070396-04 SOIL	7	1	SOIL	07/21/08 12:50
10	12M22754	L08070358-01 TCLP	17	1		07/21/08 13:24
11	12M22755	L08070358-03 TCLP	17	1		07/21/08 13:58
12	12M22756	L08070358-05 TCLP	17	1		07/21/08 14:33
13	12M22757	L08070358-09 TCLP	17	1		07/21/08 15:07
14	12M22758	L08070406-01 TCLP	17	1		07/21/08 15:42
15	12M22759	L08070406-02 TCLP	17	1		07/21/08 16:17
16	12M22760	L08070406-03 TCLP	17	1		07/21/08 16:51
17	12M22761	L08070406-04 TCLP	17	1		07/21/08 17:25
18	12M22762	L08070406-05 TCLP	17	1		07/21/08 17:59
19	12M22763	L08070396-03 SPLP	16	1		07/21/08 18:33
20	12M22764	L08070396-05 SPLP	16	1		07/21/08 19:07
21	12M22765	L08070374-01 10X TCLP	17	10		07/21/08 19:40
22	12M22766	BAKE OUT	17	1		07/21/08 20:14
23	12M22767	BAKE OUT	17	1		07/21/08 20:47

Comments

Seq.	Rerun	Dil.	Reason	Analytes
2				
	88 +-30% but +-40% low and 55 +-40% high			
7				
	PHL is high			
10				
	PHN and FRY are low due to SMI.			
12				
	FFP and PHL are +-5%. OK per very basic matrix			
21	X	2	Analyzed too dilute	

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

Microbac

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 PROCEDURES FOR ANALYSIS OF VOLATILE ORGANIC ANALYTES BY METHODS 8260A AND 8260B SOP MSV01

Revision 14

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Microbac Laboratories
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Marietta, Ohio 45750

Approved By:

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David L. Bumgarner, Quality Assurance Officer — Date 1/14/10

David E. Vandenberg, Managing Director — Date 1/14/10

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
[signature]	1/13/10
Tiffany Bailey	1/13/10
Cassie A. Augenstein	1/13/10
[signature]	1/13/10
Wadi. Dr	1-14-10

TABLE OF CONTENTS

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.

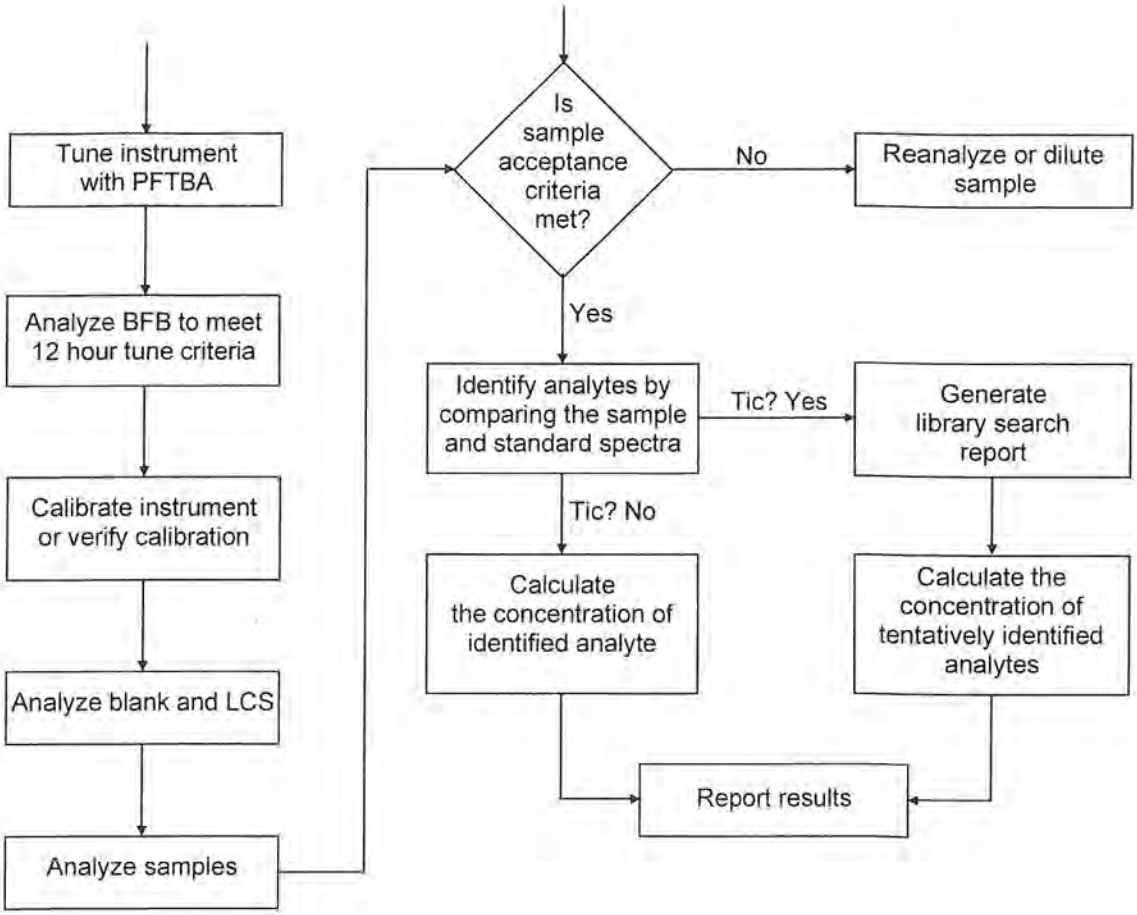
8.13 For analysis of OVAP samples by 8260A and 8260B, n-hexane will be included in the list of calibration check compounds (CCC). Therefore n-hexane will be required to pass CCC acceptance criteria.

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):

$$s = \sqrt{\frac{\sum \left(x - \bar{x}\right)^2}{n-1}}$$

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): $s = \sqrt{\frac{\sum \left(x - \bar{x}\right)^2}{n-1}}$

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:

$$C_x = (C_{ix})\left(\frac{C_x}{C_{istd}}\right)\left(\frac{V_f}{W_i}\right)(DF)(1000)$$

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):

$$\frac{ug}{L} = \frac{(A_x)(I_s)(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

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 ≥ 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			10.12	5		5	5
L08020355-09	A	04/20/08 11:25	06/05/08 09:50	79.08			10.13	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		30	15.7188618	5

Comments: [illegible]

Analyst: [signature]

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.

Microbac

STANDARD OPERATING PROCEDURE
ORGANIC ANALYSIS OF PCB'S
METHOD 8082/8082A

Issue/Implementation Date: 15 January 2010

Last Review Date: 15 January 2010

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TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

This method describes the procedures used to analyze PCBs in a variety of sample matrices. This method uses fused silica columns in order to separate target compounds. Tables 1 and 2 list the compounds that may be determined by this method. This method follows the procedures outlined in SW-846 Update III Method 8082 and Method 8082A Revision 1.

2.0 SAFETY PRECAUTIONS

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 personal 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.

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 Hewlett-Packard 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 will also allow 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 will also allow for the sample to be re-extracted should the need arise.

3.2 Sample collection should be performed according to the outline set forth in SW-846. This will give 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 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 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 of the 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 should 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 HP 5890 GC equipped with dual ECDs and capillary port injectors.

6.2 Restek RTX-CLPesticides Megabore column 30 m x 0.53 mm I.D. and 0.5 um film thickness.

6.3 Restek RTX-CLPesticides 2 Megabore column 30 um x 0.53 mm I.D. and 0.42 um film thickness.

6.4 HP 7673 dual tower autosampler

6.5 HP 3365 Chemstation analytical software.

6.6 IBM compatible PC with a 486 or higher micro-processor, hard drive storage, and QIC tape driven back-up capabilities.

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 PCB Stock Standards - Each of the PCB stock solutions are prepared in an identical manner. Each PCB high level solutions are purchased from Supelco at a concentration of 1000 ppm in iso-octane. From these high level ampules exactly 1.0 mL is transferred to a 100 mL volumetric flask containing approximately 80 mL of pesticide grade hexane. The Supelco standard numbers are:

Ar1016	4-8097	or equivalent
Ar1221	4-8098	or equivalent

Ar1232	4-4805	or equivalent
Ar1242	4-4806	or equivalent
Ar1248	4-4807	or equivalent
Ar1254	4-4808	or equivalent
Ar1260	4-4809	or equivalent

AR1016 and AR1260 are combined and analyzed together. Surrogate compounds are added by transferring 0.25 mL of stock surrogate standard to the volumetric flask. The flask is filled to the mark with pesticide grade hexane and inverted three times to ensure proper mixing. This yields standards having PCB concentrations at 10.0 ppm and surrogate concentrations at 0.50 ppm. The intermediate standard is now ready for use to prepare daily calibration standards. Secondary check standards stocks are prepared in an identical manner except that the high level source purchased from Accustandard or an equivalent source. All PCB standards are prepared individually and all contain the method surrogates. All PCB standards are stored at -10° C and disposed of 6 months after the original date of preparation.

7.3 Surrogate Stock Standard – A surrogate stock standard of 2,4,5,6-tetrachloro-m-xylene and decachlorobiphenyl is purchased from Accustandard, standard # CLP-032-R or equivalent. This ampule contains 1.0 mL of 200 ppm of each of the two surrogate compounds.

7.4 Calibration Standards:

PCB's - The PCB calibration standards are prepared from the working stock at the following levels:

Std # 1 - 50 ppb
Std # 2 - 100 ppb
Std # 3 - 250 ppb
Std # 4 - 500 ppb
Std # 5 - 1000 ppb
Std # 6 - 2000 ppb (optional)

These are made up by dilution on the working stock standard using the following scheme:

#1 - 20 x on the 1000 ppb #5 (950 µL hexane: 50 µL #5)
#2 - 10 x on the 1000 ppb #5 (900 µL hexane: 100 µL #5)
#3 - 4 x on the 1000 ppb #5 (750 µL hexane: 250 µL stock)
#4 - 20 x on the stock solution (10.0 ppm) (950 µL hexane: 50 µL stock)
#5 - 10 x on the stock solution (10.0 ppm) (900 µL hexane: 100 µL stock)
#6 - 5 x on the stock solution (10.0 ppm) (800 µL hexane: 200 µL stock)

The secondary source standards are prepared at Level #3 of the initial calibration curve.

7.5 DDT analogue standard: A standard containing DDT, DDD and DDE. The pesticide calibration standard from GCS09 section 7.2 may be used for this purpose.

8.0 CALIBRATION PROCEDURE

8.1 Recommended Gas Chromatograph Conditions

Columns -	See Sections 6.2 – 6.6.
Temp Prog -	160° C for 3.0 min
	20° C/min to 320° C for 3.0 min
Carrier Gas -	Helium at 7 mL/min
Ionizing Gas -	Ar/Me @ 50 mL/min
ECD Temp -	350° C
Injector Temp -	225° C
Injection volume -	0.5 uL

8.2 Initial Calibration

8.2.1 Run the following series of calibration standards after a hexane blank used to assess system cleanliness:

50 ppb Aroclor 1016/1260
100 ppb Aroclor 1016/1260
250 ppb Aroclor 1016/1260
500 ppb Aroclor 1016/1260

1000 ppb Aroclor 1016/1260
2000 ppb Aroclor 1016/1260 (optional)

250 ppb Aroclor 1016/1260 (second source)
500 ppb Aroclor 1221
500 ppb Aroclor 1232
500 ppb Aroclor 1242
500 ppb Aroclor 1248
500 ppb Aroclor 1254

500 ppb Aroclor 1221 (second source)

500 ppb Aroclor 1232 (second source)
500 ppb Aroclor 1242 (second source)
500 ppb Aroclor 1248 (second source)
500 ppb Aroclor 1254 (second source)

This sequence must be run whenever any major changes are made to the instrument. This curve must have a %RSD [standard deviation/average calibration factor (Cf)] of less than or equal to 20 % using the calculations in section 12.1 and 12.3. If the % RSD is greater than 20 the analyst quantitates samples by plotting against the 5 point calibration using linear regression (using the calculation in section 12.8), provided the coefficient of correlation is 0.995 or greater. If the coefficient of correlation is less than 0.995, then a new curve must be run.

8.2.2 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 includes many of the peaks represented in the other five Aroclor mixtures. Such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine single point calibration factors for each Aroclor, assuming that the Aroclor 1016/1260 mixture demonstrates the linearity of the detector.

8.2.3 The DOD QSM requires multi-point calibrations for other Aroclors, if detected. These calibration curves must meet the requirements of 8.2.2 on the primary and secondary columns. Other project QAPPs may also have this requirement.

8.3 Calibration Verification

8.3.1 Initial calibration verification (ICV): The second source standards must have a % difference or % drift of less than 20 using the formula in section 12.2. Inability to achieve %D of less than 20 when running an ICV standard indicates a problem and will necessitate instrument maintenance.

8.3.2 Continuing calibration verification (CCV): The CCV standards must have a % difference or % drift of less than 20 using the formula in section 12.2. Inability to achieve %D of less than 20 when running a set of CCV standards indicates a problem and will necessitate instrument maintenance.

8.3.3 Certain projects may require the use of Method 8082; if this is the case, the ICV and CCV standards must have a % D ≤15.

8.3.4 The sequence of the sample run then proceeds as follows:

10 samples
Aroclor 1016/1260 CCV @ 500 ppb
10 samples

Aroclor 1016/1260 CCV @ 250 ppb
10 samples
Aroclor 1016/1260 CCV @ 500 ppb – etc.

If a CCV fails to yield Cf's with % difference of ± 15 % then a second CCV may be analyzed from the same source as the first CCV. Upon failure of two consecutive CCVs a new curve must be run and the samples, bracketed within the bad CCV, must be re-run. 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. If a CCV fails to yield %D with a % difference of ±20% (±15% for 8082), then a second CCV may be analyzed, etc.

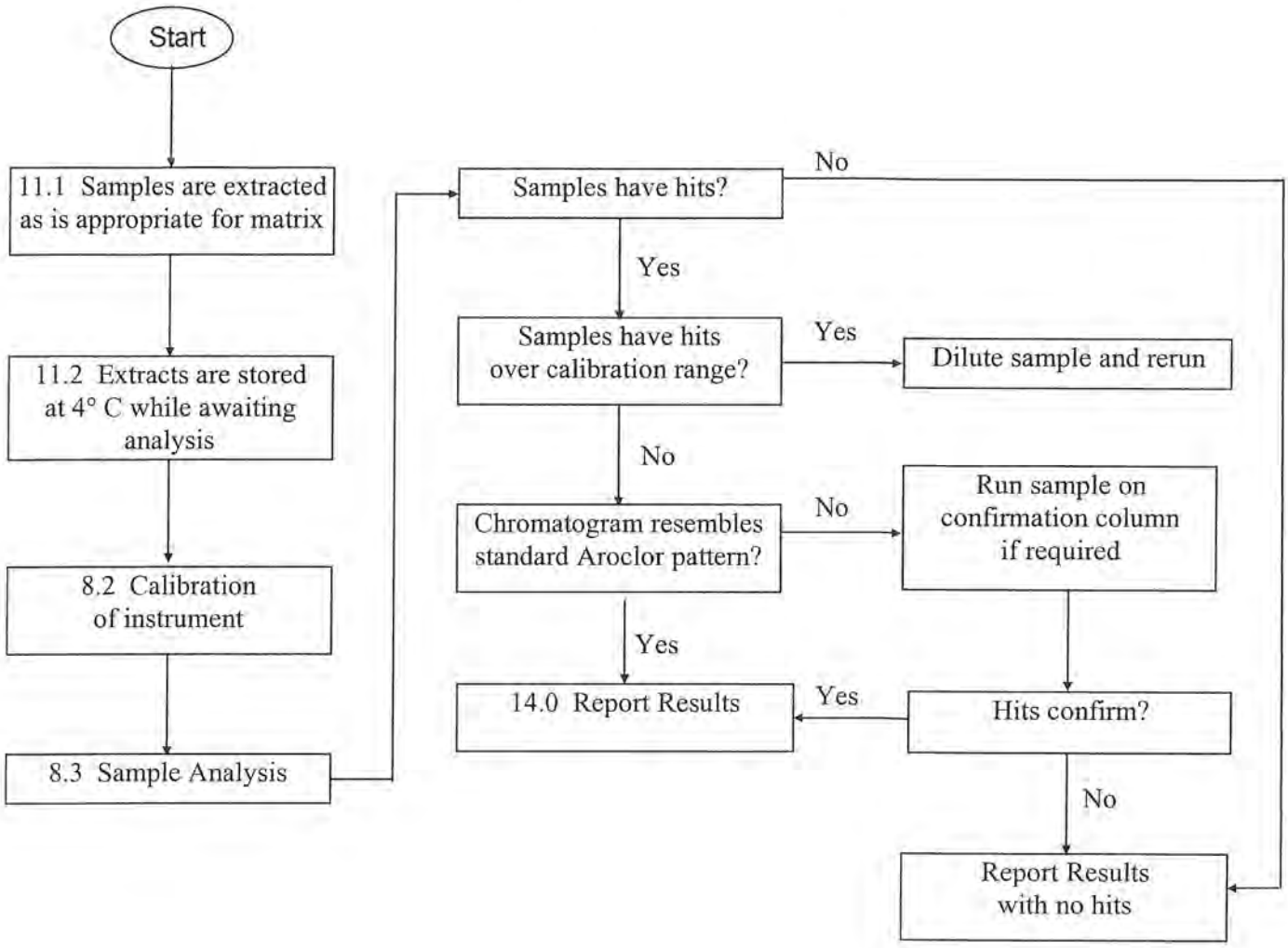
9.0 SAMPLE PREPARATIONS

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.

10.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



11.0 ANALYTICAL PROCEDURES

11.1 All samples are extracted by the appropriate technique.

11.2 The instrument(s) are calibrated according to section 8.0 of this document.

11.3 A compound is considered tentatively identified if a peak is found with the calculated daily retention time windows. The retention time windows are calculated by analyzing a standard over a 72 hour period and calculating the standard deviation of each of the target compounds' retention time and multiplying each of these standard deviations by 3. This ± value is the retention time window for that compound. The mean absolute retention time used for defining windows is taken from the continuing calibration standards. Multiresponse compounds such as Aroclors will have retention time windows calculated. However, the identification of these compounds is based primarily on pattern recognition using 3 - 5 peaks of the multiresponse compound.

11.4 An aroclor is identified by comparison of the chromatographic pattern with the pattern of a standard of the suspected aroclor.

11.5 The aroclor component peaks must fall with the calculated retention time window. If coelution or interfering components prohibits accurate assignment of the aroclor, another Aroclor component may be used provided the calibration criteria are re-assessed using the new component.

11.6 Since PCBs are identified primarily by pattern recognition, a second column confirmation is not necessary unless required by a specific project (the DOD QSM is an example). Second column analysis may be used as an aid in the identification of difficult (e.g. weathered) PCBs.

11.7 Quantitation of Aroclors is performed by selecting at least three (3) major peaks and treating them as single components. Each peak must pass all calibration criteria listed in sections 8.2.2, 8.3.1 and 8.3.2 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 Aroclor.

11.8 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.

11.9 The raw data is processed using the chem station software and the data is uploaded into the LIMS.

11.10 The data is reviewed two times by the data review team; once for completeness and a second time for data validation.

11.10.1 In order to ensure that DDT, DDE and DDD do not co-elute with any of the Aroclor peaks chosen for quantitation, a DDT analogue standard is analyzed after each column change or whenever retention time windows are established. If any of the DDT analogue peaks elute at the same retention time as an Aroclor quantitation peak, an alternate peak should be chosen.

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

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

$$\frac{\sum XY - \sum X \sum Y / n}{\sqrt{\left(\sum X^2 - \left(\sum X\right)^2 / n\right)\left(\sum Y^2 - \left(\sum Y\right)^2 / n\right)}}$$

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

12.7 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:

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:

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 or quadratic 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 or quadratic 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. The MS/MSD samples may be waived if insufficient sample is available. 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. If a second column is used for confirmation, the confirmation column must meet all quality control acceptance criteria.

The procedures, required frequency, acceptance criteria, and the required corrective action measures are outlined in Table 3. Special requirements and corrective action for Ohio VAP are included in the sections that follow.

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. A standard containing a mixture of Aroclor 1016 and Aroclor 1260 is used as the spike in the LCS, MS, and MSD. The LCS, MS and MSD are spiked so as to yield a concentration that falls within the mid-point of the curve. At least four (4) LCS spikes must be performed per year for the following Aroclors:1221, 1232,1242,1248 and 1254. **Note:** The extraction lab supervisor must obtain approval from the project chemist before changing the LCS spike. It may be necessary to have more than one LCS per batch to meet all project and method requirements.

13.2 Section 8 describes the acceptable criteria for the initial calibration verification (ICV) and continuing calibration verification (CCV) standards.

13.3 The Laboratory Control Sample (LCS) is spiked by the extraction laboratory with Aroclor 1016 and 1260 at concentrations of 2.5 ug/L for waters and 83.3 ug/kg for soils. 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 3 are used in the absence of a project QAPP, or program specified control limits. If the LCS recovery for 1016/1260 is 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. 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. Ohio VAP requires the method blank to not be greater than the RL. Samples must be re-extracted and re-analyzed if the method blank does not meet this criteria.

13.4 The method blank cannot contain amounts of any target analytes, which are over one-half of the reporting limit (RL). If any target analytes are found in the method blank with concentrations higher than one half of 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.

13.5 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 Tables 1 and 2. If any individual sample has both surrogates outside the given limits, then the sample must be re-extracted. **Note**: Ohio VAP requires that all surrogates be within the acceptable limits for all batch QC samples (method blank, LCS< LCSD) and for all field samples, except in cases of obvious matrix interference.

13.6 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 PCB. To calculate the window the analyst is required to perform statistical operations on chromatograms from 3 non-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 PCB.

When required by a specific project, aroclors are confirmed on a second column of different polarity. If the results between the primary and secondary column vary by more that 40%, the higher of the results is reported unless sample matrix effects (e.g. interfering peaks) are present.

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 3 does not meet acceptance criteria. When nonconforming data occurs the employee initiates a Nonconformance Report (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 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, 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. Leak tests are performed on the ECD's semi-annually. The ECD's are changed out when the calibration of the instruments becomes increasingly difficult.

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. 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.

2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, December 1996. Update III Method 8082.

3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8082A Revision 1, February 2007.

4. Microbac SOP LQAP, "Laboratory Quality Assurance Plan"

5. Microbac SOP45, "Standard Operating Procedures for Method Validation Procedures"

6. Microbac SOP GP-CAPA, "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

7. Microbac SOP GP-RCA, "Root Cause Analysis"

8. Microbac SOP33, "Laboratory Waste Management"

TABLE 1
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR PCB ORGANIC ANALYSES OF GROUNDWATER

PARAMETER	CAS #	EPA SW-846 METHOD*	ACCURACY (% RECOVERY)*	PRECISION (% RPD)*	OBSERVED MDL WATER (ug/L)	REPORTING LIMITS WATER (ug/L)
PCB 1016	12674-44-2	8082/8082A	40-140	0-30	0.25	0.50
PCB 1221	11104-28-2	8082/8082A	41-136	0-30	0.25	0.50
PCB 1232	11141-16-5	8082/8082A	41-136	0-30	0.25	0.50
PCB 1242	53469-21-9	8082/8082A	39-150	0-30	0.25	0.50
PCB-1248	12672-29-6	8082/8082A	41-136	0-30	0.25	0.50
PCB-1254	11097-69-1	8082/8082A	29-141	0-30	0.25	0.50
PCB-1260	11096-82-5	8082/8082A	40-140	0-30	0.25	0.50
Surrogates						
2,4,5,6-Tetrachloro-m-xylene	NA	8082/8082A	30-132	NA	NA	NA
Decachlorobiphenyl	NA	8082/8082A	40-133	NA	NA	NA

* 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 PCB ORGANIC ANALYSES OF SOLID WASTE

PARAMETER	CAS #	EPA SW-846 METHOD*	ACCURACY (% RECOVERY)*	PRECISION (% RPD)*	OBSERVED MDL SOIL (ug/Kg)	REPORTING LIMITS SOIL (ug/Kg)
PCB 1016	12674-11-2	8082/8082A	40-140	0-50	8.25	17
PCB 1221	11104-28-2	8082/8082A	45-136	0-50	8.25	17
PCB 1232	11141-16-5	8082/8082A	45-136	0-50	8.25	17
PCB 1242	53469-21-9	8082/8082A	43-150	0-50	8.25	17
PCB-1248	12672-29-6	8082/8082A	44-136	0-50	8.25	17
PCB-1254	11097-69-1	8082/8082A	41-141	0-50	8.25	17
PCB-1260	11096-82-5	8082/8082A	60-130	0-50	8.25	17
Surrogates						
2,4,5,6-Tetrachloro-m-xylene	NA	8082/8082A	29-133	NA	NA	NA
Decachlorobiphenyl	NA	8082/8082A	60-125	NA	NA	NA

* Values are statistically derived from laboratory control samples and are evaluated annually. Actual control limits may vary.

TABLE 2 (continued)
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR PCB ORGANIC ANALYSES OF SOLID WASTE

Compound Name	Accuracy (% Recovery)	Wipe Reporting Limits ug/wipe
Ar1016	89-158	0.5
Ar1221	NA	0.5
Ar1232	NA	0.5
Ar1242	NA	0.5
Ar1248	NA	0.5
Ar1254	NA	0.5
Ar1260	92-150	0.5

Detection limits are determined by statistical analysis of 7 replicates of spiked (reagent) waters. MDL = 3.14 (Standard Deviation) of the 7 replicates.

TABLE 3
Quality Control Criteria
PCBs
Method 8082/8082A

Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	Initially and upon failure of Two consecutive CCV's	Linear – mean RSD for all Analytes ≤ 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 8082)	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 8082)	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	< ½ of the RL 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.
(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.

Figure 1

Run Log ID: 26554

Microbac Laboratories Inc.
Instrument Run Log

Instrument: HP9 Dataset: 021609
Analyst1: ECL Analyst2: NA
Method: 8082 SOP: GCS10 Rev: 10

Maintenance Log ID:

Column 1 ID: Column 2 ID: RTX-CLP2
Workgroups: WG294648, WG294933
Internal STD: NA Surrogate STD: STD30125 Calibration STD:
Comments:

Seq	File ID	Sample Information	Mat	Dil	Reference	Date/Time
1	9GR50623.R	WG294996-01 1660 CCV 500 PPB	7	1	STD30336	02/16/09 12:50
2	9GR50624.R	WG294648-01 BLANK	7	1	SOIL	02/16/09 13:42
3	9GR50625.R	WG294648-02 LCS	7	1	SOIL	02/16/09 13:59
4	9GR50626.R	WG294648-03 LCS DUP	7	1	SOIL	02/16/09 14:17
5	9GR50627.R	L09020243-01	7	1	SOIL	02/16/09 14:34
6	9GR50628.R	L09020218-01	7	1	SOIL	02/16/09 14:52
7	9GR50629.R	L09020218-02	7	1	SOIL	02/16/09 15:10
8	9GR50630.R	L09020218-03	7	1	SOIL	02/16/09 15:27
9	9GR50631.R	L09020218-04	7	1	SOIL	02/16/09 15:45
10	9GR50632.R	WG294996-02 1660 CCV 250 PPB	7	1	STD30336	02/16/09 16:02
11	9GR50633.R	WG294933-02 BLANK	7	1	SOIL	02/16/09 16:20
12	9GR50634.R	WG294933-03 LCS	7	1	SOIL	02/16/09 16:38
13	9GR50635.R	L09020281-01	7	1	SOIL	02/16/09 16:55
14	9GR50636.R	L09020281-02 REP	7	1	SOIL	02/16/09 17:13
15	9GR50637.R	L09020281-04 MS	7	1	SOIL	02/16/09 17:30
16	9GR50638.R	L09020281-05 MSD	7	1	SOIL	02/16/09 17:48
17	9GR50639.R	L09020281-07	7	1	SOIL	02/16/09 18:05
18	9GR50640.R	L09020281-08	7	1	SOIL	02/16/09 18:23
19	9GR50641.R	L09020281-11	7	1	SOIL	02/16/09 18:41
20	9GR50642.R	WG294996-03 1660 CCV 500 PPB	7	1	STD30336	02/16/09 18:58

Comments

Seq	Rerun	Dil	Reason	Analytes

Figure 2

Checklist ID: 35884

Microbac Laboratories Inc.

Data Checklist

Date: 09-FEB-2009
Analyst: ECL
Analyst: NA
Method: 8082
Instrument: HP9
Curve Workgroup: NA
Runlog ID: 26451
Analytical Workgroups: L09010619, L09020081, L09020109

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)	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	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	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: 10-FEB-2009

Secondary Reviewer: 11-FEB-2009

CHECKLIST1 - Modified 03/05/2008
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Appendix I
South Carolina Requirements

The LCS recovery limits for samples analyzed for the state of South Carolina are 70-130%.

Microbac

STANDARD OPERATING PROCEDURE SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTIONS FOR PESTICIDES AND PCB'S SW846 Method 3510C, 3500C, and CFR Method 608

Issue/Implementation Date: 15 April 2010

Last Review Date: 15 April 2010

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved By:

Chad E. Barnes, Extraction Supervisor — Date 4/7/10

David L. Bumgarner, Technical Director/QAO — Date 4/6/10

David E. Vandenberg, Managing Director — Date 4/8/10

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

This method describes a separatory funnel procedure for extracting and isolating pesticides, PCB's as Aroclors, and PCB congeners from aqueous samples. The method also describes the concentration and cleanup procedures involved in preparing the extract for analysis by Methods 608, 8081, and 8082. This method follows the procedures outlined in SW-846 Method 3510C, 3500C and 40 CFR Chapter 1 (7-1-95 Edition).

NOTE: The extraction of low level pesticides and/or PCB's will follow this SOP with the following exceptions:

1. The spikes and surrogates will be ten (10) times lower in concentration.
2. The entire extract (1.0 mL) will go through florisil clean-up and no acid clean-up on PCB's.
3. The final volume will be 1.0 mL instead of 10.0 mL.

2.0 SAFETY PRECAUTIONS

2.1 The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined. However, each chemical compound should be treated as a potential health hazard.

2.2 Proper lab coat, gloves, and safety glasses must be worn while performing this extraction.

2.3 **CAUTION:** If using a regular 2 L separatory funnel instead of a vent-separatory, initial venting of methylene chloride should be done immediately after the separatory funnel has been sealed because methylene chloride creates excessive pressure and should be done periodically while shaking the funnel.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Samples should be collected in 1 L glass containers with teflon-lined caps.

3.2 Sample volume needed for analysis is 1 L but if sample volume is less than 1 L, record the actual volume of sample and then dilute sample to 1 L with deionized water to perform extraction. If sample is significantly less than 1 L, notify the TSR.

3.3 Sample preservation should be at 4° C, and the sample maximum holding time from date of collection is seven (7) days.

4.0 INTERFERENCES AND CORRECTIVE MEASURES

4.1 Interferences may be caused by contaminants in solvents, reagents, glassware and other sample preparation.

4.2 Emulsions after a shake may occur in which the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation or other physical methods. If the emulsion cannot be broken (recovery of less than 80 % of the methylene chloride) transfer sample, solvent, and emulsion into extraction chamber of a continuous extractor and proceed with a continuous extraction method.

4.3 Other interferences that may be encountered are discussed in Method 3500C SW-846.

5.0 EQUIPMENT AND SUPPLIES

5.1 Major Instrumentation

5.1.1 Zymark TurboVap II concentration workstation

5.1.2 Glas-Col 3D Floor Shaker or equivalent

5.1.3 Nitrogen Evaporator: Meyer N-Evap Analytical Evaporator or equivalent

5.1.4 Solid Phase Extraction (SPE) Manifold

5.2 Apparatus or equipment

5.2.1 Kuderna-Danish (K-D) apparatus:

5.2.1.1 Concentrator tube: 15 mL graduated (Supelco 6-4684M or equivalent)

5.2.1.2 Evaporation Flask: 50 mL attached to a concentrator tube with a clip or spring

5.2.1.3 Snyder column: Three ball macro

5.2.2 Water bath: Heated, with concentric ring cover, capable of temperature control (± 5° C). The bath should be used in a hood.

5.3 Glassware

5.3.1 2 L separatory funnel with teflon stopcock or an automatic 2 L venting separatory funnel with teflon stopcock.

5.3.2 Graduated cylinder: 1 L

5.3.3 250 mL ground glass erlenmeyer or flask

5.3.4 Zymark 200 mL concentrator tubes with 1.0 mL endpoint

5.3.5 Calibrated 40 mL vial

5.4 Other Supplies

5.4.1 Syringes - various sizes

5.4.2 pH paper: Wide range (0-13).

5.4.3 Glass wool.

5.4.4 Stainless steel funnel or glass funnel

5.4.5 Autosampler vials: 2 mL capacity

5.4.6 Boiling chips: 10/40 mesh

5.4.7 Florisil column: 1000 mg (J.T. Baker 7213-07 or equivalent)

5.5 Glassware is cleaned by following Microbac SOP 37.

6.0 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.

6.1 Deionized water.

6.2 Sodium hydroxide solution 10 N: Dissolve forty (40) grams NaOH in deionized water and dilute to 100 mL.

6.3 Sodium sulfate: Granular, anhydrous.

6.4 Methylene chloride: Pesticide grade or equivalent.

6.5 Hexane: Pesticide grade or equivalent.

6.6 Sulfuric acid solution (1:1): Slowly add 50 mL of sulfuric acid to 50 mL of deionized water.

6.7 Ethyl ether: Pesticide grade or equivalent.

6.8 Pesticide surrogate (Supelco 4-8460) mix: Methanol solution containing:

Decachlorobiphenyl	1.0 ug/mL
2,4,5,6-tetrachloro-m-xylene	1.0 ug/mL

6.9 PCB Spike - Aroclor 1660 (Mix of Aroclor 1016 and 1260) 25 ug/mL

NOTE: Other Aroclors such as 1221, 1232, 1242, 1248 and 1254 may be used.

6.10 Pesticide spike mix: Methanol solution containing:

Heptachlor	2.0 ug/mL
Aldrin	2.0 ug/mL
Dieldrin	2.0 ug/mL
Endrin	2.0 ug/mL
4,4'-DDT	2.0 ug/mL
4,4'-DDD	2.0 ug/mL
4,4'-DDE	2.0 ug/mL
Alpha-BHC	2.0 ug/mL
Alpha-Chlordane	2.0 ug/mL
Beta-BHC	2.0 ug/mL
Gamma-BHC	2.0 ug/mL
Gamma-Chlordane	2.0 ug/mL
Delta-BHC	2.0 ug/mL

Endosulfan I	2.0 ug/mL
Endosulfan II	2.0 ug/mL
Endosulfan sulfate	2.0 ug/mL
Endrin aldehyde	2.0 ug/mL
Heptachlor epoxide	2.0 ug/mL
Endrin ketone	2.0 ug/mL
Methoxychlor	2.0 ug/mL

6.11 Nitric Acid – dilute

6.12 Copper Powder – Remove oxides by treating with dilute nitric acid and rinsing with deionized water to remove all traces of acid, then rinse with acetone to remove H_2O and dry with n-evap to remove acetone.

6.13 PCB congener surrogate: Acetone mix containing only 2,4,5,6-tetrachloro-m-xylene [TMX] at 1.0 ug/mL.

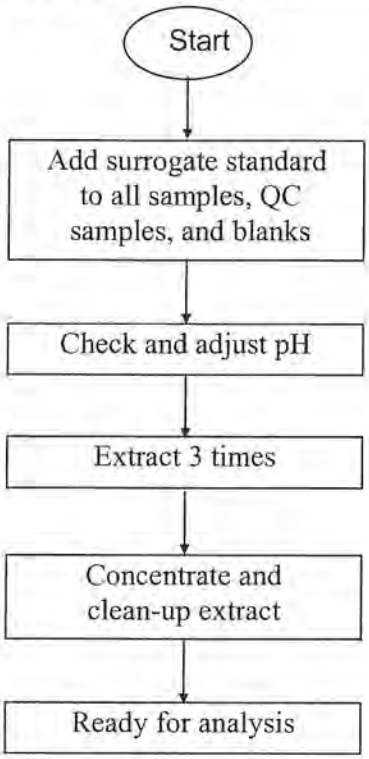
6.14 PCB congener spike: Acetone mix at 2.0 ug/mL containing the following 19 congeners:

2-chlorobiphenyl
2,3-dichlorobiphenyl
2,2',5-trichlorobiphenyl
2,4',5-trichlorobiphenyl
2,2',3,5'-tetrachlorobiphenyl
2,2',5,5'-tetrachlorobiphenyl
2,3',4,4'-tetrachlorobiphenyl
2,2',3,4,5'-pentachlorobiphenyl
2,2',4,5,5'-pentachlorobiphenyl
2,3,3',4',6-pentachlorobiphenyl
2,2',3,4,4',5'-hexachlorobiphenyl
2,2',3,4,5,5'-hexachlorobiphenyl
2,2',3,5,5',6-hexachlorobiphenyl
2,2',4,4',5,5'-hexachlorobiphenyl
2,2',3,3'4,4'-5-heptachlorobiphenyl
2,2',3,4,4',5,5'-heptachlorobiphenyl
2,2',3,4,4',5',6-heptachlorobiphenyl
2,2',3,4',5,5',6-heptachlorobiphenyl
2,2',3,3',4,4',5,5',6-nonachlorobiphenyl

7.0 SAMPLE PREPARATION

Extraction Method 3510C and 3500C from SW-846

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 ANALYTICAL PROCEDURES

9.1 Rinse all extraction glassware with methylene chloride.

9.2 Mark the water meniscus on the side of the sample bottle for later determination of sample volume, and pour entire sample into a 2 L separatory funnel. If the sample volume is significantly less than 1 L, record initial sample volume in logbook and dilute the sample volume to 1 L using deionized water. Add 200 uL of the pesticide surrogate to all samples spikes and blanks. Add 250 uL of the pesticide spike solution to the LCS (Laboratory Control Sample) and to samples designated as matrix spikes. (Add 100 uL of the PCB spike solution to the LCS (Laboratory Control Sample) and to samples designated as matrix spikes for PCB only analysis). Record sample volume and all surrogate and spike additions. For PCB congener analysis add 200 uL of the congener surrogate (see section 6.13) to all samples, spikes and blanks. Add 250 uL of the PCB congener spike (see section 6.14) to the LCS and/or LCS2, MS and MSD's.

[**NOTE:** NELAC standards and the DOD QSM require the laboratory to include all of the PCB Aroclors in the LCS over a two (2) year period. It will be necessary to include multiple LCS spikes in some batches in order to meet the method, NELAP, and project requirements, i.e. one (1) LCS spiked with 1660, and another LCS spiked with another Aroclor.]

9.3 Check the pH of the sample with wide-range pH paper and, if necessary, adjust the pH to between five (5) and nine (9) using 10N sodium hydroxide solution or sulfuric acid (1:1).

9.4 Add 60 mL of methylene chloride to the sample bottle and transfer this rinse solvent to the separatory funnel.

9.5 Seal the funnel with a teflon stopper and shake the separatory funnel vigorously for two (2) minutes with periodic venting to release excess pressure. **NOTE:** Methylene chloride creates excessive pressure rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. If using the automatic venting separatory funnel, screw teflon cap on and turn separatory funnel upside down. Place the stopcock in the venting position and place funnel on 3-D shaker. Once funnel is secured in place, shake for 2 minutes.

9.6 Allow the layers to separate for at least ten (10) minutes. The methylene chloride layer will be on the bottom. Drain the methylene chloride layer through a funnel

plugged with glass wool and filled with about 10-20 g of granular sodium sulfate into a 250-mL Erlenmeyer flask.

9.7 Repeat the extraction (9.5-9.6) two (2) more times using 60 mL of fresh methylene chloride each time and combine the extracts in a 250-mL Erlenmeyer flask.

9.8 Concentration of extracts using the Kuderna-Danish apparatus.

9.8.1 Attach a 15-mL concentrator tube to a 500-mL Kuderna-Danish (K-D) flask with a spring and a clip.

9.8.2 Transfer the extract to the K-D flask and rinse the Erlenmeyer flask with 10-20 mL of methylene chloride and add it to the K-D flask.

9.8.3 Add a boiling chip to the K-D flask and attach the Snyder column to the top. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80° - 90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. When the apparent volume reaches 1 mL, remove the K-D apparatus from the water bath and allow it to cool for at least ten (10) minutes.

9.8.4 Add 50 mL of hexane through the Snyder column into the K-D flask and concentrate the extract as described in 9.8.3, raising the temperature of the water bath to 90° - 95° C.

9.8.5 Adjust the final volume to 10 mL by either adding hexane or concentrating it with the nitrogen evaporator (N-evap). To use the N-evap, turn on the heater to 35° C. Remove the concentrator tube from the K-D apparatus and place it in the N-evap. Turn on the gas tank and lower the needle so that it is about a centimeter above the liquid. Remove the concentrator tube from the N-evap when the apparent volume is about 9 mL. Rinse the sides of the tube with hexane and adjust the volume to 10 mL. Record the final volume.

9.9 Concentration of extracts using the Turbovap II.

9.9.1 Rinse the concentration (200 mL) tubes with methylene chloride.

9.9.2 Transfer the extract into the tube, rinse the Erlenmeyer and add it also to the tube.

9.9.3 Place the tube into the Turbovap II, which is set at 45° C, and close the cover.

9.9.4 Press the sensor button on the Turbovap and start the concentration process for each cell by pushing the start/stop button for each cell.

9.9.5 Adjust the gas flow to get a nice helical flow that does not spit out the sample.

9.9.6 When the endpoint is reached, the light next to its start/stop button will blink and the beeper sounds briefly every thirty seconds.

9.9.7 Remove the tube from the Turbovap and add 50 mL of hexane making sure that a proper exchange has occurred. Place the tube back into the Turbovap and concentrate the extract as described in 9.9.1 - 9.9.6.

9.9.8 When the endpoint is reached remove the tube from the Turbovap and transfer the extract into a calibrated 40 mL vial.

9.9.9 Adjust the final volume to 10 mL by adding hexane. Record the final volume.

9.10 Sulfur clean up will be used if the client requests this or if sulfur is known to be an interference with the extract.

9.10.1 Add 2 grams of cleaned copper powder for every 1 mL of extract. Vigorously shake the extract and the copper powder for 1 minute. Allow phases to separate and then proceed with the next cleanup.

9.11 Florisil Cleanup (for PCB only analysis skip to 9.12)

9.11.1 Prepare the Florisil column by pre-eluting the column with 5 mL of hexane. (Slowly open the valves and let some hexane go through). Close the valves and let soak for five (5) minutes (keep vacuum on). Do not allow cartridge to go dry, if cartridge goes dry, repeat the previous step. After five (5) minutes, let the hexane go through with a little bit above the frit. (Do not allow cartridge to go dry). Discard the hexane.

9.11.2 Just prior to the exposure of the florisil to air, transfer 5 mL of extract to the florisil column and collect the extract in a 40 mL pre-cleaned VOA vial calibrated to 5 mL.

9.11.3 After the sample has gone through the florisil, elute the column with 1 mL of 94:6 hexane: ether followed by 3 mL of 1:1 hexane: ether followed by 1 mL of 94:6 hexane: ether. Collect these elutions in the VOA vial.

9.11.4 Concentrate the volume to 5 mL using the N-evap as in step 9.8.5.

9.11.5 Transfer the extract to an autosampler vial and seal, mark sample level and label it with sample ID, extraction date and test.

9.12 Acid/Florisil Cleanup (**for PCB's only**)

9.12.1 Add 5 mL of concentrated sulfuric acid to the extract.

9.12.2 Stopper the tube and shake well for one (1) minute, venting once or twice to release any pressure. Allow the layers to separate for ten (10) minutes.

9.12.3 Pipet the cleaned up extract off the top and repeat if necessary. Pipet off 5 mL of extract for florisil cleanup.

9.12.4 Prepare florisil column and add the extract as in step 9.11.

9.12.5 After the sample has gone through the florisil elute the column with 3 mL of 94:6 hexane: ether. Collect in the VOA vial.

9.12.6 Concentrate the volume to 5 mL using N-evap as in step 9.8.5.

9.12.7 Transfer the extract to an autosampler vial and seal; mark the sample level and label with sample ID, extraction date and test.

10.0 QUALITY CONTROL (QC) REQUIREMENTS

10.1 A method blank, laboratory control sample, matrix spike and matrix spike duplicate are extracted with each batch. The only exception is if the client does not send enough sample to perform a matrix spike and matrix spike duplicate. Then a reagent blank, laboratory control sample and a laboratory control sample duplicate will be performed.

10.2 All batch quality control samples are subjected to exactly the same extraction and clean-up procedures as those used on actual samples in the extraction batch.

10.3 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).

11.0 WASTE MANAGEMENT AND POLLUTION CONTROL

11.1 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.

11.2 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.

11.3 The following are waste streams in the sample preparation area for the water extraction of pesticides and PCBs.

11.3.1 Halogenated solvents: Methylene Chloride

11.3.2 Non-Halogenated solvents: Hexane, Ether, Methanol, and Acetone

11.3.3 Solid Waste: Filters, tongue depressors, gloves, and any solid material that is a waste after being processed in the lab.

11.3.4 TCLP extract: the extract fluid that remains after extraction

11.3.5 PCB Acid: Acid waste that remains after the acid clean up of PCB extracts

12.0 REFERENCES

1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Update I, II, IIA, and III Method 3510C, 3500C, 3660B, 3665A, and 3620B.

2. CFR Chapter 1 (7-1-95 Edition) Method 608

3. Microbac SOP37, "Standard Operating Procedure for Extraction Glassware Washing"

4. Microbac SOP 33, "Laboratory Waste Management"

5. Microbac SOP LQAP "Laboratory Quality Assurance Plan"

6. Microbac SOP GP-CAPA, "Corrective Action/Preventive Action Initiating, Tracking and Monitoring"

7. Microbac SOP GP-RCA, "Root Cause Analysis"

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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

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David L. Bumgarner, Technical Director/QAO — Date: 09/03/2009

David E. Vandenberg, Managing Director — Date: 9/8/09

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
[signature]	9-03-09
Sheri L. Rodgers	9/3/09
[signature]	9-3-09

TABLE OF CONTENTS

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:

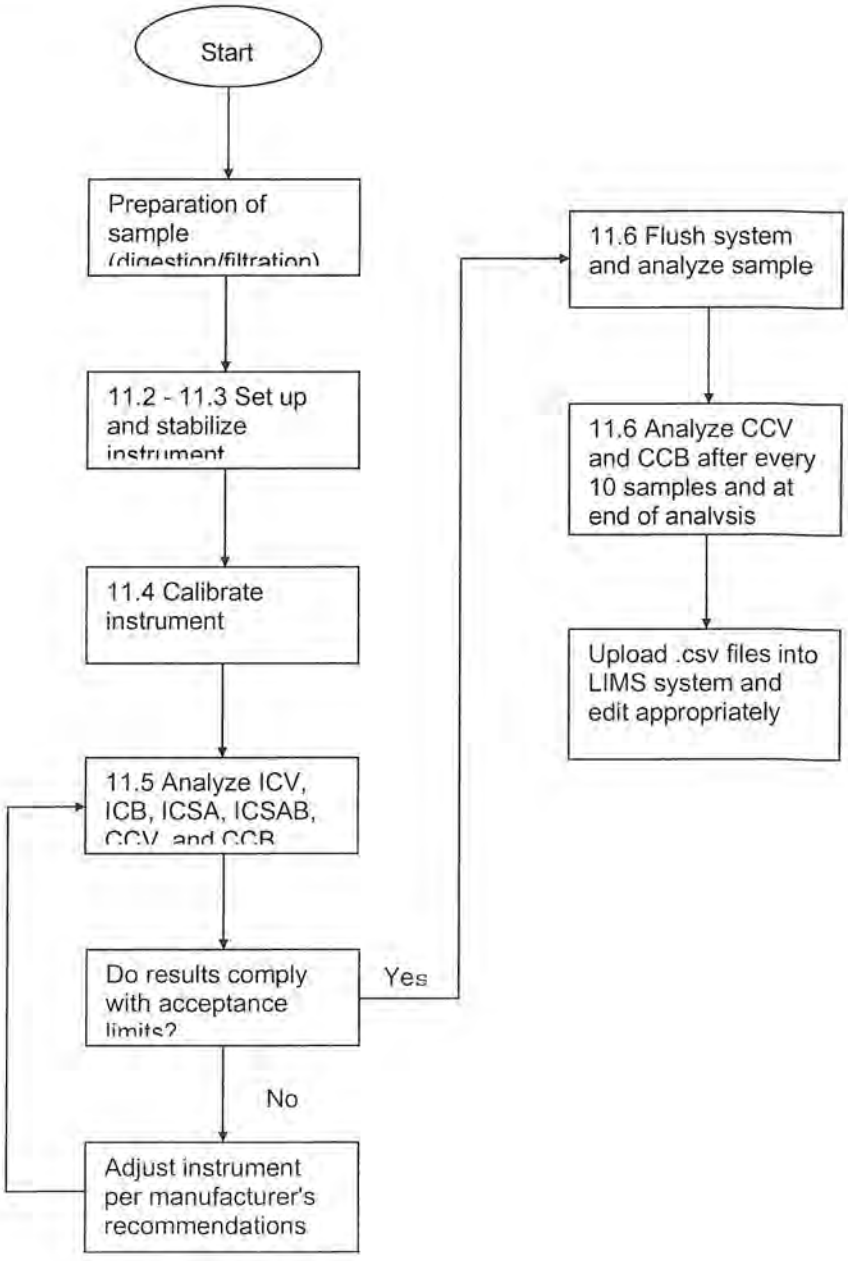
ME401 - Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP, SW846 Method 3005A.

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 $\geq$ 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 $\pm$ 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 $\leq$ 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 $\leq$ 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	$\pm$ 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 $\geq$ 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 $\pm$ 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 $\leq$ 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 $\leq$ 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	$\pm$ 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 (ug/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.

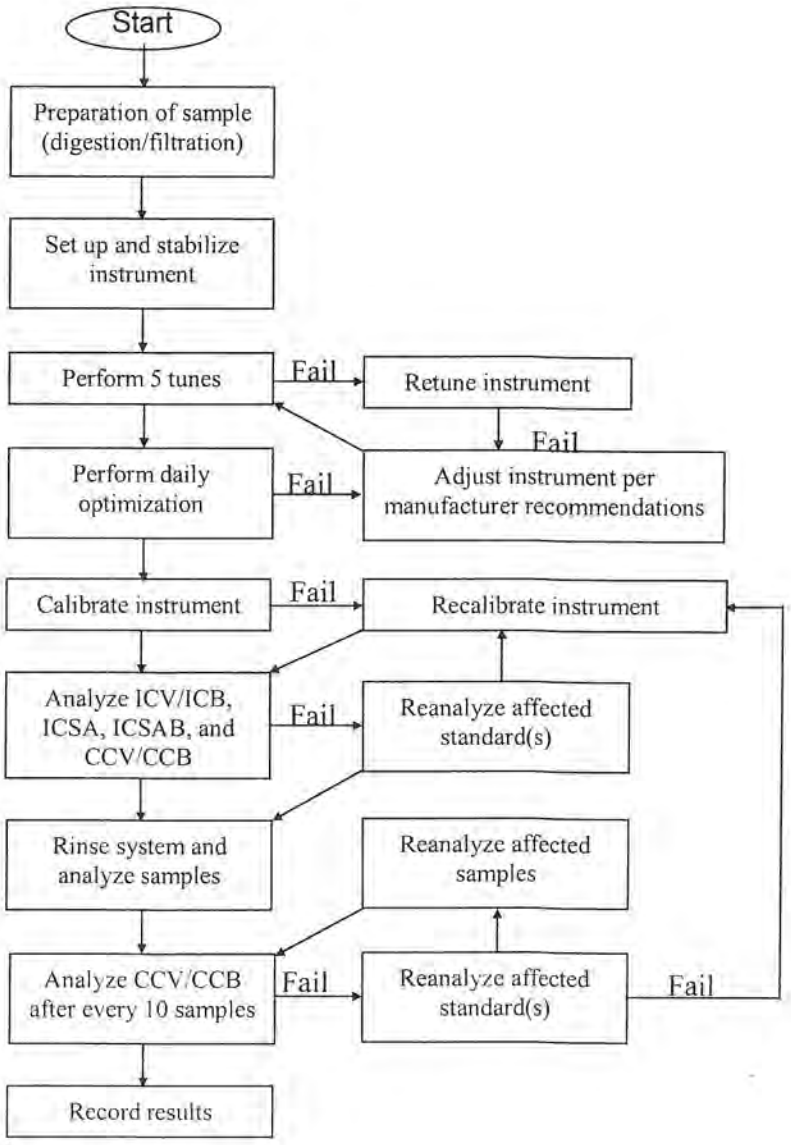
Extraction:

The process, which releases or removes specific target analytes for subsequent analyses.

FID:

Flame Ionization Detector

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:

$$\%R = \left[\frac{\left(C_{spk} - C_x\right)}{C_t}\right]100$$

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, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the 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 the 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 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):

$$Cr = Cs \times \frac{Vf}{Vi} \times D$$

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
Cr = 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):

$$Cr = Cs \times \frac{Vf}{Vi} \times D$$

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
Cr = Concentration of element in (ug/kg)	40

4.0 Adjusting the concentration to dry weight:

$$Cdw = \frac{Cr \times 100}{\%s}$$

Where:	Example:
Cr = Concentration calculated as received (wet basis)	40
$\%s$ = Percent solids of sample (%wt)	80
Cdw = 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:

Secondary Reviewer:
24-MAR-2009

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.

Microbac

STANDARD OPERATING PROCEDURE
MICROWAVE DIGESTION - AQUEOUS
SW846-3015

Issue/Implementation Date: 15 December 2009

Last Review Date: 15 December 2009

Microbac Laboratories, Inc.
158Starlite Drive
Marietta, Ohio 45750

Approved By:

Robert Kyer/BEP — 12/15/09
Robert Kyer, Metals Digestion Group Leader — Date

Maren M Beery — 12/15/09
Maren Beery, Metals Supervisor — Date

David L. Bumgarner — 12/16/09
David L. Bumgarner, Technical Director/QAO — Date

David E. Vandenberg, Managing Director — Date

Microbac

Table of Contents

1.0 SCOPE AND APPLICATION

1.1 This procedure utilizes SW-846 method 3015 and is an acid digestion procedure used to prepare surface water, groundwater, TCLP and mobility procedure extracts, and waste samples that contain suspended solids for analysis by atomic absorption spectroscopy (AA) or by inductively coupled argon plasma spectroscopy (ICP) or by ICP-MS. Samples prepared by this method may be analyzed by ICP or ICP-MS for the following metals:

Aluminum	Magnesium
Antimony	Manganese
Arsenic	Molybdenum
Barium	Nickel
Beryllium	Potassium
Cadmium	Selenium
Calcium	Silver
Chromium	Sodium
Cobalt	Thallium
Copper	Vanadium
Iron	Zinc
Lead	Uranium

1.2 For the analysis of dissolved metals, the sample is filtered at the time of collection, prior to acidification with nitric acid.

2.0 SAFETY PRECAUTIONS

2.1 Safety glasses, gloves and lab coats must always be worn when doing this procedure.

2.2 Always use a fume hood when adding concentrated Nitric acid (HNO_3) to the vessels.

2.3 Venting of the vessels should only be done when contents are at room temperature inside a hood with shield lowered to avoid the potential for chemical burns.

3.0 SAMPLE PRESERVATION AND STORAGE

Measurement	Digestion Volume Requirement (mL)	Collection Volume (mL)	Preservative/ Holding Time
Total	40	600	HNO_3 to pH <2 / 6 months
Dissolved	40	600	Filter on site; HNO_3 to pH <2 / 6 months
Suspended	40	600	Filter on site / 6 months

3.2 All sample containers must be pre-washed with detergents, acids, and ASTM Type II water.

3.3 Sampling:

3.3.1 Total recoverable metals: All samples must be acidified at the time of collection with concentrated nitric acid (HNO_3)(5 mL/L).

3.3.2 Dissolved metals: All samples must be filtered through a 0.45 micrometer filter and then acidified at the time of collection with concentrated nitric acid (HNO_3)(5 mL/L).

3.4 Aqueous waste waters must have the pH checked & adjusted to 2 or less with nitric acid (HNO_3) and the pH recorded in the pH logbook before digestion.

3.5 All TCLP extracts (except matrix spikes) must be immediately preserved with nitric acid and the pH recorded in the pH logbook.

4.0 INTERFERENCES AND CORRECTIVE MEASURES

Very reactive or volatile materials that create high pressure when heated may cause venting of the vessels with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds may cause enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller amount of sample.

5.0 EQUIPMENT AND SUPPLIES

5.1 Major Instrumentation

5.1.1 Mars Xpress unit. Microwave unit must provide programmable power with a minimum of 574W and can be programmed to within ±10W of required power.

5.1.2 75 mL Vessels for the Mars Express

5.2 Apparatus or Equipment

Beckman GS-6 centrifuge or equivalent.

Analytical balance (600g capacity) or equivalent.

5.3 Other Supplies

Graduated digest tubes (50 mL or 100 mL Capacity)

Quantitative filter paper, Whatman 41 or equivalent

Volumetric pipettes

VWR 50 mL disposable centrifuge tubes or equivalent

6.0 STANDARDS AND REAGENTS

Acids used in the preparation of samples must be reagent grade or better. Redistilled acids may 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.

6.1 ASTM Type II Water (ASTM D1192): Water must be monitored for impurities.

6.2 Concentrated Nitric Acid (HNO_3) (Baker Instra analyzed or equivalent): acid must either be analyzed to determine impurities or be certified by the manufacturer.

6.3 QC-MS-1 Spike – 10 mg/L, As, Al, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Sb,Se, Ag, Tl, U, V and Zn in 2% HNO3 and tr HF – CPI International.

6.4 Custom multi-element solution MIC-SPK-1A from Inorganic Ventures with the following concentrations:

Table 6.1
Concentrations of Elements in Purchased Standard MIC-SPK-1A

Element	Concentration (mg/L)
Potassium Sodium	250
Aluminum Calcium Magnesium	50
Silicon	25
Iron	20
Boron	10
Antimony	6
Barium Lithium Molybdenum Strontium Titanium Vanadium Zinc	5
Chromium Copper Manganese Nickel Lead Thallium	2.5
Arsenic Selenium Silver	2
Cobalt	1
Beryllium Cadmium	0.25

7.0 CALIBRATION PROCEDURES

7.1 The Mars Express is calibrated annually by the Manufacturer.

8.0 SAMPLE PREPARATIONS

Samples are shaken to homogenize prior to digestion.

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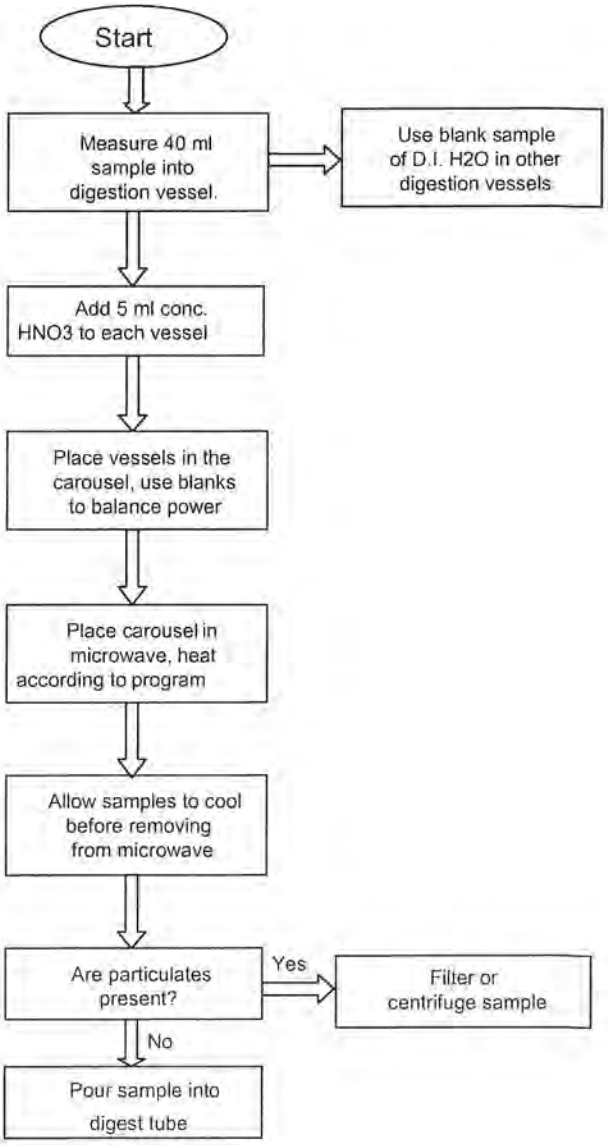
9.0 DIAGRAM OF TABLES TO OUTLINE PROCEDURES



10.0 STEP-BY-STEP ANALYTICAL PROCEDURES

10.1 Choose 20 samples of similar matrix for the preparation batch.

10.2 Measure 40 mL of a well shaken sample into a digest tube and transfer the aliquot into a digestion vessel liner. Add 5 mL concentrated nitric acid (HNO_3) to each vessel. **NOTE** -If a high organic content is suspected, such as in TCLP extracts, 5 mL or less may be used (the difference is made up with deionized water). The blank and laboratory control sample are made by using 40 mL of deionized water. The LCS and MS/MSD's are spiked with the appropriate spike (Regular water batches get 0.25 mL of QC-MS-1 spike (6.3) CPI International and TCLP batches get 5 mL for LCS and 5 mL for MS/MSD of Custom Multi-element solution MIC-SPK-1A (6.4) Inorganic Ventures.

10.2.1 As per method 200.8, there must be a sample duplicate per every batch of twenty (20) samples or less and a sample matrix spike (MS) per every ten (10) samples or less.

10.3 Mars Xpress – Follow these guidlines

10.4 Seal all samples with rubber stoppers and also the vessel cap. Hand tighten vessel cap only.

Weigh vessels and record weight in microwave electronic benchsheet.

Place vessels in Carousel making sure they are pushed down completely.

Select "Load Method" from main menu.

Select "User Directory".

Select appropriate 3015 method to be used (EPA 3015-8V-Xpress, - 16 Xpress, -24 Xpress).

Push start button.

10.4.1 After the program has finished, allow sample to cool down.

10.4.2 Remove Carousel from unit and reweigh vessels and record weight in electronic benchsheet. If the weight has decreased more than 1% from the original weight, discard sample and start the sample over again.

10.4.3 Transfer the solution to a graduated digest tube and bring up to a 50 ml volume with DI H_2O.

10.4.4 Centrifugation: Transfer sample in 50 graduated centrifuge tube and place in centrifuge for at least 10 minutes at 2,000 – 3,000 RPM. Slowly decant sample into a clean digest tube and bring up to a 50 mL volume with DI Water. The sample is now ready for analysis. If filtration is needed after centrifugation, this is done by filtering sample through a funnel lined with whatman 934-AH filter paper right into a clean digest tube and bringing up to a 50 mL volume with DI water. The sample is now ready for analysis.

11.0 QUALITY CONTROL (QC) REQUIREMENTS

11.1 Each batch of up to 20 samples requires the following:

- Method blank – a 40ml aliquot of deionized water that is digested with the sample batch.
- Laboratory Control Sample (LCS) – a 40 ml aliquot of deionized water that is spiked with spiking solution and digested with the sample batch.
- Sample duplicate – Batches that include samples for method 200.8 will include a sample prepared in duplicate, both carried through the batch digestion.
- Matrix spike and matrix spike duplicate – two additional aliquots of a sample that are spiked with spiking solution and digested with the sample batch. Sn spike is added to each MS/MSD that Sn is needed. Batches that include samples for method 200.8 will include a spiked sample for every ten 200.8 samples.

11.2 Results of the analysis of the QA/QC samples are kept for easy reference.

11.3 All batch quality control samples are subjected to exactly the same digestion as those used on actual samples in the digestion batch.

11.4 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).

11.4.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13.1 and 13.2 of SOPs ME600E, ME600F and ME600G and Tables 13.1 and 13.4 of SOP ME 700, does not meet acceptance criteria. When nonconforming data occurs the employee initiates a Nonconformance Report (NCR) and proceeds with indicated corrections as per Tables 13.1 and 13.2 of SOPs ME600E, ME600F and ME600G and Tables 13.1 and 13.4 of SOP ME 700.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch Figure 14.1 of SOPs ME600E, ME600F, ME600G and ME700. 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)

11.4.2 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.

11.4.3 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.

12.0 PREVENTATIVE MAINTENANCE

12.1 Monthly examine the door, door seals and door interlocks to verify they are clean and working properly. Ensure that the door closes securely.

12.2 Clean the inside of the microwave cavity, including the exhaust screen at the back of the cavity, with warm soapy water applied with a soft cloth. Rinse and thoroughly dry all cleaned areas.

12.3 Clean the exhaust outlet by removing the exhaust hose and wiping the space inside the exhaust outlet with a disposable cloth. To clean the exhaust hose, disconnect if from the blower exhaust duct, flush it with water and allow it to dry before reconnecting it to the blower duct.

13.0 WASTE MANAGEMENT AND POLLUTION CONTROL

13.1 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.

13.2 The following are waste streams in the sample preparation area.

13.2.1 Non – Halogenated solvents: Acetone

13.2.2 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

13.2.3 Acid: Dilute acid waste from soak tanks.

13.3 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 received 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.

14.0 REFERENCES

14.1 Microwave Assisted Acid Digestion of Aqueous Samples and Extracts, US EPA SW-846 Method 3015, Revision 0, September 1994, EPA Publication SW-846.

14.2 Microwave Assisted Acid Digestion of Aqueous Samples and Extracts, US EPA SW-846, Method 3015A, Revision 1, February 2007, EPA Publication SW-846.

14.3 SOP LQAP – "Laboratory Quality Assurance Plan"

14.4 SOP 33- "Laboratory Waste Management"

14.5 SOP 45 – "Method Validation Procedures"

14.6 SOP GP-CAPA – "Corrective Action and Preventive Action; Initiating, Tracking, and Monitoring"

14.7 SOP GP-RCA – "Root Cause Analysis"

14.8 SOP 600E – "Perkin Elmer OPTIMA 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy"

14.9 SOP 600F – "Thermo Jarrell Ash IRIS Advantage Inductively Coupled Plasma Atomic Emission Spectroscopy"

14.10 SOP ME600G – "Thermo iCAP 6000 Series Inductively Coupled Plasma Atomic Emission Spectroscopy"

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14.11 SOP ME700- "Perkin Elmer Elan 6100 Inductively Coupled Plasma/Mass Spectrometer"

Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method:

HNO_3 – Nitric Acid

Microbac

STANDARD OPERATING PROCEDURE

MICROWAVE DIGESTION OF SEDIMENTS, SLUDGES, SOILS AND OILS (3051)

Issue / Implementation Date: 15 November 2009

Last Review Date: 15 May 2010

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Robert Kyer, Digest Supervisor	5/3/10 Date
Maren Beery, Metals Supervisor	05/03/2010 Date
David L. Bumgarner, Technical Director/QAO	05/04/2010 Date
David E. Vandenberg, Managing Director	5/7/10 Date

Table of Contents

1.0 SCOPE AND APPLICATION

1.1 This procedure references SW-846 method 3051 and is applicable to the microwave assisted acid digestion of sediments, sludges, soils, and oils. Samples prepared by this method may be analyzed by flame atomic absorption (FAA), by inductively coupled argon plasma spectroscopy (ICP), or by ICP-MS for the following metals:

Aluminum	Copper	Silver
Antimony	Iron	Sodium
Arsenic	Lead	Strontium
Boron	Magnesium	Thallium
Barium	Manganese	Uranium
Beryllium	Molybdenum	Vanadium
Cadmium	Nickel	Zinc
Calcium	Phosphorus	Zirconium
Chromium	Potassium	
Cobalt	Selenium	

Samples prepared by this method may be analyzed by ICP for additional metals if the QC criteria of the method can be achieved.

2.0 SAFETY PRECAUTIONS

2.1 Safety glasses, gloves and lab coats must always be worn when doing this procedure.

2.2 Always use a fume hood when adding concentrated Nitric acid (HNO_3) and Hydrochloric Acid (HCl) to the vessels and when manually venting the vessels to avoid exposure to toxic fumes.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in SW-846.

3.2 All sample containers are certifiable pre-cleaned from the manufacturer.

3.3 Samples must be refrigerated upon receipt.

3.4 Samples may be held a maximum of 180 days prior to digestion. Samples digested after 180 days will be considered to have exceeded the holding time.

4.0 INTERFERENCES AND CORRECTIVE MEASURES

Very reactive or volatile materials that create high pressure when heated may cause venting of the vessels with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds may cause enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller amount of sample.

5.0 EQUIPMENT AND SUPPLIES

5.1 Major Instrumentation

5.1.1 MARS Express microwave unit or equivalent. Microwave unit must provide programmable power with a minimum of 574W and can be programmed to within ± 10W of required power.

5.1.2 75 mL vessels for the Mars Express.

5.2 Apparatus or Equipment

5.2.1 Beckman GS-6 centrifuge or equivalent.

5.2.2 Analytical balance (600 g capacity) or equivalent.

5.3 Other Supplies

5.3.1 Quantitative filter paper, Whatman 41 or equivalent

5.3.2 Volumetric pipettes

5.3.3 50 mL disposable centrifuge tubes or equivalent.

6.0 STANDARDS AND REAGENTS

6.1 ASTM Type II Water (ASTM D1192):
Water should be monitored for impurities.

6.2 Concentrated Nitric Acid (HNO_3) and concentrated Hydrochloric Acid (HCl) (Baker Instra - analyzed or equivalent): acid should either be analyzed to determine impurities or be certified by the manufacturer. If the method blank is less than the method detection limit, the acid may be used.

6.3 Spiking Solutions

a) If digesting for ICP-MS, use 0.5 mL of QC-MS-1, CPI International.
b) If digesting for ICP, use 5 mL of MIC-SPK-, Inorganic Ventures.

6.4 Single element Tin: 1000 ug/mL from Inorganic Venutres

6.5 Single element Phosphorus: 1000 ug/mL from Inorganic Ventures

6.6 Single element Zirconium: 1000 ug/mL from Inorganic Ventures

7.0 CALIBRATION PROCEDURES

7.1 The Mars Express is calibrated annually by the service engineer.

8.0 SAMPLE PREPARATION

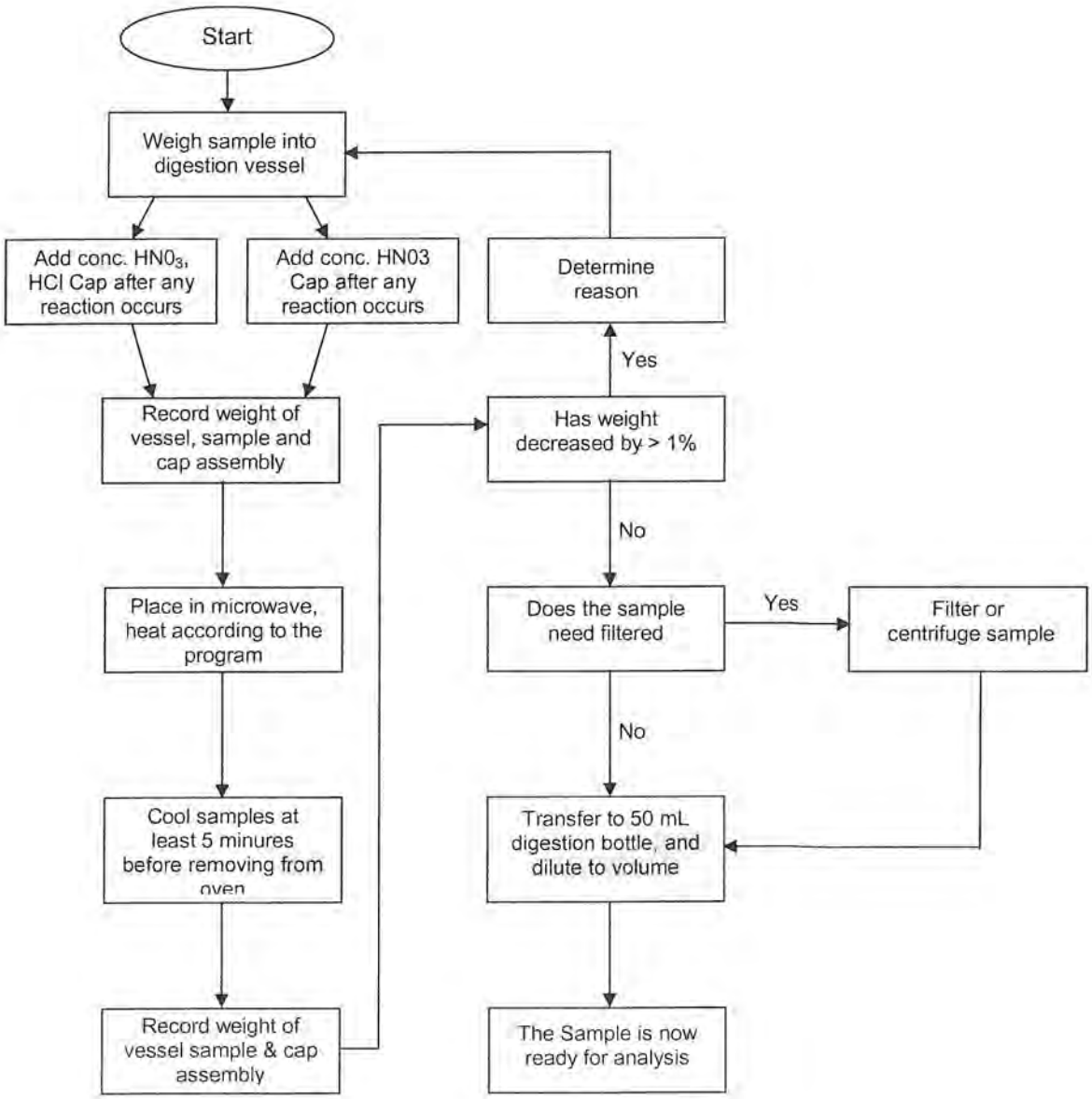
8.1 Samples are digested using SW-846 Method 3051A utilizing the appropriate acids.

8.2 Soil subsampling

a) Remove sample bottle contents and place in a tray lined with wax paper mix sample with an inert rod or scoop and break up lumps. Remove all large stones, sticks, leaves, etc. Do not overmix the sample
b) Obtain representative sample either by random removal of 3-10 portions of the sample from the pan or by using a "standard" scoop designed to retrieve a linear cross-section of the pan contents
c) The analyst will not attempt to target an exact weight once a method specified minimum amount is weighed.
d) The remaining sample will be returned to the sample container.

9.0 DIAGRAM OF TABLE TO OUTLINE PROCEDURES

Microwave Digestion of Sediments, Sludges, Soils and Oils



10.0 ANALYTICAL PROCEDURES

10.1 MARS Express – follow these guidelines.

10.1.1 Choose no more than 20 soil samples in a preparation batch.

10.1.2 Following making of workgroup, go to "Sample Data", "Digestions", then "Microwave". Here is where initial weights are recorded.

10.1.3 For ICP-MS analysis, weigh a 0.5 g portion of a well mixed sample into a digestion vessel. Add 10 mL of HNO_3 to each vessel. For Blanks and LCS/LCS Dup weigh 0.5 g or approximately 6 Teflon chips. Add 10 mL of HNO_3 to the blank and LCS. The LCS, MS, and MSD are spiked with 0.5 mL of microwave spike (6.3a).

For ICP analysis, weigh out 1.3 - 1.5 g of a well mixed sample into a digestion vessel. The LCS, MS, and MSD are spiked with 5 mL of general spike (6.3b). 25 uL of tin is added to each LCS. 25 uL of phosphorus and zirconium are added when needed. Teflon chips are used for blanks and LCS, approximately 12 Teflon chips. Add 9 mL of HNO_3 and 3 mL of HCl to each vessel. For oils weigh out 0.25 g. Add 10 mL of HNO_3 to the vessels. For Blank, LCS and LCS Dup weigh 0.25 g or approximately 3 Teflon chips. Add 10 mL of HNO_3. Spike LCS/LCS Dup with 5 mL of spiking solution (6.3b).

Seal all samples with rubber stoppers and also the vessel cap. Hand tighten vessel cap only.

Weigh vessels and record weight under "Sample Data".

Place vessels in Carousel making sure they are pushed down completely.

Select "Load Method" from main menu.

Select "User Directory".

Select appropriate 3051 method to be used (EPA 3051-8V-Xpress, - 16 Xpress, -24 Xpress).

Push start button.

10.1.4 After the program has run, allow samples to cool down.

10.1.5 Remove Carousel from unit and reweigh vessels and record weight under "Sample Data". If the weight has decreased more than 1% from the original weight, discard sample and start the sample over again.

10.1.6 Transfer the solution to a graduated centrifuge tube. Rinse the microwave vessel two times with DI water and transfer the rinsates to the tube.

10.1.7 Centrifugation: Place sample in centrifuge for at least 10 minutes at 2,000 – 3,000 RPM. Slowly decant sample into a clean digest tube, add volume up to 50 mL with the DI water. The sample is now ready for Analysis. If filtration is needed after centrifugation, this is done by filtering sample through a funnel lined with Whatman 934-AH filter paper right into a clean digest tube. The sample is now ready for analysis.

11.0 QUALITY CONTROL (QC) REQUIREMENTS

11.1 Each batch of up to 20 samples requires the following:

- Method blank – for ICP-MS analysis, weigh 0.5 g or approximately 6 Teflon chips. 10 mL of concentrated nitric acid is digested with the sample batch. For ICP analysis, weigh 1.0 g or approximately 12 Teflon chips, 9 mL of concentrated HNO_3 and 3 mL of concentrated HCl is used.

- Laboratory Control Sample (LCS) – for ICP-MS analysis, 0.5 g or approximately 6 Teflon chips, that is spiked with spiking solution and digested with the sample batch. For ICP analysis, use 1.0 g of Teflon chips, approximately 12 Teflon chips.

- Sample duplicate – a sample prepared in duplicate, both carried through the batch digestion.

- Matrix spike and matrix spike duplicate – two additional aliquots of a sample that are spiked with spiking solution and digested with the sample batch.

11.2 Results of the analysis of the QA/QC samples are kept for easy reference.

11.3 All batch quality control samples are subjected to exactly the same digestion as those used on actual samples in the digestion batch.

11.4 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).

11.4.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13-1 and 13-2 of SOPs ME600E, F or G and Tables 13-1, 13-4, and 13-6 of SOP ME700, does not meet acceptance criteria. When nonconforming data occurs the employee initiates a Nonconformance Report (NCR) and proceeds with indicated corrections as per Tables 13-1 and 13-2 of SOPs ME600E, F or G and Tables 13-1, 13-4, and 13-6 of SOP ME700.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 14.1 of SOPs ME600 E, F, and G and ME700). 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)

11.4.2 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-RAC, corrections, corrective action (s) and evidence of effectiveness.

11.4.3 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.

12.0 PREVENTATIVE MAINTENANCE

12.1 Monthly examine the door, door seals and door interlocks to verify they are clean and working properly. Ensure that the door closes securely.

12.2 Clean the inside of the microwave cavity, including the exhaust screen at the back of the cavity, with warm soapy water applied with a soft cloth. Rinse and thoroughly dry all cleaned areas.

12.3 Clean the exhaust outlet by removing the exhaust hose and wiping the space inside the exhaust outlet with a disposable cloth. To clean the exhaust hose, disconnect if from the blower exhaust duct, flush it with water and allow it to dry before reconnecting it to the blower duct.

13.0 WASTE MANAGEMENT AND POLLUTION CONTROL

13.1 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 of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

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.

13.2 The following are waste streams in the sample preparation area.

13.3.1 Non – Halogenated solvents: Acetone

13.3.2 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

13.3.3 Acid: Dilute acid waste from soak tanks.

NOTE: 13.3.1,and 2 are all kept in satellite containers in each lab and are combined into the proper 55 gallon waste drums in our explosion proof waste building located outside of the Microbac laboratory facility by a waste disposal technician. 13.3.3 is neutralized and disposed in the municipal sewer system as per agreement with the city.

14.0 REFERENCES

14.1 Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils, US EPA SW-846 Method 3051A, Revision 1, February 2007, EPA Publication SW-846.

14.2 SOP LQAP "Laboratory Quality Assurance Plan"

14.3 SOP 45 "Method Validation Procedures"

14.4 SOP 600E "Perkin Elmer Optima 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy"

14.5 SOP 600F "Thermo Jerrel Ash Iris Advantage Inductively Coupled Plasma Atomic Emission Spectrascopy".

14.6 SOP 600G "Thermo iCAP 6000 Series Inductively Coupled Plasma Atomic Emission Spectroscopy"

14.7 SOP 700 "Perkin Elmer Elan 6100 Inductively Coupled Plasma/Mass Spectometer.

Definitions and Acronyms

HCl – Hydrochloric Acid

HNO_3 – Nitric Acid

ICP – Inductively Coupled Plasma

ICP-MS – Inductively Coupled Plasma – Mass Spectrometry

FAA – Flame Atomic Absorption

For a more comprehensive list of common terms and definitions, consult Appendix A in SOP LQAP.

Appendix I

California ELAP samples digested by method 3051A (SOP ME406) will not have valid results reported below the nominal reporting limit. Should such "J" flagging of results occur, they must be considered insignificant.

Microbac

STANDARD OPERATING PROCEDURE
SONICATION EXTRACTIONS
FOR POLYAROMATIC HYDROCARBONS
Method 3550B AND 3550C

Issue/Implementation Date: 15 February 2010

Last Review Date: 15 February 2010

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Chad E. Barnes, Extraction Supervisor — Date 2/9/10

David L. Bumgarner, Technical Director/QAO — Date 2/11/10

David E. Vandenberg, Managing Director — Date

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure for extracting and isolating polyaromatic hydrocarbons (PAHs) from solid samples. This method also describes the concentration and cleanup procedure involved in preparing the extract for analysis by GC. The primary reference for this method is SW-846 3550B and 3550C.

1.2 A weighed amount of sample, usually 30 grams, is extracted with methylene chloride using a sonicator. The extract is concentrated and cleaned up for analysis by GC.

2.0 SAFETY PRECAUTIONS

2.1 Proper gloves, lab coat, safety glasses and ear protection must be worn while performing this extraction procedure.

2.2 The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined. However, each chemical compound should be treated as a potential health hazard.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Samples should be collected in widemouth glass containers with teflon lined caps.

3.2 Sample weight needed for analysis is 30 grams but if sample weight is less than 30 grams, record the actual weight of sample and contact the TSR.

3.3 Sample preservation should be 4° C, and the sample maximum holding time from date of collection is 14 days.

4.0 INTERFERENCES & CORRECTIVE MEASURES

4.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing.

4.2 Other interferences that may be encountered are discussed in Method 3500C from SW846.

5.0 EQUIPMENT AND SUPPLIES

5.1 Major Instrumentation

5.1.1 Zymark Turbo Vap II concentration workstation

5.1.2 Ultrasonic cell disrupter: Misonix Model XL2020 sonicator or equivalent with a ¾" tapped disrupter horn.

5.1.3 Nitrogen Evaporator: Meyer N-Evap Analytical Evaporator or equivalent

5.2 Apparatus or equipment

5.2.1 Kuderna-Danish (K-D) apparatus:

5.2.1.1 Concentrator tube: 15 mL graduated (Supelco 6-4684M or equivalent)

5.2.1.2 Evaporation Flask: 500 mL attached to a concentrator tube with a clip or spring.

5.2.1.3 Snyder column: Three-ball macro

5.2.2 Water bath: Heated, with concentric ring cover, capable of temperature control (± 5° C). The bath should be used in a hood.

5.3 Glassware

5.3.1 Beakers: 250 mL

5.3.2 Erlenmeyer flask: 250mL

5.3.3 Autosampler vials: 2-mL capacity

5.3.4 Zymark 200 mL (with a 1.0 mL stem)

5.3.5 Calibrated 40 mL vials

5.4 Other Supplies

5.4.1 Stainless steel funnel or glass funnel

5.4.2 Boiling chips: 10/40 mesh

5.4.3 Syringes – various sizes

5.4.4 Filter paper: Whatman #41 or equivalent

6.0 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.

6.1 Deionized water.

6.2 Acetone: reagent grade or equivalent.

6.3 Sodium sulfate: Granular, anhydrous.

6.4 Sodium sulfate: Powdered, anhydrous.

6.5 Methylene chloride: Pesticide grade or equivalent.

6.6 Sulfuric acid solution (1:1): Slowly add 50 ml of sulfuric acid to 50 ml of deionized water.

6.7 Surrogate for low level PAH (827-PAHL)[use 250 uL):

2-Fluorophenol	20 ug/mL
Phenol-d5	20 ug/mL
2,4,6-Tribromophenol	20 ug/mL
2-Fluorobiphenyl	10 ug/mL
Nitrobenzene-d5	10 ug/mL
p-Terphenyl-d14	10 ug/mL

6.8 AccuStandard custom PAH standard or equivalent at 1.0 ug/mL for 827-PAHL extractions (use 1.0 mL):

Acenaphthene	Dibenzo(a,h)anthracene
Acenaphthylene	Fluoranthene
Anthracene	Fluorene
Benzo(a)anthracene	Indeno(1,2,3-cd)pyrene
Benzo(a)pyrene	1-Methylnaphalene
Benzo(b)fluoranthene	2-Methylnaphthalene
Benzo(g,h,i)perylene	Naphthalene
Benzo(k)fluoranthene	Phenanthrene
Chrysene	Pyrene

7.0 CALIBRATION PROCEDURES

7.1 The probe or microtip should not be immersed in the liquid or come in contact with the work surface when tuning.

7.2 When operating with liquids at extreme temperature, immerse the probe in the liquid for a few minutes, remove from the liquid, then tune.

7.3 Secure the clamp to the converter housing only. Clamping the horn or the front driver will prevent ultrasonic sound waves from traveling through the horn.

7.4 Turn OUTPUT CONTROL knob counter-clockwise to zero.

7.5 Press POWER SWITCH to ON (up) position. The switch will illuminate.

7.6 When the prompt [for tuning procedure refer to manual] appears, press TUNE key. Screen will read: [TUNING --- PROBE ACTIVE].

7.7 Turn the Output Control Knob towards setting 3.

7.7.1 Note the position of the Bar Graph on the LCD Display Screen. Do **NOT** exceed 70%.

7.7.2 Rotate the Tuning Knob clockwise or counter-clockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.

7.8 Turn Output Control Knob towards setting 6.

7.8.1 Again, note the position of the Bar Graph and do not exceed 70%.

7.8.2 Rotate the tuning knob until you obtain a meter reading of 20% or below.

7.9 Repeat step 5 on power setting 10. Minimize the reading one last time to 20% or less.

7.10 Press the TUNE key to display prompt for programmed or continuous operation.

NOTE: If unit will not be used immediately, turn power switch to off

8.0 SAMPLE PREPARATION

8.1 Extraction Method 3550B and 3550C from SW-846.

8.2 Sample Homogenization

8.2.1 Remove sample bottle contents and place in tray lined with aluminum foil.

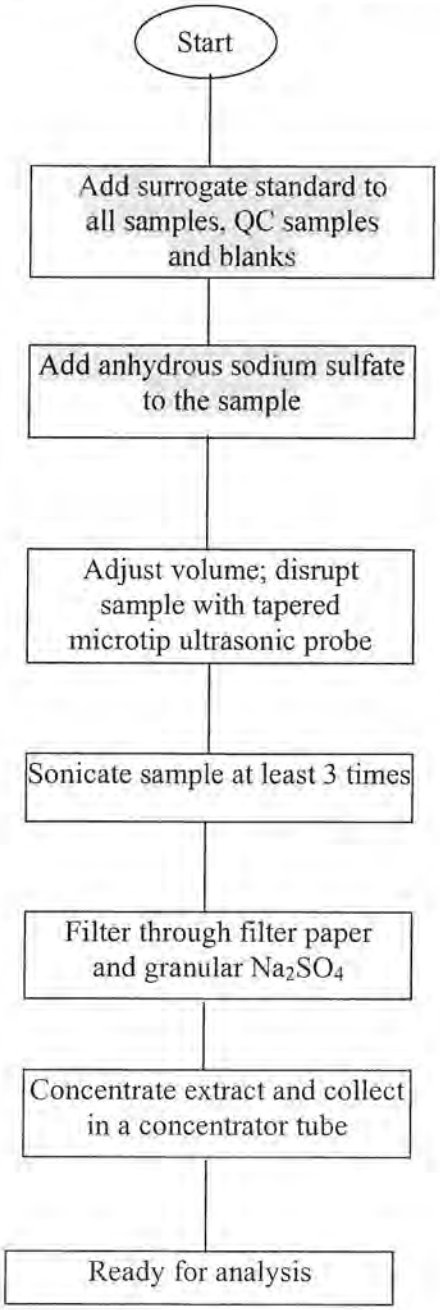
8.2.2 Mix sample with an inert rod or scoop and break up lump. Remove all large stones, sticks, leaves, etc. Do not overmix the sample.

8.2.3 Obtain representative sample either by random removal of 3-10 portions of the sample from the pan or by using a "standard" scoop designed to retrieve a linear cross-section of the pan contents.

8.2.4 The analyst will not attempt to target an exact weight once a method specified minimum amount is weighed.

8.2.5 The remaining sample will be returned to the sample container.

9.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



10.0 ANALYTICAL PROCEDURES

10.1 Rinse all extraction glassware with methylene chloride.

10.2 Weigh out 30 grams [±15%, do not target weight – see section 8.2] of sample into a 250 ml beaker and record to the nearest 0.1 g. Add 100 uL of surrogate (See Section 6.7 or 6.9) to all samples, spikes and blanks. Add 250 uL of the PAH spike (See Section 6.8 or 6.10) solution to the LCS and the samples designated as matrix spikes. Add powdered sodium sulfate to the sample and mix until it is free flowing. Immediately add methylene chloride to the 100 ml mark on the beaker. Record the volume of surrogate and spike additions.

10.3 To sonicate place the bottom of the tip of the horn below the surface of the liquid but above the sediment layer.

10.4 Sonicate for three (3) minutes with a 1 second pulse making sure the liquid is mixed thoroughly with the sample.

10.5 Decant the extract through a funnel lined with filter paper and granular sodium sulfate into a 250 ml Erlenmeyer flask. Rinse the horn with methylene chloride between sonications.

10.6 Repeat the extraction (10.3 - 10.5) two (2) more times adding methylene chloride to the 100 ml mark each time and combine the extracts in a 250 ml Erlenmeyer flask.

10.7 Concentration of extracts using the Kuderna-Danish apparatus.

10.7.1 Attach a 15 ml concentrator tube to a 500 ml Kuderna-Danish (K-D) flask with a spring and a clip.

10.7.2 Transfer the extract to the K-D flask and rinse the Erlenmeyer flask with 10-20 ml of methylene chloride and add it to the K-D flask.

Add a boiling chip to the K-D flask and attach the snyder column to the top. Prewet the snyder column by adding about 1 ml of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80 - 90 ° C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. When the apparent

volume reaches 1 ml, remove the K-D apparatus from the water bath and allow it to cool for at least ten (10) minutes.

10.7.3 Remove the concentrator tube from the K-D flask and place it on the N-evap.

10.7.4 To use the N-evap, turn on the heater to 40° C. Turn on the gas and lower the needle so that it is about a centimeter above the liquid. When the apparent volume of liquid reaches 0.5 ml, remove the concentrator tube from the N-evap and allow to cool. Adjust the final volume to 1 ml with methylene chloride. Record final volume.

10.7.5 Transfer the extract to an autosampler vial and seal, mark the sample layer and label with sample ID, extraction date and test.

10.8 Concentration of extracts using the TurboVap II.

10.8.1 Rinse the concentration (200 ml) tubes with the methylene chloride.

10.8.2 Transfer the extract into the tube, rinse the Erlenmeyer and add it also to the tube.

10.8.3 Place the tube into the TurboVap II, which is set at 45° C, and close the cover.

10.8.4 Press the sensor button on the TurboVap II and start the concentration process for each cell by pushing the start/stop button for each cell.

10.8.5 Adjust the gas flow to get a nice helical flow that does not spit out the sample.

10.8.6 When the endpoint is reached, the light next to its start/stop button will blink and the beeper sounds briefly every thirty seconds.

10.8.7 Adjust the final volume to 1 ml with methylene chloride and transfer the extract to an autosampler vial marked with the sample ID, extraction date, test, and fraction also on the vial. Seal the vial.

11.0 QUALITY CONTROL (QC) REQUIREMENTS

11.1 A reagent blank, laboratory control sample, matrix spike and matrix spike duplicate are extracted with each batch. The only exception is if the client does not send enough sample to perform a matrix spike and matrix spike duplicate.

Then a reagent blank, laboratory control sample and a laboratory control sample duplicate may be performed at the client's request.

11.2 All batch quality control samples are subjected to exactly the same extraction and clean-up procedures as those used on actual samples in the extraction batch.

11.3 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).

12.0 WASTE MANAGEMENT AND POLLUTION CONTROL

12.1 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.

12.2 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.

12.3 The following are waste streams in the sample preparation area.

12.3.1 Halogenated solvents: Methylene Chloride

12.3.2 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

13.0 REFERENCES

1. Test Methods for Evaluation Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition Method 3500C, 3550B, 3550C Update I, II, IIA and III.

2. Microbac SOP 33, "Laboratory Waste Management"

3. Microbac SOP LQAP "Laboratory Quality Assurance Plan"

4. Microbac SOP GP-CAPA, "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

5. Microbac SOP GP-RCA, "Root Cause Analysis"

APPENDIX C

DEQ Memorandum of January 8, 2008

VIRGINIA DEPARTMENT OF ENVIRONMENTAL QUALITY

VIRGINIA DEPARTMENT OF ENVIRONMENTAL QUALITY
WASTE DIVISION
OFFICE OF HAZARDOUS WASTE

Memorandum

TO: Matthew Stepien
Environmental Engineer Sr.

THROUGH: Jutta Schneider
Hazardous Waste/Groundwater Manager

FROM: Fuxing Zhou
Environmental Specialist II

DATE: January 8, 2008

SUBJ: WELLS ABANDONMENT FOR HWMU 4
Radford Army Ammunition Plant, Radford; EPA ID# VA1210020730

I have reviewed the facility's letter dated December 4, 2007 and pertaining to groundwater monitoring well abandonment at HWMU #4 (Unit 4). The facility's letter provides notification for the abandonment of sixteen (16) groundwater monitoring wells at Unit 4.

Unit 4 was a Former Acid and Wastewater Equalization Basin. The unit was operated from 1971 to 1986, and was clean closed for soils in 1989. The unit had been in interim status in accordance with 40 CFR 265.90-94 (Groundwater Quality Assessment) from that time until August 2007. The Department acknowledged that clean closure for groundwater at Unit 4 had been achieved by letter dated August 16, 2007. Data indicated that there were no contaminants in the groundwater at the unit above risk-based levels.

Based on my review of these and other related documents for Unit 4 (monitoring system and hydrogeologic characteristics, 2006 annual report), there are at least twenty two (22) wells installed near Unit 4. The facility plans to abandon sixteen (16) of these, which are:

Upgradient: 4WC11;
Downgradient: 4W2B, 4WC21, 4WC22, 4WC23, 4WC32, 4WC41, CW42, 4WC43, 4W5A, 4W6A, and 4W7A and
Other (Downgradient) wells (not included in the current monitoring system): 4WC31, S4W2, S4W3, and S4W4.

The facility is retaining the following six (6) wells near the unit for possible future usage:

Upgradient: 4P3 and S4W1; and

Downgradient: 4W4B, 4MW7 and 4WC9B; and
Observation: 4WC8B.

Since clean closure for groundwater at the unit has been achieved, it is reasonable for the facility to abandon the groundwater monitoring wells at the unit as described.

The well abandonment methodology attached to the facility's letter is appropriate. A copy of the Department's IDW policy is attached for the facility's reference during the well abandonment process. Also, the facility should provide a report on well abandonment procedures as well as an abandonment certification to the Director within thirty (30) days from the date the wells are abandoned.

METHODOLOGY FOR GROUNDWATER MONITORING WELL ABANDONMENT

The facility shall receive prior approval from the Department to abandon any monitoring wells and/or piezometers. The following procedures shall be used

A. Monitoring wells and/or piezometers will be abandoned by pressure grouting methods. Surface installations (protective covers or manholes) will be removed and an attempt to pull the casing string with the rig will be made. Once this has either been accomplished or has failed, grouting operations will commence as described below.

1. Monitoring well abandonment will be accomplished by lowering a tremie pipe to the bottom of the borehole.

2. Portland cement/bentonite grout will then be pumped down the tremie pipe until an even flow of consistent grout returns at the surface.

3. The tremie pipe will be removed from the borehole on completion of grouting operations and a minimum four inch thick and six foot diameter concrete cap will be constructed over the grouted borehole.

B. Removed casings will be steam cleaned, cut up into manageable sections, and disposed of as refuse.

C. All tremie rods and other downhole equipment will be steam cleaned prior to introduction into the hole or well.

D. All purge water, decontamination water, used personal protective equipment (PPE), and disposable sampling equipment such as tubing are typical wastes that will be generated during quarterly sampling events. All decontamination fluid, used PPE and disposable sampling equipment will be managed and disposed in accordance with DEQ's Policy for Investigative Derived Waste (**IDW**, attached).

E. Well plugging methods and abandonment certification shall be submitted to the Director within thirty (30) days from the date the wells are removed from the monitoring program.

Department of Environmental Quality
Waste Operations
Policy for the Handling of
Investigation Derived Waste (IDW)

The Department of Environmental Quality (DEQ), Waste Operations has received a request for guidance from the regulated community concerning the Commonwealth of Virginia's requirements regarding the management and disposal of investigation derived waste (IDW). Because Virginia administers an authorized state RCRA program, the Virginia Solid Waste Management Regulations (VSWMR) and the Virginia Hazardous Waste Management Regulations (VHWMR) will serve as the governing requirements in lieu of Federal RCRA regulations contained in the Code of Federal Regulations (40 CFR 260 - 270) except for the Land Disposal Restrictions of 40 CFR 268. For reference, please see the Virginia Waste Management Act, Code of Virginia §10.1-1400 et seq.; the Virginia Hazardous Waste Management Regulations (VHWMR) (VR 672-10-1); the Virginia Solid Waste Management Regulations (VSWMR) (VR 672-20-10); Federal: the Resource Conservation and Recovery Act (RCRA), 42 USC 6901; and the U. S. Department of Transportation Rules for the Transportation of Hazardous Materials, 49 CFR Part 107, 171.1 - 172.558.

With regard to IDW, it is the site manager's responsibility to determine whether the wastes generated during an investigation meet the definition of a solid or hazardous waste. The site manager will be either the on-scene coordinator (i.e., either the federal official predesignated by the Environmental Protection Agency (EPA) or the U.S. Coast Guard to coordinate and direct federal responses under subpart D or the official designated by the lead agency to coordinate and direct removal actions under subpart E of the National Contingency Plan (NCP)), or the remedial project manager (i.e., the official designated by the lead agency to coordinate, monitor, or direct remedial or other response actions under subpart E of the NCP).

If there is a possibility that either the ground water or the soil at the location where a monitoring well is installed is contaminated, the site manager must determine whether or not the well cuttings, purge water, and/or other IDW are contaminated (i.e., whether they are solid or hazardous wastes). In these cases, the site manager may use knowledge of the contaminated media to declare that the IDW is solid or hazardous waste. If analysis shows that no contamination is present in the soil or the ground water at the location where the monitoring well is installed; neither the well cuttings, nor the purge water would be regulated as a solid waste. An example of a situation where the site manager might use knowledge to determine proper disposition (i.e., testing would not be required) would involve materials generated at locations where wells are installed for the purpose of ascertaining naturally occurring levels of

inorganic constituents and there is no basis to expect contamination, i.e., there is no past history of hazardous waste management activities or releases in these areas. If this is the case, the soils, cuttings, purge water, etc. would not be regulated as solid wastes. Test results or knowledge of the waste should be used to screen the well cuttings, purge water and other IDW to demonstrate that concentrations of contaminants are below or equal to background levels.

Purge water, well cuttings from monitoring wells, and other IDW, if tested, must be done so in accordance with EPA SW-846, Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, 3rd edition, 1986, as updated. If contaminant levels are found to be above background levels, the IDW would be considered a solid waste. Should test results further indicate that the IDW contains a listed hazardous waste, or if the IDW exhibits a characteristic of hazardous waste, the IDW is a hazardous waste and must be managed and disposed in accordance with the VHWMR. Alternatively, contaminated IDW that contains a listed hazardous waste must be managed as a hazardous waste until it no longer "contains" the hazardous waste, i.e., until the constituent levels are below site specific risk based levels. This is consistent with EPA's Contained In Policy. The DEQ should be contacted directly to determine the site specific risk based levels that would apply to IDW that contains listed hazardous waste.

If the IDW is not a hazardous waste, but contains levels of contaminants above background levels, the IDW must be managed in accordance with the VSWMR. Solid waste generated from cleanup or investigation activities is considered a special waste under Part VIII of the VSWMR. Prior to acceptance of a special waste for disposal at a solid waste management facility, the operator must obtain prior authorization from the Department. Purge water, on the other hand, must be disposed at a publicly owned treatment works (POTW) or other wastewater treatment system operating in accordance with its Virginia Pollutant Discharge Elimination System (VPDES) permit, provided that all other pertinent criteria are satisfied.

The on-site treatment, storage, or disposal of IDW must be authorized by a permit from the DEQ. A generator of hazardous IDW may accumulate such wastes in tanks or containers in accordance with VHWMR §6.4.E. Treatment of hazardous waste in tanks or containers within the 90 day accumulation period may only occur upon prior written approval from the appropriate DEQ Regional Office.

This policy may be revised or rescinded at any time as Federal and/or State regulations change.

Signed:

Hassan Vakili

Hassan Vakili, Director
Waste Operations

6-28-95
Date

ADDENDUM

Department of Environmental Quality
Waste Operations
Policy for the Handling of
Investigation Derived Waste (IDW)

This Addendum is being provided to clarify the distinction between the disposal requirements for Investigation Derived Waste (IDW) that is generated from an undefined area, and the requirements for soil and sediment IDW when soil and sediment is generated from an area of known contamination subject to further response measures with oversight from DEQ and/or EPA.

"DEQ Policy for the Handling of Investigation Derived Waste" dated July 5, 1995, specifies that IDW contaminated above background levels is considered a solid waste and must be managed in accordance with the Virginia Solid Waste Management Regulations. If the IDW contains a listed hazardous waste or exhibits a characteristic of hazardous waste, it must be managed as hazardous waste in accordance with the Virginia Hazardous Waste Management Regulations. Under this policy, returning contaminated IDW to the location from which it is generated is prohibited.

However, this policy was not intended to address the requirements associated with soil and sediment IDW generated from an area of known contamination when this area is subject to future response activities with oversight from DEQ or EPA. In such a case, the management and disposal of the IDW should be in accordance with the pertinent EPA guidance governing the applicability of RCRA land disposal restrictions. ("Management of IDW During SI's", EPA/540/G-91/009).

Under the EPA guidance, replacement of soil and sediment IDW into the area of contamination from which it is generated is permissible provided that the waste is not treated prior to placement. Therefore, the above-referenced DEQ IDW policy now recognizes that if soil and sediment IDW is generated from an area of known contamination, and this area is subject to further response measures with oversight from DEQ and/or EPA, the IDW may be placed back into the area from which it is taken provided there is no treatment of this waste prior to placement.

Signed Hassan Vakili Date 7/24/96

Hassan Vakili, Director
Division of Waste Operations