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FIG. 2.1 (PROJECT TEAM ORGANIZATION CHART); AQUATIC SAMPLING SITE MAPS

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The Sauget Area 2 Sites Group appreciated the opportunity to meet with you, Illinois EPA, U.S. Fish & Wildlife, and CH2M Hill representatives during our two-day meeting on April 19th and 20th. During this meeting we discussed and resolved the major issues and questions collectively raised by the reviewing agencies. The attached, revised documents detail the increased numbers of samples and analysis agreed upon during this meeting, as well as supplementing the review of historical investigations and other miscellaneous changes. We believe the comments were either resolved in our recent two-day meeting, or addressed in the attached revision.

Copies of these document are also being sent to the following individuals in a similar manner:

One Copy
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If you have any questions on the attached, please call.

Sincerely,

Steven D. Smith
Project Coordinator - SA2SG

cc: SA2SG Members

VOLUME 3A

SURFACE WATER, SEDIMENT AND AQUATIC BIOTA FIELD SAMPLING PLAN

SAUGET AREA 2 SITES
SAUGET, ILLINOIS

[USEPA Docket Number R8H-5-00-003]

May 25, 2001

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1.0 ECOLOGICAL ASSESSMENT FIELD SAMPLING PLAN

This document constitutes the Field Sampling Plan (FSP) for the Ecological Risk Assessment. It describes the conceptual approach for a Reconnaissance Survey and how data from the Mississippi River, to be used in the Ecological Risk Assessment (ERA), will be collected during the Main Sampling Event. It also describes planned stations for collection of surface water, sediment, and fish samples, collection procedures, number of samples to be collected, container and preservation requirements, and holding times. In this document, the terms "station" and "location" are used interchangeably to describe the discrete points at which samples will be collected. Further field documentation procedures required for the FSP, including labeling and chain-of-custody requirements, are included in Section 5 of the QAPP. The FSP procedures to collect fish fillets supports the Human Health Risk Assessment for ingestion of fish as a potential exposure pathway. Field sampling personnel will follow the procedures documented in this FSP, Section 5.0 of the QAPP, the associated field sampling SOPs included in Appendix A of the QAPP, and the Ecological Risk Assessment Health and Safety Plan (HASP), attached as Volume 3C. The field sampling procedures may be refined as a result of the Reconnaissance Survey.

1.1 STUDY AREA

The Site encompasses approximately 344 acres situated adjacent to the Mississippi River. The Site, found within the villages of Sauget and Cahokia, Illinois, is roughly bounded to the west by the Mississippi River, to the north by the MacArthur bridge railroad tracks, to the east by Illinois State Highway 3, and to the south by Cargill Road. The Site fronts approximately 8,000 feet of the Mississippi River.

The Sauget Area 2 Site includes five former disposal areas, Sites O, P, Q, R and S, adjacent, or in close proximity, to the Mississippi River. These five disposal areas were given letter designations by the Illinois Environmental Protection Agency (IEPA) in the 1980s. Two of these sites, Sites Q and R, are located on the wet side of the floodwall and levee which is operated and maintained by the US Corps of Engineers and the Metro East Sanitary District. The floodwall is designed to protect the City of East St. Louis and the Villages of Sauget and Cahokia from flooding. Sites O, P and S are located on the dry side of the floodwall and levee.

Site O is located on Mobile Avenue in Sauget and occupies approximately 20 acres northeast of the American Bottoms Regional Wastewater Treatment Facility (ABRTF). Site P is located between the Illinois Central Gulf Railroad and the Terminal Railroad and is north of Monsanto Avenue in the Village of Sauget. Site P occupies approximately 20 acres of land. Site Q occupies approximately 90 acres and is bordered by the Sauget Site R and the old Union Electric Power Plant on the north, the Illinois Central Gulf Railroad and the U.S. Corps of Engineers flood control levee on the east, and the Mississippi River on the west. The two ponds created by the borrow pit operations are located at the southern end of Site Q. Site R is located adjacent to the Mississippi and has had a temporary cap placed on it. Site S is a small drum disposal area west-southwest of Site O.

The ecological assessment will focus on portions of the Mississippi River downgradient of Areas O, P, Q, R, and S, shown in Figure 1-1. The aquatic sampling study area includes the near-shore portion of the Mississippi River where facility groundwater may be discharging to surface water, referred to here as "plume discharge area" (PDA) and sites upstream of the discharge area (UDA) and downstream of the discharge area (DDA). The UDA and DDA sites have not yet been identified (see Section 1.2.1 below) and as such are not shown on the figure. The plume discharge area is defined based on the terrestrial boundaries of Sites P through Q. A description of sampling sites and site photos will be provided after the reconnaissance survey described in Section 1.2.1.

1.2 FIELD SAMPLING RATIONALE AND SAMPLING LOCATIONS

The Ecological Risk Assessment FSP consists of two separate sampling field events: a Reconnaissance Survey and the Main Sampling Event.

1.2.1 Reconnaissance Survey Objectives

A Reconnaissance Survey (Survey) will be conducted to evaluate physical conditions at the planned sampling locations and to refine the Field Sampling Program. The field observations made during the Survey are used to provide a verbal and photographic description of the site, finalize sampling locations, procedures, and determine the number of aquatic samples that can be realistically collected during the main sampling event. The objectives and justification for the Survey activities are described below:

- **Photodocument Areas O, P, Q, R, and S**

- **Locate aquatic sampling locations** for the collection of water, sediment, and biota samples during the Main Sampling Event. This will be done by collecting a series of sediment samples for the analysis of volatile organic compounds (VOC) (by method 8260B), and by making qualitative observations of the sediments including the presence of any benthic invertebrates and grain size. Sediment samples will be collected using a ponar sampler near the bank of the Mississippi River, from the northernmost end of site P to the southernmost end of site Q. Samples will be collected every 500 ft (a total of approximately 28 samples) on a line approximately 50 ft from the bank.

- **Select two upstream / downstream sites** for comparison of ecological impact in the plume discharge area to areas where no facility groundwater discharge occurs. These sites will include an Upstream (UDA) and Downstream (DDA) site, with nine sampling stations within each site. One set of samples will be collected at each of these stations during the Main Sampling Event. The UDA sampling stations will be selected, to the extent practicable, in areas of similar grain size and habitat as the sampling sites adjacent to areas O, P, Q, R, and S. The UDA sites will be located as far north as River mile 190 (and will not be north of Lock and Dam 26). The DDA stations will be supported by a review of river currents and bathymetric data maintained by the U.S. Army Corps of Engineers and other agencies to verify the downstream samples can all be located along the eastern shore of the Mississippi River. To the extent practicable, DDA stations will be selected in depositional environments that have potentially been impacted by erosion and subsequent deposition of sediments from the plume discharge area. A minimum of four downstream samples will be located in depositional areas. These sampling stations will be investigated for site-related impacts during the Main Sampling Event. Overall, the upstream / downstream sites should be reasonably comparable to the plume discharge area in terms of habitat and sediment characteristics. These characteristics will be limited to those that can be visually observed in the field (e.g. adjacent stream bank condition, depth of water, approximate velocity, sediment grain size, color, odor, presence of organic matter, and presence of benthic macroinvertebrates).

- **Finalize the list of the selected representative receptor species** that are using the Mississippi River habitats and represent assessment endpoints for the baseline ERA.

- **Conduct a fish habitat evaluation** for the planned sampling locations in the Mississippi

River. The purpose of this evaluation will be to determine if these portions of the Mississippi River support the types of fish that are planned for collection to represent different trophic levels for the ERA and game fish (for ingestion pathway) for the human health risk assessment. This evaluation will include discussions with a professional fisherman and an aquatic scientist familiar with the river.

- **Perform a habitat assessment for benthic invertebrates** by documenting the physical and environmental conditions for the planned ERA sampling locations, including the upstream / downstream sites. Observations of the bottom substrate, available cover, estimation of embeddedness, estimation of the flow or velocity and depth regime, channel morphology, and riparian and bank structure (such as bank stability, bank vegetation, and streamside cover) will be documented to evaluate the benthic habitat.

- **Determine the most appropriate sampling techniques** for water, sediment and fish based on Survey observations. Due to the expected swift current of the Mississippi River, and the anticipated coarse texture of the sediments, different sampling techniques will be evaluated to ensure that the Main Sampling Event will generate representative samples for use in the ERA.

- **Evaluate the need for modifications of analytical preparation methods** based on visual observations of the "wetness" (percent moisture) of the sediments to evaluate the need for freeze-drying techniques to increase the solids content as a means to achieve adequate chemical detection levels.

After the reconnaissance survey is completed, a technical memorandum, summarizing the results of the survey will be prepared and submitted to US EPA. This memo will summarize the chemical results of screening sample analysis, present observations made during the reconnaissance survey, and confirm the sampling locations to collect aquatic and terrestrial samples during the main sampling event. Relevant information from the Survey will be incorporated into the final planning of the Main Sampling Event.

1.2.2 Main Sampling Objectives

The Main Sampling Event, which is scheduled for July-August 2001, will consist of collection of surface water, sediment and fish samples from the Mississippi River and analyses of

these samples to determine the concentration of site-related constituents in these three media and assess their impact, if any. The sampling locations and rationale are provided in the next section (1.2.3). The sampling activities will consist of the following collections:

- **Sediment samples for chemical and physical analyses**: at 45 locations (nine sampling stations at each of the three plume discharge area sites and two upstream / downstream sites) for use in the ERA as part of the sediment triad approach.

- **Sediment samples for toxicity testing**: at 45 locations (nine sampling stations at each of the three plume discharge area sites and two upstream / downstream sites) for chronic toxicity tests and bioaccumulation tests for use in the ERA and as part of the sediment triad approach. Chronic toxicity testing will use two indicator species, *Hyalella azteca* and *Chironomus tentans*; a total of 90 sediment toxicity tests will be performed (two species x 45 locations). Also, for two stations for each toxicity test species, the sediment will be examined for VOC concentrations at the beginning and the end of the bioassay to assess VOC concentration changes over the bioassay period.

- **Sediment samples for bioaccumulation testing**: at 15 locations (on sediment samples collected at the center sampling station on each transect in each of the three plume discharge area sites and two upstream / downstream sites) for bioaccumulation tests for use in the ERA and as part of the sediment triad approach. The bioaccumulation test will use *Corbicula fulminea* (Asiatic clam) to assess the potential for contaminant bioaccumulation in the tissue of sediment-dwelling organisms; a total of 15 bioaccumulation tests will be performed (15 locations).

- **Benthic invertebrates samples**: at each of the 9 of the upstream and 9 downstream sampling sites, and at each of the 27 site-related sampling sites (9 sampling sites within each of the three sampling sites) = 45 samples for analysis of community structure (species identification to the lowest practical taxon, species richness, and biomass) for use in the ERA and as part of the sediment triad approach.

- **Surface water samples for chemical analyses**: at 45 locations (nine sampling stations at each of the three plume discharge area sites and two upstream / downstream sites) for chemical analysis for use in the ERA. In addition to the unfiltered surface water samples collected, a filtered surface water sample will be collected at each sampling

location for trace metals analysis. Filtered sample results are required in order to compare to ambient water quality criteria.

- **Surface water samples for toxicity**: at 45 locations for chronic surface water toxicity (bioassay) tests using two indicator species, the cladoceran *Ceriodaphnia dubia* and the fathead minnow, *Pimephales promeles,* for use in the ERA. A total of 90 surface water toxicity tests will be performed (two species x 45 locations).

- **Fish samples for chemical analysis**: at three locations within the plume discharge area and at upstream and downstream sites for three trophic levels (bottom feeder, forager, and predator) for use in the ERA and for predator/game fish for use in the Human Health Risk Assessment. The weight, length, and width of each fish will be recorded. The stomach contents of a representative subset will be observed. The general health (and any visual anomalies) will be assessed by a qualified aquatic biologist.

- **Field observations** of the fish community and their habitat for use in the ERA, as necessary.

- **QC samples**: including field duplicates, field equipment rinsate blanks, trip blanks, and additional sample volumes for matrix QC of matrix spike/matrix spike duplicates at the frequencies defined in Section 3 of the QAPP and detailed, per media and parameter type, in Tables 1-1 through 1-6 at the end of this FSP.

1.2.3 Sampling Locations

Surface water, sediment, and fish samples will be collected along three transects running parallel to the river bank at five stations: 1) downgradient of Site P, 2) downgradient of Sites O, R, S and the northern end of Site Q, 3) downgradient of the southern end of Site Q, 4) a reference site upstream of Sauget Area 2, and 5) a site downstream of Sauget Area 2 (upstream and downstream sampling sites are to be selected based on results of the reconnaissance survey). Transects will be located 50, 150 and 300 feet from shore. Each transect will consist of three sampling stations; thus, there are nine sampling locations for each of the five sites listed above (a total of 45 sampling locations). The general locations of PDA transects are shown on Figure 1-1 and generalized sampling locations within each sample site are shown in Figure 1-2. Actual sampling sites will be finalized based on visual

observations, sediment physical characteristics, and VOC analysis of sediment samples collected during the reconnaissance survey. Coordinates for all final sampling locations will be established in the field using a differential Geographical Positioning System (GPS) as well as by line of sight.

Reconnaissance survey analytical data, collected as part of the May 2000 W.G. Krummrich Accelerated Corrective Action AOC, indicate that these sampling transects will be located in the likely plume discharge area. During this survey, which was conducted in September 2000 by Menzie-Cura & Associates, sediment samples were collected in the Mississippi River downgradient of the Krummrich plant along three transects running from the bank toward center of the river. Analytical results from the reconnaissance survey are summarized below:

	Distance from Bank, feet								
Total VOCs, ppb	50	200	300	400	500	600	700	1000	1400
North Transect	644	NS	854	ND	ND	NS	ND	ND	ND
Center Transect	1300	ND	NS	NS	ND	NS	NS	NS	NS
South Transect	45	NS	473	NS	NS	1	NS	NS	NS

Surface water, sediment, and fish samples collected during the main sampling event will be analyzed for chemicals of concern (target analytes) listed in Tables 1-1 through 1-7 as presented in Section 1.0 of the QAPP. Specific numbers of samples, types of samples to be collected, container specifications, etc. are described below. Analyses of chemicals in fish fillets will be used in the Human Health Risk Assessment to evaluate human exposure due to ingestion. Fillets of buffalo will be analyzed and used in human health risk assessments. Whole bodies of Channel Catfish will be analyzed and used in ERA evaluation of risks to Piscivorous animals. Benthic community structure will be evaluated to provide data for sediment triad evaluation. Bioassays will be conducted on surface water and sediment samples to determine the toxicity, if any, of these environmental media to sensitive organisms.

Fish will be sampled in three sites in the Mississippi River: 1) downgradient of Site P, 2) downgradient of Sites O, R, S and the northern end of Site Q, and 3) the southern end of Site Q. A food source approach will be used to select fish for fish tissue analysis:

Primary Food Source	Fish	Trophic Level	Endpoint Organism
Detritus, benthic invertebrates, piscines	Channel Catfish	Bottom Feeder / Predator	Piscivorous animals
Plankton	Shad (Large)	Forager	Osprey
Plankton	Shad (Small)	Forager	Heron
Aquatic insects, piscines	Buffalo	Bottom Feeder / Gamefish	Recreational Fisher

These fish tissue samples, collected in the plume discharge area, will be used to determine the impact, if any, of groundwater discharge on higher trophic level organisms. Fish will also be sampled from sites upstream and downstream of Sauget Area 2.

Information collected as part of the Surface Water, Sediment and Aquatic Biota Sampling Plan will be used in the Human Health Risk Assessment and the Ecological Risk Assessment. With five disposal sites located adjacent to or near the east bank of the Mississippi River, the primary ecological exposure pathway is groundwater discharge to surface water. Other exposure pathways include terrestrial organism exposure to site soils and aquatic organism exposure to water and sediments in on-site ponded areas. These exposure pathways will be included in the conceptual site model section of the Ecological Risk Assessment Workplan. Additional exposure pathways include surface runoff from the site to the Mississippi River and seeps along the riverbanks. Surface runoff sampling will be conducted by URS as part of the Remedial Investigation, and is described in a separate document.

All of the above sampling and analysis activities will be performed under the direction of AMEC field team leader. Samples to be collected from the various media and the chemical, physical, and toxicity analyses to be performed in support of the Main Sampling Event for Sauget Area 2 investigations are summarized in Table 1-1. Analytical methods to be used for analyses are presented in Section 7 of the QAPP.

1.3 SURFACE WATER SAMPLING

1.3.1 Water Sample Collection

Surface water samples will be collected at the 45 sampling locations identified in Figure 1-1 and Figure 1-2. Surface water samples will be collected at the sediment-water interface (within one foot of the bottom) at all stations. Surface water samples will be collected using the standard operating procedure, *Surface Water Sampling* (Appendix A). Note that this SOP may be modified after the reconnaissance survey and during actual sampling based on logistics of surface water sampling in the Mississippi River. Because of the volumes required and sampling limitations due to the fast currents, a peristaltic pump system will be utilized. Decontaminated tubing will be used at each location. A decontaminated Niskin sampler may also be utilized to collect water sample at depth. All chemical analysis in surface water samples will be conducted in unfiltered samples. For trace metals analysis, in addition to unfiltered samples, a filtered water sample will also be collected using disposable 0.45 um filters connected in-line to the pump.

In all cases, the performance criteria for sampling are to collect water samples in a manner that prevents contamination from the sediments, minimizes the potential for contamination by the sampling system, and provides a representative sample of the water column above the sediments. Potential contamination of surface water samples by sediments will also be minimized by either conducting all surface water sampling prior to the sediment sampling event or by performing surface water sampling prior to any sediment sampling at a location (if the sampling events are combined). Surface water samples will be collected in the following locations:

Site P - Surface water samples will be collected along three transects running parallel to the bank from the north to south end of Site P. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One surface water sample will be collected at each sampling station and analyzed for hardness, metals (both dissolved and total), VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Surface water samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 1-2) will be analyzed for dioxins. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Ceriodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

Sites O, R, S and Northern End of Site Q - Surface water samples will be collected along three transects running parallel to the bank from the north to south end of Site R. Sites O and S and the northern end of Site Q are located upgradient of Site R. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One surface water sample will be collected at each sampling station and analyzed for hardness, metals (both dissolved and total), VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Surface water samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 1-2) will be analyzed for dioxins. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Ceriodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

Southern End of Site Q - Surface water samples will be collected along three transects running parallel to the bank from the south end of Site R to the south end of Site Q. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One surface water sample will be collected at each sampling station and analyzed for hardness, metals (both dissolved and total), VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Surface water samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 1-2) will be analyzed for dioxins. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Ceriodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

Upstream Reference Site - Surface water samples will be collected at nine locations upstream of Sauget Area 2. Sampling locations will be selected during a reconnaissance survey conducted prior to sample collection.

One surface water sample will be collected at each sampling station and analyzed for hardness, metals (both dissolved and total), VOCs, SVOCs, dioxins, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Cerodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

Downstream Sampling Site - Surface water and sediment samples will be collected at nine locations in the Mississippi River downstream of Sauget Area 2. Sampling locations will be selected during a reconnaissance survey conducted one month prior to sample collection.

One surface water sample will be collected at each sampling station and analyzed for metals (both dissolved and total), VOCs, SVOCs, dioxins, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Cerodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

1.3.2 Water Sample Analytes, Containers, and Shipment Requirements

All surface water samples will be analyzed for the target compounds in the following categories: hardness, metals, VOCs, SVOCs, pesticides, herbicides, dioxin and PCBs. In addition, field measurements of temperature, pH, dissolved oxygen, and conductivity and

laboratory measurements during bioassays will be conducted to characterize the water quality, as described in Section 1.3.3, below. In addition to collecting an unfiltered water sample, water samples collected for trace metals analysis will be filtered in the field using a disposable 0.45 um filter. The VOC samples will be collected by directly filling 40-mL VOC vials from the peristaltic pump tubing to minimize VOC loss and avoid preservative loss. Table 1-2 summarizes the number of samples, sample container types, preservation, and holding time requirements for all compounds in surface water samples. Field duplicates, field blanks and MS/MSD samples for QC will be collected at frequencies as described in Section 3 of this document. QC samples are also listed in Table 1-1 as a summary of all the types of samples planned for collection and analysis.

All surface water samples will be appropriately labeled with location, date, sampler's initials, test type and preservative and stored on ice, maintained at 4°C and delivered to the appropriate laboratory within 24 hours of collection via FedEX or similar overnight carrier for next day delivery. The chain-of-custody (COC) documentation will accompany all samples from the field to the laboratory (see Section 5 of this QAPP).

1.3.3 Supporting Measurements for Surface Water Quality

At each location, the following water quality parameters will be measured in the field: turbidity, dissolved oxygen, specific conductivity, temperature, and pH. Measurements will be made at the surface (top), middle, and bottom portion of the water column, consistent with the surface water sample collection protocols. Depending on water flow conditions, water quality parameter measurement may be restricted to surface waters only. Water quality measurements will be made using a Horiba U-10 meter for turbidity, dissolved oxygen (DO), specific conductivity, temperature, and pH. Calibration of field equipment will be performed as described in the field measurement SOP included in Appendix A of the QAPP.

1.4 SEDIMENT SAMPLING

1.4.1 Sediment Sample Collection

Sediment samples will be collected at each of the 45 sediment locations using a Van Veen grab sampler (3.2 L or 11.1 L). The equipment is deployed from a davit along the side of the boat and collected using winch assistance. Prior to sampling at a location, the grabs and all other sampling devices such as spoons are decontaminated in accordance with the: SOP,

Equipment Decontamination (Appendix A). Table 1-3 lists the number of sediment samples to be collected, container and preservation specifications and holding time requirements for the chemical analysis to be performed on the sediment samples. Table 1-4 and 1-5 list the number of sediment samples, container and preservation specifications and holding time requirements for the benthic community evaluation and bioassays, respectively, to be performed in support of the ERA.

The goal of the sampling program is to collect representative sediment from the upper few inches (5-6 cm). This depth interval was selected as the zone most relevant to exposures of ecological receptors. The sediments will be collected and analyzed for the chemicals and sediment parameters presented in Tables 1-1 through 1-7 of the QAPP. Several grab samples may need to be collected at each location to provide sufficient sample volume for all the parameters to be tested including the target compound chemical analyses (metals, VOCs, SVOCs, pesticides, herbicides, dioxin and PCBs), the non-target analysis of pH, total organic carbon (TOC) and grain size, collection of benthic invertebrates for community evaluation, and for the sediment toxicity (bioassay) tests. Section 1.5 discusses collection of benthic invertebrate samples and Section 1.6 discusses collection of samples for bioassay toxicity testing.

Samples for VOCs will be obtained from the first grab. All other samples will be drawn from a composite made up of the upper few inches of sediment from additional grab samples. The VOC sample will be collected using a 5 mL modified syringe/plunger. The plunger tube will be inserted into the sediment below the surface and removed with care taken to prevent loss. The plunger will then be utilized to push the sample into the bottle. This method is the only feasible method for collecting samples in sediments that are very coarse-grained. These samples will be released into 1) laboratory water preservative for low-level analysis, 2) methanol preservative for high-level analysis. The number of sediment samples to be collected for VOC analysis, container specifications, preservation, and holding time requirements are listed in Table 1-3.

For all other parameters, the sediment (~5-6 cm) will be removed from the grab and homogenized in a stainless steel bowl using a stainless steel spoon or scoop. Sufficient sediment will be collected and mixed in the bowl so that sub-samples can be taken for chemical analysis of SVOCs, pesticides, herbicides, metals, dioxins, PCBs and TOC, physical analysis of grain size distribution, and sediment toxicity (bioassay) testing. Each of

these sub-samp1es will be taken using either a stainless steel spoon and placed in the appropriate container (see Section 1.4.2, below).

Site P - Sediment samples will be collected along three transects running parallel to the bank from the north to south end of Site P. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.
One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Sediment samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 1-2) will be analyzed for dioxins. Bioassays, using the amphipod *Hyallela azteca* and the midge larvae *Chironomus tentans*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

Sites O, R, S and Northern End of Site Q – Sediment samples will be collected in the Mississippi River along three transects running parallel to the bank from the north to south end of Site R. Sites O and S and the northern end of Site Q are located upgradient of Site R. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Sediment samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 1-2) will be analyzed for dioxins. Bioassays, using *Hyalella* and *Chironomus*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

Southern End of Site Q - Sediment samples will be collected in the Mississippi River along three transects running parallel to the bank from the south end of Site R to the south end of Site Q. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Sediment samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 1-2) will be analyzed for dioxins. Bioassays, using *Hyalella* and *Chironomus*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

Upstream Reference Site - Sediment samples will be collected at nine locations in the Mississippi River upstream of Sauget Area 2. Sampling locations will be selected during a reconnaissance survey conducted one month prior to sample collection. One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, dioxins, metals, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Bioassays, using *Hyalella* and *Chironomus*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

Downstream Sampling Site - Sediment samples will be collected at nine locations in the Mississippi River downstream of Sauget Area 2. Sampling locations will be selected during a reconnaissance survey conducted one month prior to sample collection. One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, dioxins, metals, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Bioassays, using *Hyalella* and *Chironomus*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

1.4.2 Sediment Sample Analytes, Containers, and Shipment Requirements

Required sample sizes are listed in Table 1-3 along with sample container types, preservation, and holding times. Sediment samples will be analyzed for VOCs, SVOCs, metals, pesticides, herbicides, dioxin, PCBs, TOC, pH, and grain size using methods listed in Section 7 of the QAPP. Field QC samples, including field duplicates, field rinsate blanks and trip blanks, and matrix QC, described in Section 3 of this QAPP will be collected at the frequencies summarized in Table 1-1.

Each sample container will be labeled to identify sample location, analysis required and preservation, date, and the initials of the collector. All sediment samples will be kept on ice within coolers maintained at 4°C and delivered to the appropriate laboratory within 24 hrs of collection via FedEx or similar overnight carrier. The chain-of-custody (COC) documentation will accompany all samples from the field to the laboratory (see Section 5 of the QAPP).

1.5 Benthic Invertebrate Sample Collection

Benthic invertebrates will be collected to evaluate the composition and abundance of the benthic community as part of the triad approach in the ERA. Information on the composition and abundance of benthic invertebrates will be used to evaluate potential effects of target compounds on the benthic invertebrate community. The data will be analyzed for taxa richness, abundance, percent dominant taxon/taxa, and community composition (see Section 7 of QAPP). Table 1-1 summarizes the number of samples to be collected at each location and Table 1-4 specifies the numbers of samples from each location, container specification, preservation, and holding time requirements for the benthic invertebrate samples for use in support of the ERA.

1.5.1 Benthic Invertebrate Collection for Community Evaluation

One sediment grab sample will be collected at each of the nine sampling stations in each site (three sites in the PDA; one UDA and one DDA). This will yield a total of 45 benthic invertebrate samples for community evaluation. Community analyses will be done for species identification to the lowest practical level taxon, species richness, and biomass.

Sediment samples will be collected with a Van Veen or similar large volume grab sampler using techniques described in the standard operating procedure, *Sediment Sampling*

(Appendix A of the QAPP). Benthic community samples will be taken after sub-sampling for VOCs and prior to homogenization for sediment chemistry sub-sampling. Samples will be labeled using the sample identification scheme in associated site documents. Sample locations, time and date of collection, and initials of the collector will be on each sample label and the chain of custody form. This information will also be documented in a field notebook or log sheet. The samples will be preserved with 70% isopropyl alcohol and transported to the laboratory for analysis.

1.5.2 Assessment of Habitat Conditions at Benthic Sampling Location

Habitat conditions at each of the 45 sample stations (nine sample stations at each of the five sites) will be evaluated in the field and by several of the analytical measurements made in the laboratory. Qualitative field observations of sediment type, water depth, river flow velocity, width of water body, shoreline characteristics, and bordering vegetation will be recorded in a field notebook or log sheet. These observations for the Habitat Assessment will be guided by the *Physical Characterization/Water Quality Field Data Sheet and the Habitat Assessment Field Data Sheet, USEPA Rapid Bioassessment Protocols,* 1999 (Forms 1 and 2, respectively, in Appendix A-4 of the QAPP).

1.6 Bioassay Toxicity Tests

1.6.1 Surface Water Toxicity Tests

Samples for the surface water toxicity (bioassay) tests will be taken from all 9 sample stations at each of the five sites (Tables 1-1 and 1-5). At each of the 45 locations, 2.5 gallons of water (at depth) will be collected and placed into plastic bottles, labeled, and placed on ice in a cooler. Samples will be labeled using the sample identification scheme in associated Site documents. Sample locations, time and date of collection, and initials of the collector will be on each sample label and the chain of custody form which will accompany each shipment to the laboratory. Samples will be provided to the laboratory within 24 hours of collection via FedEx or similar overnight carrier. At the laboratory, the surface water samples for toxicity testing will be refrigerated to 4° C and protected from light prior to use in testing to maintain the integrity of the original sample (Table 1-5).

The surface water toxicity tests will be used to evaluate whether chemicals in surface water within the plume discharge area, upstream of this area, and downstream of this area are

toxic to benthic invertebrates. Chronic toxicity tests will be conducted at all 45 sampling stations with two indicator species, the cladoceran *Ceriodaphnia dubia* and the fathead minnow *Pimephales promeles*. The laboratory will monitor the samples and if after acute time periods 50% or more of the animals survive, chronic testing will continue for these two species; chronic testing will not continue if >50% mortality is observed. This eliminates the need to set up and run long term tests for water in which the organisms cannot survive. The acute and chronic tests are summarized in Table 1-5 and the test methods are described in Section 7 and Appendix C of the QAPP.

1.6.2 Sediment Toxicity Tests

Samples for the sediment toxicity (bioassay) tests will be taken from the same homogenized composite sediment sample that is collected from all nine sample stations at each of the five sites (Tables 1-1 and 1-5) for chemical and physical analyses. This homogenized sample will consist of the upper few inches of sediment from several grabs. A clean scoop will be used to transfer the sediment sample from the mixing bowl to the sample container. Unrepresentative material *(e.g., stones, wood chips)* will be removed from the sample at the discretion of the field sampler and will be documented in the field log.

Approximately one gallon of sediment will be collected from each of the 45 sample stations and placed into plastic bottles, labeled, and placed on ice in a cooler. The one gallon sample will be sufficient for the bioassays for both indicator species. Samples will be labeled using the sample identification scheme in associated Site documents. Sample locations, time and date of collection, and initials of the collector will be on each sample label and the chain of custody form which will accompany each shipment to the laboratory. Samples will be provided to the laboratory within 24 hours of collection via FedEx or similar overnight carrier. At the laboratory, sediment samples for toxicity testing will be refrigerated to 4° C and protected from light prior to use in testing to maintain the integrity of the original sediment (Table 1-5).

The sediment toxicity tests will be used to evaluate whether chemicals in sediments within the plume discharge area, upstream of this area, and downstream of this area are toxic to benthic invertebrates. Chronic toxicity tests will be conducted at all 45 sampling stations with two indicator species, the amphipod *Hyalella azteca* and the midge larvae *Chironomus tentans*. The sediment toxicity tests are summarized in Table 1-5 and the test methods are

described in Section 7 and Appendix C of the QAPP. In order to monitor VOC concentration changes during the test periods, sediment samples will be collected by the laboratory following the aforementioned VOC collection SOP at the beginning and the end of the bioassay for a subset of two *Hyallela* and two *Chironomus* tests.

1.6.3 Sediment Bioaccumulation Test

Samples for the sediment bioaccumulation test will be taken from the same homogenized composite sediment sample (described above) that is collected from the center sampling station on all three transects at each of the five sites (i.e., 15 samples; Tables 1-1 and 1-5) for chemical and physical analyses. Approximately one gallon of sediment will be collected from each of the 15 center sample stations and placed into plastic bottles, labeled, and placed on ice in a cooler. Samples will be labeled using the sample identification scheme in associated Site documents. Sample locations, time and date of collection, and initials of the collector will be on each sample label and the chain-of-custody form which will accompany each shipment to the laboratory. Samples will be provided to the laboratory within 24 hours of collection via FedEx or similar overnight carrier. At the laboratory, sediment samples for bioaccumulation testing will be refrigerated to 4° C and protected from light prior to use in testing to maintain the integrity of the original sediment (Table 1-5).

The bioaccumulation test provides an estimate of contaminant uptake by benthic fauna, providing information on the potential for trophic transfer of bioaccumulative contaminants to higher organisms. The bioaccumulative contaminants which will be analyzed in the test organisms include PCBs, chlorinated pesticides, and dioxins. The bioaccumulation test involves exposing the test organism, *Corbicula fulminea*, for a 28-day period.

1.7 Fish Sample Collection

Fish sampling will be performed at the three sites in the plume discharge area as well as the sites located upstream and downstream of plume discharge. The objectives of the fish sampling program are summarized below:

- Identify the composition and general abundance of fish in the five sites to determine if the plume discharge area is depauperate in species composition or relative abundance in comparison to the upstream and downstream locations. The upstream location may be used as a reference site;

- Examine the age structure and the weight and length relationships for species in the five locations; examine the overall health of the fish in the plume discharge area and upstream / downstream sites;
- Evaluate the habitat quality of the sampling locations with respect to supporting different fish species;
- Determine the potential of the plume discharge area for supporting recreational (game) fishing;
- Analyze the gut contents of fish in different trophic levels to document food sources of the fish species;
- Measure body burdens of chemicals in fish tissues of different trophic levels and species, for use in the ecological and human health risk assessments; and
- Examine fish for gross histopathological anomalies and compare these between the plume discharge area and upstream and downstream locations.

1.7.1 Habitat Assessment

Field assessments of the fish species and habitat present at each of the five sample sites will be made during both the Reconnaissance Event and the Main Sampling Event. General field observations made during the Reconnaissance Event will be used to finalize sample locations and methods during the Main Sampling Event.

Habitat assessment will be performed by the Ecological Project Manager and Field Team Leader in cooperation with a qualified aquatic ecologist or fisheries biologist. These field observations of habitat and fish will be conducted in accordance with the *Fish Collection* and *Collection and Treatment of Fish Field Data* standard operating procedures and using the *Fish Sampling Field Data Sheet* provided in Appendix A of the QAPP. This assessment will be qualitative and based on a combination of experience in similar water bodies, a qualitative assessment of the physical and water quality conditions provided by the habitat, and qualitative assessment of recreational fishing potential. Information pertinent to fish populations includes: water depth, physical sediment characteristics, type and extent of submerged and emergent vegetation, area, nature of contiguous aquatic environments, general water quality conditions (turbidity, DO, conductivity, pH, and temperature), and characteristics of the shoreline. These will be documented in the field by recording observations in log books or field data sheets and by taking photographs.

The evaluation of whether the plume discharge area can support recreational fishing will be based on the results of the fish collection and discussions with commercial fishermen and qualified scientists. For example, successful collection of adult edible game fish species such as catfish would be an indication that the sampling locations contain fish that are sought for recreational fishing.

1.7.2 Fish Collection for Tissue Analysis

To support the ERA, fish exposed through different feeding strategies (trophic levels) will be collected. In addition, game fish will be collected to support the human health risk assessment pathway of ingestion (fillets). The categories will include fish from three trophic levels (bottom feeder, forager, and predator) and a game fish, which may be an equivalent species to the predator fish. Bottom fish, such as channel catfish, are in the most intimate contact with sediments and are therefore useful species to examine potential effects of sediments. Forager fish may also be impacted by sediments and bioaccumulation effects of chemicals. Predators and game fish are those most likely to be eaten by humans and wildlife species. Some species of fish found in the Mississippi River may represent more than one category of fish. For example, the buffalo fish represents a bottom feeder fish and might also represent a game fish for human health consumption. Fish will be collected to represent components of the food chain as follows:

Trophic Level	Target Species (size)	End Point Organism
Bottom feeder / Omnivore	Channel catfish (10-12")	Piscivorous animals
Small forage fish	Small shad (4-8")	Heron
Large forage fish	Large shad (>8")	Osprey
Gamefish / Bottom feeder	Buffalo (10-12")	Recreational fisher

The fish samples of the bottom feeder and forage fishes will be composite samples consisting of the whole bodies of three to five fish. Tissue samples from the game fish will be composites of fillets of the edible portions of one to two fish. The goal of the sampling effort is to collect three composite samples from each target group at each of the five sample sites. Each of these composite samples will undergo tissue analysis for metals, SVOCs, pesticides, herbicides, and PCBs content. One composite sample from each site will be

analyzed for dioxin content. The remaining composite samples will undergo tissue analysis for QA/QC analysis (MS/MSD and QC Field Duplicates)

This fish collection and analysis scheme is tabulated at the end of this section in Tables 1-1 and 1-6. It is recognized that this goal may be difficult to meet because some of the trophic categories may not be present or fewer than three fish may be collected. In this case, the effort will focus on those fish that are collected within each of the target categories. Substitutions may be made for the expected species in each category, as necessary. The final decision on which fish to analyze will be made after the collections are complete.

Based on expected swift currents in the Mississippi River sampling locations, fish will be collected using either hoop nets (standard commercial fishing gear for the river) or other techniques used by local fishermen. Once fish are identified, length, weight, sex, species, and any gross abnormalities (e.g., lesions, tumors, and deformities) will be recorded on a fish log. Grossly deformed specimens will be photographed, preserved, labeled, and retained in a voucher collection as archived specimen. If necessary, duplicates of the voucher specimens will be collected and sent to recognized fish biology expert(s) for taxonomic confirmation. Fish samples for tissue analyses will be placed in sealable bags and stored on ice for overnight shipment via FedEx or other shipper to the analytical laboratory where they will be maintained frozen until analysis. The SOP for filleting game fish is presented in Appendix A.

Fish stomach contents will be removed from a representative subset of collected fish in each target group; this subset will include at least one fish from each trophic level from each of the five sites. The collected stomach contents will be labeled, preserved with 70% isopropyl alcohol and transported to the laboratory for analysis.

Tables 1-2 through 1-7 in the QAPP presents the target compound lists for pesticide, dioxin, and PCB in tissue and corresponding reporting limits. The lipid content of each fish composite will be determined to provide a basis for normalizing tissue concentrations for organic constituents. In addition to the field Fish Collection Log, a Sample Processing Record for Fish compositing will be completed by the laboratory prior to sample preparation and analysis. Table 1-6 lists the number of fish samples planned for collection, and container, preservation, and holding time requirements.

Table 1-1. Summary of Proposed Sampling and Analysis Program in Support of the ERA at Each of the Five Sampling Sites (Three Plume Discharge Area Sites, UDA and DDA Sites)

Sample Matrix	Field Parameters	Lab Parameters[1]	Investigated Samples	QC Field Duplicates[3]	Trip Blank[2]	QC Matrix Spike/MS Duplicates	Equipment Rinse Blank[2]	Total Number of Samples at Each Site (Excluding blanks)
Surface Water	pH, Temperature DO Conductivity Turbidity	Metals	18(9 filtered, 9 unfiltered)	2 (1 filtered and 1 unfiltered)		2	2 for each site (1 for dissolved metals; 1 for all other parameter s)	22
		VOCs	9	1	1 Per Cooler	1		11
		SVOCs	9	1		1		11
		Pesticides	9	1		1		11
		Herbicides	9	1		1		11
		PCBs	9	1		1		11
		Dioxin	PDA sites-5 each; UDA/DDA-9 each	1		1		7(each PDA site) 11(each UDA/DDA site)
		Hardness	9	1		NA		10
		Bioassay (2 Species)	18 (9 for each species)					18
Sediment	None	Metals	9	1		1	1 for each site	11
		VOCs	18 (9 high levels and 9 low levels)	2	2 per cooler (DI for low-level; Methanol for high-level)	2		22
		SVOCs	9	1		1		11
		Pesticides	9	1		1		11
		Herbicides	9	1		1		11
		PCBs	9	1		1		11
		Dioxins	PDA sites-5 each; UDA/DDA-9 each	1		1		7(each PDA site) 11(each UDA/DDA site)
		TOC	9	1	NA	NA	NA	10
		pH	9	1	NA	NA	NA	10
		Grain Size	9	1	NA	NA	NA	10
		Benthic Community Structure	9	NA	NA	NA	NA	9
		Bioaccumulation Tests	3	1	NA		NA	4
		Bioassay (2 Species)	18 (9 for each species)	1	NA		NA	19

Table 1-1. Summary of Proposed Sampling and Analysis Program in Support of the ERA at Each of the Five Sampling Sites (Three Plume Discharge Area Sites, UDA and DDA Sites) *(continued)*

Sample Matrix	Field Parameters	Lab Parameters[1]	Investigated Samples	QC Field Duplicates[3]	Trip Blank[2]	QC Matrix Spike/MS Duplicates	Equipment Rinse Blank[2]	Total Number of Samples at Each Site (Excluding blanks)
Fish Composites (Channel Catfish)	Weight, Length, Stomach contents	PCBs	3	1	NA	1	NA	5
		SVOCs	3	1	NA	1	NA	5
		Pesticides	3	1	NA	1	NA	5
		Herbicides	3	1	NA	1	NA	5
		Metals	3	1	NA	1	NA	5
		Percent Lipid	3	1	NA		NA	4
		Dioxins	1	1	NA	1	NA	3
Fish Composites (Large Shad)	Weight, Length, Stomach Contents	PCBs	3	1	NA	1	NA	5
		SVOCs	3	1	NA	1	NA	5
		Pesticides	3	1	NA	1	NA	5
		Herbicides	3	1	NA	1	NA	5
		Metals	3	1	NA	1	NA	5
		Percent Lipid	3	1	NA		NA	4
		Dioxins	1	1	NA	1	NA	3
Fish Composites (Small Shad)	Weight, Length, Stomach Contents	PCBs	3	1	NA	1	NA	5
		SVOCs	3	1	NA	1	NA	5
		Pesticides	3	1	NA	1	NA	5
		Herbicides	3	1	NA	1	NA	5
		Metals	3	1	NA	1	NA	5
		Percent Lipid	3	1	NA		NA	4
		Dioxins	1	1	NA	1	NA	3
Fish Fillets (Buffalo)	Weight, Length, Stomach contents	PCBs	3	1	NA	1	NA	5
		SVOCs	3	1	NA	1	NA	5
		Pesticides	3	1	NA	1	NA	5
		Metals	3	1	NA	1	NA	5
		Herbicides	3	1	NA	1	NA	5
		Percent Lipid	3	1	NA		NA	4
		Dioxins	1	1	NA	1	NA	3

(1) USEPA Methods for analysis are listed in Section 7 of this QAPP, analysis for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), Pesticides, Herbicides, metals, and PCBs will be performed by STL Laboratory - Savannah, Dioxins to be performed by Triangle Laboratory. Bioassays and Benthic Community Evaluations to be performed by Pennington & Associates,

(2) Field Blanks include trip blanks (TB) for VOC (water low-level TBs are preserved with HCl and high level sediment TBs preserved with methanol) and field equipment rinse blanks (EB) for all parameters.

(3) Field Duplicates and field equipment blanks for all parameters will be performed at a minimum frequency of 1 set per 10 field samples of the same media as required by USEPA Region 5, MS/MSD samples will be collected at a minimum frequency of 1 set per 20 field samples of the same media. Whole composite fish and fish fillets are considered two different media for the purposes of MS/MSD QC frequency. MS/MSD can be performed on different fish samples than indicated in this table; provided the QC frequency is compliant with QAPP criteria, NA = QC not applicable to this parameter based on data quality needs of RCRA investigation.

Table 1-2. Surface Water Samples: Number of Samples, Preservation, Container Specification and Holding Time Requirements in Support of the ERA

Parameter	Number of Samples	Sample Container(s)[1]	Preservative	Holding Time
Metals	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites – for a total of 5 sites; 9 Stations in each of the five sites; 2 samples at each station (1 filtered + 1 unfiltered) for a total of 90 Samples. 2 QC Field duplicate at each site (2 for metals) = 10 QC duplicates; 2 Equipment rinse blanks for each site = 10 Rinse Blanks 2 MS/MSD for each site = 10 MS/MSD samples Total = 90+10+10 +10 = 120 Samples	(1) 250-mL Nalgene bottles for metals for each sample	Cool, 4°C, Protected from Light HNO_3 Preservative for Metals	Extraction: within 7 days of collection Analysis: within 40 Days of extraction
Hardness SVOCs, Herbicides, Pesticides, and PCBs	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites – for a total of 5 sites; 9 Stations in each of the five sites for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates; 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5 +5 = 60 Samples	(2) 1-L glass bottles for SVOCs, Pesticides, Herbicides, and PCBs	Cool, 4°C, Protected from Light	Extraction: within 7 days of collection Analysis: within 40 Days of extraction
Dioxin	3 Sampling sites (Plume Discharge Area) – 5 Stations in each site + 2 upstream / downstream sites - 9 Stations in each site for at total of 33 samples; 1 QC Field duplicate at each site = 5 QC duplicates; 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD sample at each site = 5 MS/MSD Spike samples Total = 33+5+5 +5 = 48 Samples	(2) 1-L Amber Glass Bottles	Cool, 4°C, Protected from light	Extraction: within 30 Days of collection Analysis: within 45 Days of extraction
VOCs	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites - for a total of 5 sites; 9 Stations in each site for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates; 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5+5 = 60 Samples Plus 1 trip blank per cooler	(3) 40-mL glass vials with Teflon-lined septa	Cool, 4°C, Protected from Light, HCl to pH<2	14 days from sample collection

[1]The sample containers list for each sample and one parameter
SVOCs = Semivolatile Organic Compounds; FD = Field Duplicate Sample; VOCs = Volatile Organic Compounds
MS/MSD = Matrix Spike / Matrix Spike Duplicate lab QC; TB = Trip Blank (VOC only); PCBs = Polychlorinated Biphenyls

Table 1-3. Sediment Samples: Number of Samples, Preservation, Container Specification, and Holding Time Requirements in Support of the ERA

Parameter	Number of Samples	Sample Container(s)[1]	Preservative	Holding Time
Metals SVOCs, Herbicides, Pesticides, and PCBs	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites -- for a total of 5 sites; 9 Stations in each of the five sites for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5 +5 = 60 Samples	(1) 250-mL glass jar (1) 250-mL Nalgene container for metals	Cool, 4°C, protected from light	Extraction: within 14 days Analysis: within 40 days of extraction
Dioxin	3 Sampling sites (Plume Discharge Area) – 5 Stations in each site + 2 upstream / downstream sites - 9 Stations in each site for at total of 33 samples; 1 QC Field duplicate at each site = 5 QC duplicates; 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD sample at each site = 5 MS/MSD Spike samples Total = 33+5+5 +5 = 48 Samples	(1) 250-mL glass jar	Cool, 4°C, protected from light	Extraction: within 30 days Analysis: within 45 days of extraction
VOCs	low-level VOC: 3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites -- for a total of 5 sites; 9 Stations in each site for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC dups. 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5+5 = 60 Samples Plus 1 trip blank per cooler	(2) 40-mL VOA vials each with 5 mL reagent grade water and a Teflon-coated stir bar, each filled with 5g of sample [water must cover sample] + 125-mL glass bottle (bulk 8260B screening)	Cool, 4°C, protected from light during transit Upon receipt at the laboratory, samples frozen at < -10°C, Protected from light, until just before analysis	14 days from sample collection
	High-Level VOC: 3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites -- for a total of 5 sites; 9 Stations in each site for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC dups. 1 Equipment rinse blanks for each site = 5 RB 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5+5 = 60 Samples Plus 1 trip blank per cooler	(1) 40-mL VOA vial with 5 mL methanol filled with 5g of sample [methanol must cover sample]	Cool, 4°C, protected from light	14 days from sample collection
Total Organic Carbon (TOC)	3 Sampling sites (Plume Discharge Areas) + 2 upstream / downstream sites -- for a total of 5 sites; 9 Stations in each of the five sites for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates; Total = 45+5+ = 50 Samples	(1) 4-ounce glass jar	Cool, 4°C	28 days
Grain Size	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites -- for a total of 5 sites; 9 Stations in each of the five sites for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates; Total = 45+5+ = 50 Samples	(1) 500 mL plastic jar	Cool, 4°C	NA

[1]Number of containers for each sampling point and for each parameter
SVOCs = Semivolatile Organic Compounds
FD = Field Duplicate Sample
Duplicate lab QC; EB = Equipment Rinsate Blank
NA = Not Applicable.

VOCs = Volatile Organic Compounds
MS/MSD = Matrix Spike / Matrix Spike
TB = Trip Blank (VOC only)
PCBs = Polychlorinated Biphenyls

Table 1-4. Samples for Analysis of Benthic Community Composition and Abundance: Number of Samples, Sample Preservation, Container Specification, and Holding Time Requirements in Support of the ERA

Parameter	Number of Samples	Sample Container	Preservative	Holding Time
Benthic Invertebrates [Benthic Community Structure]	9 sampling stations at each of the 5 sample sites 1 sample from each station Total = 45 Samples	1 gallon plastic jars	70% Isopropyl alcohol [preservative must cover specimen]	NA

Note: Each benthic fauna sample will be generated from washing and sieving several sediment grabs at each location.

Table 1-5. Samples for Toxicity (Bioassay) and bioaccumulation Tests: Number of Samples, Preservation, Container Specification, and Holding Time Requirements in Support of the ERA

PARAMETER	Matrix	Number of Samples	Sample Container(s)	Preservative	Holding Time
Pimephales promelas (fathead minnow) – 96-hr acute/7-day chronic toxicity, no dilutions, survival and growth	Surface Water	9 sample stations at each of the 5 sample sites; 1 QC Field Duplicate at each site Total = 50 samples for each species	One 2.5-gallon container per sample station (1 container for both tests)	Cool, 4° C, protected from light	14 days
Ceriodaphnia dubia – 48-hr acute/7-day chronic toxicity, no dilutions, survival and reproduction					
Chironomus Tentans (midge larvae) – 7-day Chronic Toxicity, survival and growth	Sediment	9 sample stations at each of the 5 sample sites Total = 45 samples for each species	One wide-mouth 1-gallon container per sample station (1 container for both tests)	Cool, 4° C, protected from light	14 days
Hyalella azteca – 10-day acute, survival and growth					
Cobicula fulminea (Asiatic clam) – 28-day bioaccumulation analysis	Sediment	3 sample stations at each of the 5 sample sites; 1 QC Field Duplicates at each site Total = 20 samples	One wide-mouth 1-gallon container per sample station	Cool, 4° C, protected from light	14 days

Table 1-6. Fish Tissue Analysis Samples: Number, Sample Preservation, Container Specification, and Holding Time Requirements in Support of the ERA

Parameter	Number of Samples[1]	Sample Container	Preservative	Holding Time
SVOCs, Pesticides, PCBs, Metals Herbicides, and Percent Lipids	3 Composite of Channel Catfish at each of the 5 sampling sites (for a total of 15 composites); 3-5 individual Channel Catfish (Fillets) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
Dioxins	1 Composite of Channel at each of the five sampling sites (for a total of 5 composites); 3-5 individual Channel Catfish (Fillets) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
SVOCs, Pesticides, PCBs, Metals Herbicides, and Percent Lipids	3 composites of Large Shad at each of the five sampling sites (for a total of 15 composites); 3-5 individual Large Shad (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
Dioxins	1 composite of Large at each of the five sampling sites (for a total of 5 composites); 3-5 Large Shads (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
SVOCs, Pesticides, PCBs, Metals Herbicides, and Percent Lipids	3 composite of Small Shad at each of the five sampling sites (for a total of 15 composites); 3-5 individual Small Shads (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
Dioxins	1 composite of Small Shad at each of the five sampling sites (for a total of 5 composites); 3-5 individual Small Shads (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
SVOCs, Pesticides, PCBs, Metals Herbicides, and Percent Lipids	3 fillets of Buffalo at each of the five sampling sites (for a total of 15 composites); 3-5 individual Buffalos (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
Dioxins	1 fillets of Buffalo at each of the five sampling sites (for a total of 5 composites); 3-5 Buffalos (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen

[1]Whole fish will be prepared and analyzed for target SVOCs, pesticides, PCBs, herbicides, and percent lipids, as input to the ecological risk assessment. Fish fillets will be prepared and analyzed for target SVOCs, pesticides, PCBs, herbicides, percent lipids, and dioxin as input to the human health risk assessment.

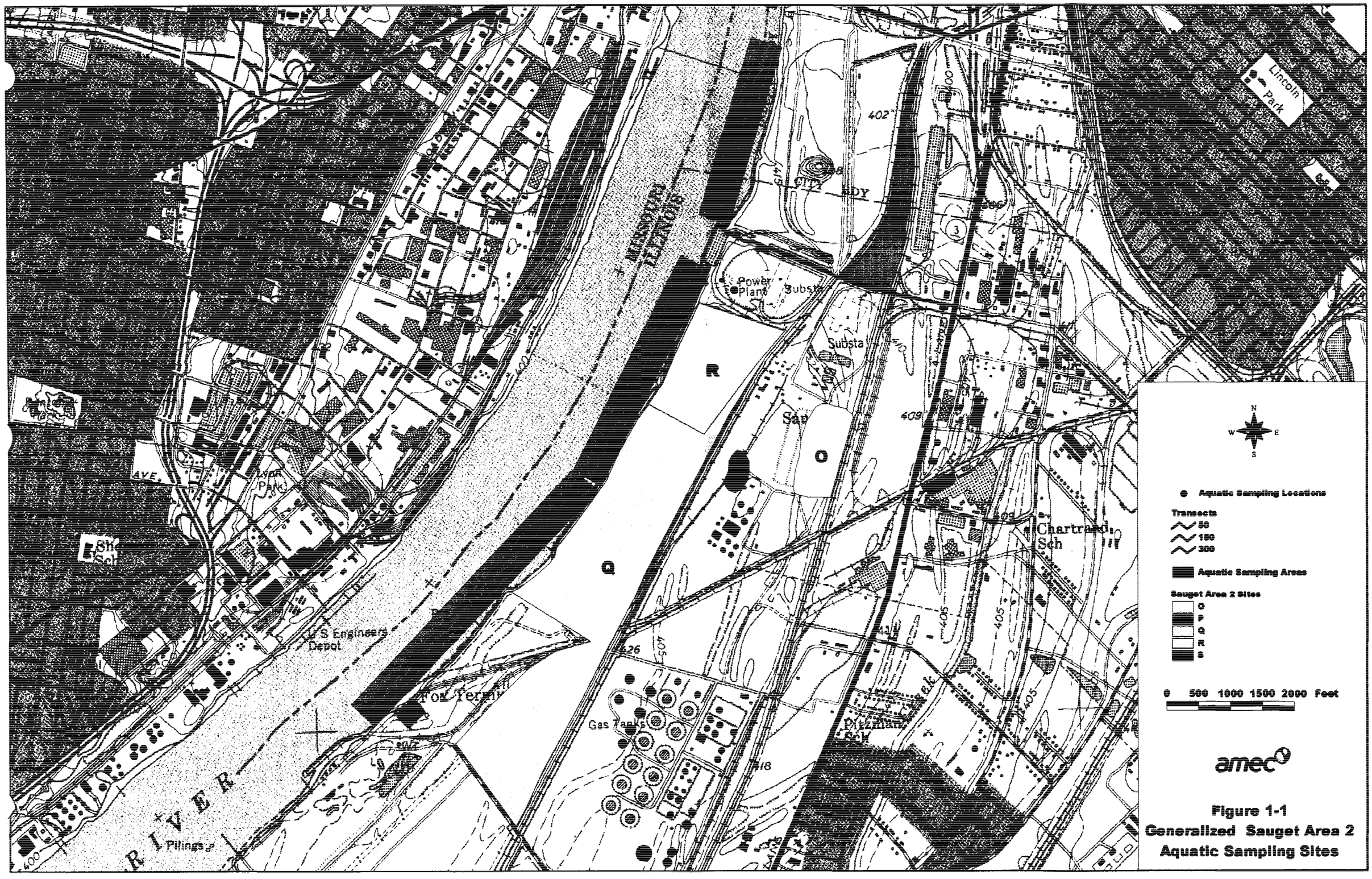

Figure 1-1
Generalized Sauget Area 2
Aquatic Sampling Sites

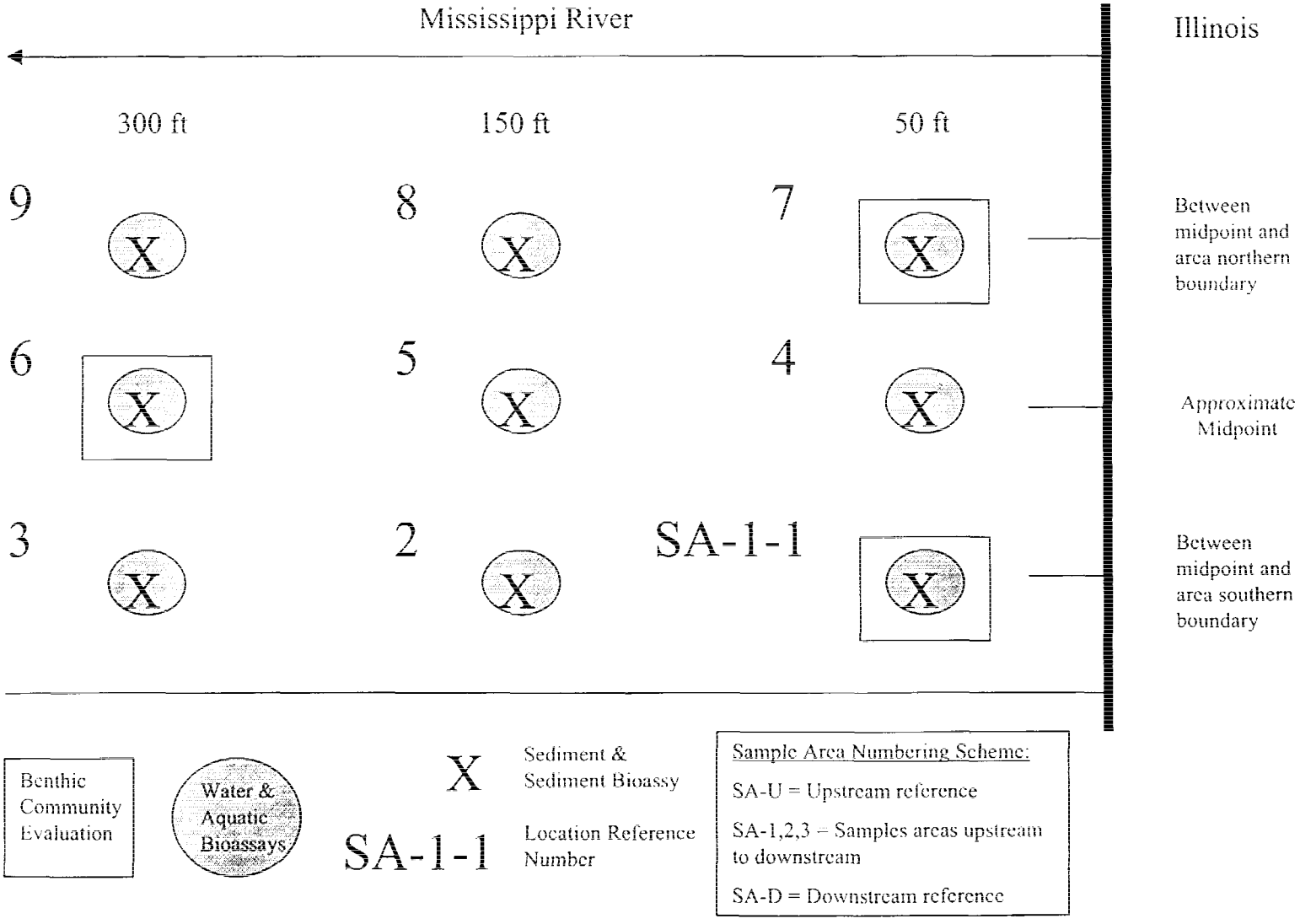

Figure 1-2. Sauget Area 2 Aquatic Sampling Scheme for Plume Discharge Area (PDA) and Reference Sites

VOLUME 3B

SURFACE WATER, SEDIMENT AND AQUATIC BIOTA QUALITY ASSURANCE PROJECT PLAN

SAUGET AREA 2 SITES
SAUGET, ILLINOIS
[USEPA Docket Number R8H-5-00-003]

May 25, 2001

Prepared for:

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1.0 PROJECT DESCRIPTION

This Quality Assurance Project Plan (QAPP) describes how data from the Mississippi River, which will be used in the Ecological Risk Assessment (ERA) will be collected, and describes the organization, objectives, planned sampling and analysis activities and the specific quality assurance/quality control (QA/QC) procedures to generate valid and usable data from surface water, sediment, and biota samples in support of the ERA at Sauget Area 2, Sauget, Illinois. The Ecological Risk Assessment is a part of the environmental investigations that will be performed to meet the requirements of Sections V. of the May 3, 2000 Administrative Order by Consent (AOC), USEPA Docket Number R8H-5-00-003, between USEPA and Solutia, Inc. Further site details and investigations are described in the Site Sampling Plan (SSP) for groundwater, surface water, and sediment, prepared by Solutia, Inc.

This QAPP has been prepared in accordance with the USEPA Region 5 QAPP Policy, April/May 1998. Specific protocols for sampling, sample handling and storage, chain-of-custody, and laboratory and field measurements are described in this QAPP. All QA/QC procedures and acceptance criteria have been developed in accordance with USEPA technical standards and requirements for the constituents of concern and media of concern.

1.1 Introduction

This QAPP has been prepared on behalf of the Sauget Area 2 Sites Group for Sauget Area 2, Sauget, Illinois, by AMEC Earth and Environmental Inc. (AMEC). The Site Sampling Plan (SSP), the Health and Safety Plan, the Ecological Risk Assessment Work Plan, and the Terrestrial Biota QAPP define the overall Workplan for the environmental investigations in support of the ecological risk assessment at this facility. This QAPP includes the Field Sampling Plan for ecological risk assessment sampling activities (Section 4) and applicable standard operating procedures (SOPs) in the attached appendices.

1.2 Site Facility Description, Historical Data and Current Status

The descriptions of the Site Facility, location, geological setting, features, historical data including past collection activities, and current status of the Facility are included in the "Site Sampling Plan" (SSP), prepared by Solutia, Inc., and wholly included in this QAPP by reference.

1.3 Project Objectives and Decision Statements

The purpose of this investigation is to gather sufficient information to quantify risk to ecological receptors in the event that environmental contamination is found to be present. In addition, chemical information from fish fillets will be used to quantify risk to human health. The objectives of the ecological investigation are to determine the nature, if any, of contamination migrating off-site, into the Mississippi River, from the facility and to collect sufficient data for all media (surface water, sediment, biota) to support the ecological risk assessment (ERA) and tissue data for input into the human health risk assessment (baseline risk assessment).

This ecological investigation will answer the following decision statement/question:

> "What is the nature, risk, and extent of select metal, volatile organic compound, semi-volatile organic compound, pesticide, herbicide, PCB, and dioxin contamination in off-site surface water, sediment, and biota that supports the case for the "no further action" alternative (i.e., does not present unacceptable risks), which would therefore not require further investigation and/or remedial action?"

Associated project-specific objectives for data collection, including constituents, target decision levels for ecological risk assessment for surface water, sediment, and fish and human health target decision levels for fish fillets are tabulated in Section 3, Tables 3-1 through 3-9 of this QAPP.

The following investigations will be performed in support of data input for the ERA:

1. A field Reconnaissance Survey - To generally evaluate site conditions, collect a series of sediment samples to and visual observations to locate discharge areas sampling transects, identify upstream and downstream sampling sites , confirm sampling methods, and choose final sampling locations for the Main Sampling Event;
2. Main Sampling Event – To collect biological, sediment and water sampling from three sites located within the plume discharge area (1. downgradient of Site P; 2. downgradient of Sites O, R, S, and the northern end of Site Q; and 3. downgradient of the southern end of Site Q), and at one site upstream and one site downstream of these areas. At each of these five sites, samples will be collected along three transects, with three sampling stations on each transect, for a total of 45 sampling stations. The following sampling collections and analyses will be carried out:
   - benthic organisms from sediment for an evaluation of benthic community structure;
   - forage fish, predator fish, bottom-feeder fish, and game fish for chemical analysis of target analytes;

- sediments for bioassay (sediment toxicity and bioaccumulation) testing;
- sediments for chemical analysis of target analytes and physical parameters;
- surface water for chemical analysis of target analytes and general water quality parameters; and
- surface water for bioassay (toxicity) testing.

Sediment and surface water toxicity tests will be conducted to evaluate the ecological risk due to exposure from facility contaminants (potentially from the groundwater plume discharge to the Mississippi River near shore environment) in surface water and sediments to representative plankton, fish, and benthic organisms according to accepted USEPA protocols as described in Table 7-1 and Appendix C.

The list of target analytes for this project, in surface water and sediment samples, was developed based on the detected compounds in the facility groundwater plume. The project target compounds list includes hardness (surface water only), metals, volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), pesticides, herbicides, dioxin/dibenzofurans and polychlorinated biphenyls (PCBs). Although there are only ten primary contaminants of concern (see Section 1.5, below, for further discussion), based on frequency and concentrations of detected compounds in the facility groundwater plume, the full Target Compound List (TCL) will be analyzed in surface water and sediment samples.

Fish tissue samples will be analyzed for the target compound list (TCL) of SVOCs, metals, dioxins/dibenzofurans, chlorinated pesticides and PCBs. Analysis of the fish samples for VOCs will not be performed since the necessary homogenization of these matrices, in preparation for analysis, will result in the loss of any volatile compounds that may have been present in the fish tissue. Most VOCs, SVOCs, herbicides and metals (with the exception of mercury, which is not a chemical of concern at the site) do not have partition coefficients as high as the PCBs, chlorinated pesticides and dioxins, and are not expected to bioaccumulate to the degree that these chemicals would. Tables 1-1 through 1-7 list the target parameters for both field and laboratory determinations. Specific analytical procedures are described in the text and tables of Section 7 of this QAPP and in Appendix B, which contains the relevant laboratory Standard Operating Procedures (SOPs) for the analytical methods.

Water quality measurements in surface water will be performed to aid in the interpretation of analytical data. These include field measurements of temperature, dissolved oxygen, pH, and conductivity as summarized in Table 1-8. Specific procedures for these water quality measurements

are described and tabulated in Section 7 and in the field SOPs provided in Appendix A. Additionally, non-target parameters of total organic carbon (TOC), and the physical parameters of percent solids and grain size will be measured in sediments in support of the ERA. Fish tissue composites will be analyzed for lipid content.

Specific procedures for the ERA, including the basis for choosing chemicals of ecological concern (COEC), protocols for habitat evaluation, rationale for defining exposure points, media of concern, etc., are provided in the Ecological Risk Assessment Workplan, which is fully incorporated into this QAPP, by reference. For purposes of performing risk assessment studies, levels of non-detected contaminants shall be assumed to be present at concentrations equal to one-half of the sample-specific reporting limit.

1.4 Sampling Plan Design and Rationale

The sampling plan design and rationale for sample locations in support of the ERA is described in detail in Section 4, Ecological Assessment Field Sampling Plan. Facility site maps showing proposed sampling locations in support of ERA activities are given in Figures 4-1 and 4-2. A phased approach is planned to initially perform a Reconnaissance Survey to choose site sampling locations within the area of plume discharge and two reference areas upstream and downstream of the area of plume discharge, within the near shore areas of the Mississippi River. The choice of reference locations will be based on qualitative observations of sediment and biological habitat, among other qualitative criteria. Following the Reconnaissance Survey, the Main Sampling Event will occur to collect and analyze the surface water, sediment, and biota (fish) samples in support of the ERA.

1.5 Target Parameters, Rationale, Media, and Frequency

The target analyte list was developed based on the potential impact of facility contaminated groundwater migration to the media that ultimately affect ecological receptors. Based on historical facility groundwater data, the ten primary contaminants of concern (COCs) include benzene, chlorobenzene, dichlorobenzenes, trichlorobenzenes, chloroanilines, phenol, chlorophenols, dichlorophenols, trichlorophenols, and pentachlorophenol. The target analyte list includes these primary COCs, as well as metals, other VOCs, SVOCs, pesticides, herbicides, dioxins/dibenzofurans and PCBs that can be analyzed using the methods defined in Section 7 of this QAPP. Tables 1-1 through Table 1-7 list the target parameters for VOCs, SVOCs, metals, pesticides, herbicides, dioxins/dibenzofurans, and PCBs in surface water, sediment, and biota (fish) including the project-required reporting limits (RL) for all target compounds.

Historical groundwater data indicate that the VOC and SVOC plumes extend from the facility process area down gradient to the Mississippi River. However, the extent of potential contamination from the facility VOC and SVOC plumes to surface water and sediment in the near shore areas of the Mississippi River is unknown. Therefore, this ecological sampling and analysis program will target the facility-specific contaminants of VOCs and SVOCs in the ecologically important media of concern in the near shore areas of the Mississippi River. Because data are limited on the presence of some compounds in groundwater, the list of analytes is expanded to include metals, pesticides, herbicides, dioxins/dibenzofurans, and PCBs.

The chemical data for surface water, sediment, and fish tissue samples will be used in the Ecological Risk Assessment. The chemical data from the fish fillets will also be used in the Human Health Risk Assessment. In addition, habitat evaluation and toxicity testing of surface water and sediments will be performed in support of the ERA. Non-target parameters that will aid in interpretation of chemistry and toxicity data in the ERA include the water quality parameters (turbidity, pH, temperature, dissolved oxygen, and conductivity), sediment parameters (total organic carbon, percent solids, and grain size), and percent lipids in fish tissue. Sample matrices, analytical parameters, and frequencies of sample collection in support of the ERA can be found in Section 4, Table 4-1 of this QAPP.

Project-required reporting limits (RL) for the target parameters were developed through the USEPA DQO process (see Section 3) and were derived from searches of background concentrations and bioaccumulation information available in the scientific literature to support the Ecological and Human Health Risk Assessment needs. Where risk information was not available for a specific compound, method and practical limits of quantitation formed the basis for the RL. In cases where the laboratory reporting limit does not meet ecological and human health risk-based criteria, a footnote appears in the tables explaining the approach to report below the RLs down to the laboratory method detection limit for detected results. This will allow for lower reporting limits for detected results. Laboratory MDLs are presented in Appendix B of this QAPP.

To meet the needs of this program, field sampling personnel, the analytical laboratory, the data validator and the risk assessors (human health and ecological) will work together on a frequent and regular basis to ensure that the resulting project RLs are as low as feasible for the media being sampled and that sample analytical results will achieve RLs within the limits of the selected analytical methods. The usability of such data with higher RLs will be evaluated during the risk assessment activities. In general, one half of the sample-specific detection levels will be used in risk calculations as a conservative estimate for compounds that do not meet the project RLs. After

review of the Ecological and Human Health Risk Assessments, there may be a need to perform additional sampling and analysis for target compounds that are drivers for the risk assessments if lower levels of detection are required.

1.7 PROJECT SCHEDULE

The overall project schedule for sampling and analysis in support of the ERA are described in the Site Sampling Plan. Based on this overall project schedule, the tentative ERA sampling and analysis project schedule is outline below.

1. August 2001: Complete Reconnaissance Survey to finalize site sampling locations within the Mississippi River. Submit a technical memorandum summarizing the results of Reconnaissance survey to USEPA.
2. September-October 2001: Complete Main Sampling Program for surface water, sediment, and fish.
3. November-December 2001: Samples are analyzed for chemical concentrations, biological characteristics, and toxicity by the analytical laboratories, and data validation is completed.
4. March 2001/April 2002: Report of Ecological Risk Assessment is prepared by AMEC.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The project organization and responsibilities for field sampling, data analysis and risk assessment in support of the ERA are defined in this section. A Project Organization Chart is presented in Figure 2-1 and includes all individuals discussed below.

2.1 USEPA PROJECT MANAGER

The USEPA Region 5 Project Manager, Michael McAteer, has the overall responsibility for the environmental activities in support of Sauget Area 2 site investigations and impact assessment work consistent with the AOC, Sections VI.Ib, 2, 3, and 4, May 3, 2000 (USEPA Docket Number RSH-5-00-003).

2.2 SITE PROGRAM MANAGER

The Site Program Manager, and Chairman of the Sauget Area 2 Sites Group, Steven Smith of Solutia, Inc. has the overall responsibility for ensuring that the project meets USEPA objectives and quality standards. In addition, he is responsible for technical quality control and project implementation and oversight. The Site Program Manager will ensure that technical, financial, and scheduling objectives are achieved. The Site Program Manager will report directly to USEPA Region 5 Project Manager and will provide the major point of contact and control for matters concerning the project. The Site Program Manager responsibilities include the following.

- Define project objectives and develop detailed work plans and schedules with the project team;
- Establish policies and procedures to address the specific needs of the project as a whole, as well as the objectives of each task;
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints;
- Orient all field leaders and project team staff concerning the project's special considerations;
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;

- Review and analyze overall task performance with respect to planned requirements;
- Approve all external reports (deliverables) before their submission to USEPA Region 5;
- Ultimately be responsible for the preparation and quality of interim and final reports; and
- Represent the project team at meetings and public hearings.

2.3 Ecological Project Manager and Field Leader for Ecological Risk Assessment

The Site Program Manager will be supported by the Ecological Risk Assessment Project Manager and Field Leader for the ERA. AMEC of Somerset, NJ, will perform the sampling and analysis activities to support the Ecological Risk Assessment evaluation at the facility. AMEC principal ecologist, Charles R. Harman, will provide the high-level technical direction for the ERA and will be the Ecological Project Manager. The Ecological Project Manager is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision in support of the ERA activities. He will report directly to the Site Program Manager. David Dean, of AMEC's Atlanta, GA office, will be the Field Leader for the Surface Water/Sediment/Aquatic Biota portion of the ERA and will coordinate the aquatics field program. Specific Ecological Project Manager/Field Leader responsibilities include the following.

- Provision of day-to-day coordination with the Site Program Manager on technical issues concerning the sampling and analysis for Ecological Risk Assessment.
- Development and implementation of the Ecological Risk Assessment Work Plan and the Surface Water/Sediment/Aquatic Biota QAPP including the specific field sampling plan activities described in Section 4.
- Coordination and management of field staff for the collection of surface water.
- Sediment, and fish and documentation of field observations important for the Ecological Risk Assessment evaluation.
- Oversight of technical efforts of subcontractors assisting the Ecological Risk Assessment team.
- Implementation of QAPP procedures for the collection and analysis of samples from all media.
- Adherence to work schedules provided by the Site Program Manager.
- Identification of problems at the field team level, discussion of resolutions and

implementation of corrective actions, as necessary, and

- Authorship, review, and approval of the Ecological Risk Assessment Report.

2.4 Ecological QA Chemists

Quality Assurance (QA) oversight for the ERA sampling and analysis activities described in this QAPP/FSP will be provided by Ms. Elizabeth Wessling and Dr. Mahalingam Ravichandran, both of AMEC. Responsibilities include:

- Preparation of the Surface Water/Sediment/Aquatic Biota QAPP in support of the Ecological Risk Assessment;
- Development of project DQOs to support the Ecological Assessment activities;
- Coordination with the analytical laboratory and field teams, as necessary, to ensure proper implementation of QAPP procedures;
- Coordination with the data validator, as necessary, to determine usability of the data for evaluation of ecological risk; and
- Technical assistance to the Ecological Project Manager and the Site Program Manager, as necessary, for chemistry and QA-related issues.

2.5 Technical Staff for the Ecological Risk Assessment Activities

The technical staff (team members) for this Ecological Risk Assessment will be assembled from AMEC staff. T he technical team staff will be utilized to gather and analyze data and to prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the Ecological Risk Assessment required work. The technical staff includes field observation and field sample collection staff, ecological risk assessors, quality assurance professionals, and regulatory experts.

2.6 Laboratory Quality Assurance Officers and Project Managers

The laboratory tasked with responsibility for chemical analyses of the surface water, sediment, and fish tissue samples in support of this project is STL – Savannah Labs, located in Savannah, Georgia. Kirstin McCracken is the Laboratory Quality Assurance Manager and Michelle Owens is the Laboratory Project Manager and primary contact for this project at STL. Triangle Laboratories in

Durham, North Carolina will perform all dioxin analyses. Helen Smpardos is the Triangle Laboratory Project Manager and Greg Johnson is the QA Manager. The toxicity testing for surface water and sediments will be performed by AMEC's San Diego, CA bioassay laboratory under the direction of Marilyn Schwartz. These laboratory QA Officers will be assisted by the laboratory operations managers and laboratory staff to assume the following laboratory responsibilities.

- Coordination of laboratory analyses including internal scheduling and tracking of sample analyses and chain-of-custody transfers;
- Oversight of data review and QA/QC documentation and results;
- Implementation of corrective actions, as necessary;
- Oversight of preparation of analytical data reports;
- Approval of laboratory data reports prior to submission to Site Program Manager;
- Ensure implementation of QAPP requirements for this project;
- Ensure implementation of laboratory QA procedures in accordance with laboratory QAPP;
- Perform periodic internal audits of laboratory and correct, edit, generate SOPs, as necessary; and
- Ensure up-to-date and compliant method detection limits and reporting limits for all methods and media of concern in support of this Facility investigation.

2.7 Data Validation

Data validation will be performed by trained personnel at AMEC, including Elizabeth Wessling, her data validation staff, and M. Ravichandran. Responsibilities of the data validation staff are to provide data validation and usability assessment services for 100% of the data collected in support of the ecological risk assessment and for the fish tissue data in support of the human health risk assessment using the regulatory and project-specific protocols defined in Section 9 of this QAPP.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, laboratory analysis, chain-of-custody, and reporting for surface water, sediment, and fish tissue samples that will provide results which are technically valid for use in the Ecological Risk Assessment. A secondary objective is to generate valid data for the fish fillets for use in the Human Health Risk Assessment. This section provides in greater detail specific project DQOs and intended data usage defined in Section 1 of this QAPP that were developed through the DQO process. The specific risk-based concentrations developed for the ERA and for the fish fillets to support the Human Health Risk Assessment can be found in the ERA Workplan, Tables 3-1 through 3-5. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, audits, preventive maintenance, and corrective action are described in other sections of this QAPP.

The Data Quality Objective (DQO) Process is a series of planning steps based on the scientific method designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate for the intended application. The DQO process is presented in Guidance for the Data Quality Objectives Process, USEPA QA/G-4 (USEPA 1994a). DQOs are quantitative and qualitative statements derived from outputs of each step of the DQO process that:

- Clarify the study objective;
- Define the most appropriate type of data to collect; and
- Determine the most appropriate conditions under which to collect the data.

The DQO process is developed through a multi-step process that includes the following:

Step 1. State the problem to be resolved.

Step 2. Identify the decision to be made.

Step 3. Identify the inputs to the decision.

Step 4. Define the boundaries of the study.

Step 5. Develop a decision rule.

Step 6. Specify the tolerable limits on decision errors.

Step 7. Optimize the design for obtaining the data.

Tables 3-1 through 3-9 define the project-specific DQOs for chemical data collected from surface water, sediment, and fish samples in support of the Ecological Risk Assessment and Human Health Risk Assessment.

3.1 Level of Quality Control Effort

The following specific quality control (QC) samples will be collected, prepared and analyzed to evaluate the quality of the data generated to support the Ecological Risk Assessment. Tables 3-1 through 3-8 define the level of the quality for the ecological assessment activities through the setting of project criteria for acceptance of QC sample results. Table 3-9 summarizes the type and frequency of QC samples in support of this QAPP.

Field blanks, laboratory method blanks, field duplicates, matrix spike duplicates, matrix spikes, laboratory control samples, surrogates, laboratory calibration QC, and tissue standard reference materials (SRM) will be analyzed to assess the quality of the data resulting from the field sampling and analysis of the surface water, sediment, and fish samples. See Appendix C for QA/QC procedures specific for the bioassay techniques in surface water and sediment.

3.1.1 Field Blanks

Field blanks for this project will consist of equipment rinsate blanks (for VOCs, SVOCs, pesticides, herbicides, metals, hardness, dioxins/dibenzofurans and PCBs) and trip blanks (VOCs only). The field equipment rinsate blank will consist of distilled interference-free water, preserved with appropriate preservative (see Tables 4-2 and 4-3), which will be provided by the laboratory, carried to the sampling site, exposed to sampling conditions, and poured over decontaminated sampling equipment and returned to the laboratory to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination that may have occurred during sample collection or handling prior to analysis.

The trip blank will consist of VOC-free water, provided by the laboratory, in a preserved VOA vial. This trip blank will be transported with the sample containers to the field and returned in the cooler with the VOC samples. The trip blank is used to measure potential cross-contamination between samples during shipment and handling. For this program, the trip blank for surface water samples will consist of water plus acid preservative (see Table 4-2). Two sediment trip blanks, one with deionized water for low-level VOC analysis and one with methanol for high-level VOC analysis will

be shipped with the sediment samples to assess potential contamination.

USEPA Region 5 requires the collection of one field blank (equipment rinse blank) for every ten investigative samples (10% frequency of collection) of a given matrix. Field blanks are not applicable to fish collection procedures, benthic community evaluation, or bioassay studies. In addition, field blanks will not be prepared for hardness measurements in surface water or for pH, TOC or grain size in sediments, as this level of QC is not required for these non-target parameters.

Based on the planned number of nine surface water samples per sample area, one field equipment rinse blank will be prepared for the surface water medium. For the analysis of dissolved metals in surface water, an additional blank will be collected by filtering DI water through the filter before the sample is taken. Based on the planned number of nine sediment samples per sample area, one field equipment rinse blank will be collected for the sediment medium, (see Table 4-1 in Section 4 of this QAPP). Collection of field blanks will comply with the Region 5 frequency requirement for this QC parameter. Note that for VOCs, both a high-level (methanol-preserved) and a low-level (reagent grade water preserved) field rinsate blank is required for sediments, consistent with the collection of high and low-level VOCs for analysis. Frequency and acceptance criteria for field trip and rinsate blanks are defined in Tables 3-1 through 3-8.

3.1.2 Field Duplicates

Field duplicates (FD) provide a measure of the reproducibility (precision) of the sampling procedures and the representativeness of the samples. Two collocated/separate samples from a single sample location are obtained, labeled with a unique sample number, and both are submitted to the laboratory for the appropriate analyses. The target frequency for field duplicate collection for laboratory analysis is one for every set of ten samples from each matrix collected by the same procedure for laboratory analysis. Frequency and acceptance criteria for FD precision are defined in Tables 3-1 thorough 3-8. For this project, one FD will be collected for the surface water samples and one FD for sediment samples from each of the five sampling areas.

3.1.3 Method Blanks

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedures. They must be performed for each extraction or digestion batch for each analytical method at a minimum frequency of one method blank per 20 field samples. Frequency

and acceptance criteria for method blank acceptance for all compounds of interest in surface water, sediments, and fish tissue are listed in Tables 3-1 through 3-8. Note that method blanks must be matrix-matched to the samples; *i.e.,* a method blank for high-level VOC analysis in sediments must contain methanol.

3.1.4 Matrix Spikes and Matrix Spike Duplicates

Matrix spikes (MS) and matrix spike duplicates (MSD) provide information about the effect of the sample matrix (media) on the accuracy (bias) and precision (reproducibility) of the digestion and measurement methodologies. One MS/MSD pair, spiked with the compounds of interest, must be generated for every 20 or fewer samples for each matrix (surface water, sediment, and fish tissue) for VOC, SVOC, pesticides, herbicides, metals, dioxin/dibenzofurans and PCB analyses. The relative percent difference between duplicate results is a measure of precision and the percent recovery of the matrix spike results is a measure of accuracy. Frequency and acceptance criteria for MS/MSD precision and accuracy are defined for this project in Tables 3-1 through 3-8. As required by the USEPA Methods in SW-846, each laboratory must routinely update the accuracy limits based upon their experience with real-world samples. Therefore, the limits tabulated are the current limits available from STL-Savannah Labs (January 2000).

3.1.5 Laboratory Control Sample/Standard Reference Material

A laboratory control sample (LCS) will be prepared and analyzed with each batch of field samples, for each matrix, at a minimum frequency of one LCS per 20 samples. The LCS will contain the compounds of interest (VOC, SVOC, pesticides, herbicides, dioxin/dibenzofurans and PCB target compound list), in an appropriate matrix to match the field samples (aqueous or solid) as available from a reliable, verifiable source *(e.g.,* NIST, certified vendor) separate from the source of the calibration standards. The results of the LCS must meet the project acceptance limits as listed in Tables 3-1 through 3-8. For SRMs, the vendor's limits for acceptance will be used.

For SVOCs in tissue, standard reference material (SRM) will be obtained for the target compounds of interest. Vendor-generated 95% confidence limits will be the acceptance criteria for the SRMs. SRMs should be analyzed at a frequency of one per 20 samples or per laboratory sample batch for tissues.

3.1.6 Surrogate Spikes

A surrogate spike contains pure substances not usually found in nature, with properties that mimic the compounds of interest. This spike is added to all organic samples prior to extraction to assess the accuracy of the method in the sample matrix. Criteria for surrogate spike recoveries are listed in Tables 3-1 through 3-8 based upon those acceptance limits developed by the analytical laboratory, STL-Savannah Labs (January 2000), consistent with USEPA method requirements in SW-846 for Methods 8260B (VOC) and 8270C (SVOC).

3.1.7 Laboratory Calibration Check Samples

A variety of QC samples are analyzed for the separate analytical methods to assess the accuracy of the analysis on a day-to-day basis. These QC checks include but are not limited to the following: criteria for initial calibration, continuing calibration, baseline drifts, contamination, etc., and are performed per method requirements by the laboratory. The details of these QC checks are available in the methods referenced in Section 7 of this QAPP and the laboratory specific SOPs for analysis included in Appendix B. A summary of laboratory and method-specific QC sample requirements is presented in Table 3-9.

3.2 Precision

Precision is a measure of the degree to which two or more measurements are in agreement. Field and laboratory precision QC requirements for this project are listed in Tables 3-1 through 3-8. Field and laboratory precision will be assessed through the calculation of relative percent differences (RPD) of the field duplicate results and matrix spike duplicate results. The equations to be used for calculation of precision criteria in this project can be found in Section 12 of this QAPP.

3.2.1 Field Precision Objectives

Field precision will be assessed through the collection and measurement of field duplicates. Field duplicates will be collected at a frequency consistent with USEPA Region 5 requirements of one duplicate per ten samples per media as previously described in Section 3.1.2, above.

3.2.2 Laboratory Precision Objectives

Laboratory precision will be assessed through the preparation and analysis of matrix spike duplicate (MSD) samples. Duplicate samples are two samples taken from and representative of the same

population and carried through all steps of the sampling and analytical procedures in an identical manner. In the laboratory, duplicate samples are analyzed to check for sampling and analytical reproducibility as a measure of precision and representativeness. The duplicate sample is a separate aliquot of a sample that the laboratory prepares and analyzes identical to the original sample; in the case of a MSD, the aliquots of a sample and a duplicate aliquot are spiked. The relative percent difference between the MS and MSD results is a measure of precision and representativeness. Section 3.1.4, above, describes the frequency and criteria references for acceptable precision of the MS/MSD results.

3.3 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference or true value. Accuracy will be assessed through the evaluation of recoveries of spiked compounds of interest into field samples, as well as the evaluation of laboratory control sample results, standard reference materials (SRM) for tissues, and through the evaluation of field and laboratory blanks. Tables 3-1 through 3-8 provide accuracy criteria for matrix spike (MS) and matrix spike duplicate (MSD) samples, laboratory control samples (LCS), and blanks for this project. The equations to be used for accuracy in this project can be found in Section 12 of this QAPP.

3.3.1 Field Accuracy Objectives

Accuracy in the field will be assessed through the use of field blanks (equipment rinsate blanks and trip blanks) and through the strict adherence to all sample handling, preservation, and holding times to maintain the integrity of the samples. Frequency and criteria for field blanks has been described in Section 3.1.1, above.

3.3.2 Laboratory Accuracy Objectives

Laboratory accuracy will be assessed through the analysis of MS/MSD, standard reference materials (SRM), LCS and surrogate compounds, and the determination of percent recoveries. These QC measures have previously been described in Sections 3.1.4 through 3.1.6, above. Accuracy control limits and required frequency for these QC measures are given in Tables 3-1 through 3-8 and also in the applicable SOPs as referenced in Section 7 of this QAPP.

3.4 Sensitivity - Reporting Limit Requirements

The sensitivity or reporting limit (RL) requirements for this project were defined to meet Ecological

Risk Assessment requirements in all three media and human health risk assessment requirements in fish fillet tissue, as best achievable based on the USEPA methods. Tables 1-1 through 1-8 list the compounds of concern, the media to be sampled and analyzed, and the risk-based project-required reporting limits for the level of detection.

Sensitivity may also be affected by contamination as observed in field and laboratory blanks (see Sections 3.1.1 and 3.1.3, above). Blank results will be used to assess contribution of contamination in attainment of project required RLs. These reporting limits will be achieved in surface water, sediment, and tissue samples through following the procedures as specified in this QAPP in Section 7. Note that the achievable reporting limits in sediment and tissue samples may be affected by matrix interferences. Sample cleanups, such as GPC and silica gel, may be performed by the laboratory to minimize matrix effects and to obtain project-reporting limits.

Additionally, Section 1.5 presents the rationale for the project reporting limits and discussion of approach to report down to the MDLs when necessary to achieve risk-based levels of detection. If the laboratory reports down to the MDL for any compound, they will flag the data with a "J" as an estimated value (reported below their PQL).

3.5 COMPLETENESS

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. The equation for completeness is presented in Section 12 of this QAPP.

3.5.1 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The field completeness objective for this project is greater than or equal to 90 percent. Note that to support the ecological risk assessment, field completeness refers to the collection of surface water, sediment, and fish samples, the documentation of ecological observations on site, and the planned surface water field measurements.

3.5.2 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The laboratory completeness objective for this project, with

respect to the chemical data being generated for biota in support of the Ecological Risk Assessment is greater than or equal to 90 percent.

3.6 REPRESENTATIVENESS

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition within a defined spatial and/or temporal boundary. Representativeness is dependent upon the proper design of the sampling program. The field sampling rationale, as presented in the Ecological Risk Assessment work plan and the Section 4 of this QAPP, has been developed to collect representative samples of surface water, sediment, and fish to assess potential ecological impacts of facility groundwater in the Mississippi River habitat locations planned for sampling.

3.6.1 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the procedures described in the Ecological Risk Assessment workplan and the field sampling procedures in Section 4 and Appendix A of this QAPP are followed. The media of concern for sampling in this QAPP are surface water, sediment, and fish. One measure of the representativeness of the samples to the site includes the precision of the field duplicate measurements for surface water and sediments as previously described in Section 3.1.2.

3.6.2 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the analytical procedures defined in this QAPP (see Section 7), maintaining proper preservation and meeting sample holding times to maintain sample integrity, performing appropriate homogenization and aliquoting procedures to ensure representative samples for analysis, and analyzing and assessing field and laboratory duplicate samples. A quantitative measure of representativeness of laboratory data is the precision of MS/MSD measurements, as previously described in Section 3.1.4.

3.7 COMPARABILITY

Comparability is an expression of the confidence with which one data set can be compared to another. Comparability is dependent upon the proper design of the field sampling and analytical measurement program.

3.7.1 Measures to Ensure Comparability of Field Data

Comparability will be satisfied by ensuring that the field sampling protocols defined in Section 4 and Appendix A of this QAPP are followed.

3.7.2 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented as required by this QAPP. Comparability is also dependent on similar QA objectives. As such, comparability of data for the surface water, sediment, and fish tissue to be sampled and analyzed in support of the Ecological Risk Assessment will be achieved by following the sampling procedures for collection as described in Section 4 of this QAPP, by using standard USEPA methods for analysis of VOCs and SVOCs as described in Section 7 of this QAPP, and by evaluating the validity and usability of the data generated for the risk assessment using standard EPA procedures and QA/QC criteria defined in this QAPP as described in Section 9 of this QAPP.

3.8 Decision Rules

Data quality objectives (DQOs) are qualitative and quantitative statements that specify the quality of the data required to support project decisions. The DQO process is a series of planning steps based on the scientific method that is designed to ensure that the type, quality, and quantity of environmental data used in decision-making are appropriate for the intended application. In this case, the USEPA DQO process was followed to establish the project DQOs for the Ecological Risk Assessment sampling and analysis activities. The seven steps of the DQO process, consistent with EPA guidance, were followed by the project team to generate the DQOs included in this section.

1. *Identify the principal study decision* - What is the nature, risk, and extent of select volatile organic compound and semivolatile organic compound contamination in off-site surface water, sediment, and biota that supports the case for the "no further action" alternative (i.e., does not present unacceptable risks), which would therefore not require further investigation and/or remedial action? Do chemical contaminants in surface water, sediments, or fish pose an unacceptable environmental risk to ecological receptors as represented by assessment endpoints?
2. *Define alternative actions that could result from resolution of the principal study question.* Information on ecological risks might be used to 1) determine if any remedial activities are

needed; 2) plan remedial activities for environmental media within and adjacent to the plume discharge area in the Mississippi River; and/or 3) determine the potential risks associated with remediation.

3. *Combine the principal study question and the alternative actions into a decision state* – Decide if remedial activities are needed to reduce unacceptable risks to ecological receptors. Identify which risks need to be addressed. Decide if remediation would result in net environmental benefits to ecological receptors.

4. *Identify the information that will be required to resolve the decision statement* – To resolve the decision statement, the following information needs to be collected and/or measured: 1) Measurements of surface water and sediment toxicity; 2) community evaluation of benthic organisms; 3) measurements of chemical contaminants in surface water, sediment, and fish; and 4) habitat evaluation.

5. *Identify the information that is needed to establish the action level* – a discrete action level is inappropriate for evaluating ecological risk. Instead, the multiple lines of evidence approach will be used. In accordance with this method, the risk assessor needs to the following information: 1) the confidence in each measure of risk (measurement endpoint), 2) the response in that measure (based on toxicity or chemical results), and 3) the concordance among measures (variability). This approach is described in the ERA Workplan. As an initial screening level of risk, and to help establish project-specific reporting limit requirements, risk-based concentrations (RBCs) of the contaminants to be measured in each media were established to help set the DQOs for ecological risk assessment. These risk-based levels, and their sources, are tabulated in the ERA Workplan.

6. *Identify practical constraints on data collection* – Collection efforts will be influenced by weather conditions (especially rainfall), the swift current of the Mississippi River which introduces practical constraints on sampling protocols, and by the suitability of habitats to support biota such as benthic invertebrates for collection of community structure information. Matrix effects on the accuracy of the chemical measurements for sediment and tissue analyses may pose a practical constraint on the usability of the data for ecological risk calculations. Such effects will be minimized by using cleanup techniques in the laboratory during sample preparation (see Section 7 of the QAPP for further information).

   A phased approach to the collection of required information and samples has been developed to address practical constraints of defining appropriate and representative

sampling locations and reference locations in support of the ERA. A Reconnaissance Survey (Survey) and a Main Sampling Event will be performed. The Survey will be used to refine the Main Sampling Event by making observations of habitat for biota and performing and a qualitative bathymetric survey. The field observations made during this Survey will also be used to finalize sampling locations and sampling procedures.

During the Main Sampling Event surface water, sediment, and fish samples will be collected and analyzed for target compounds and surface water and sediment will be collected to assess toxicity using laboratory bioassays. Additionally, benthic invertebrates will be collected from sediment to perform a benthic community evaluation. A summary of the proposed sampling and analysis program is provided in Section 4, Table 4-1. Analyses of chemicals in fish fillets will also be used in the Human Health Risk Assessment to evaluate human exposure due to ingestion. The Human Health Risk Assessment Workplan describes the details for evaluating this pathway.

7. *Specify the statistical parameter that characterizes the population of interest* - A number of statistical methods will be utilized to evaluate risk. However, chemical concentration data will be expressed as arithmetic means, medians, and 95th confidence intervals on the mean (or maximum values) for risk assessment calculations.

8. *Develop a decision rule* - The multiple lines of evidence approach will be used. In accordance with this method, the risk assessor considers: 1) the confidence in each measure of risk (measurement endpoint), 2) the response in that measure, and 3) the concordance among measures. This approach is described in the ERA Workplan.

9. *Determine the possible range of the parameter of interest* - The measurement methods defined can accommodate a wide range of chemical concentrations for each analyte of interest. Project-specific RLs were developed, using the DQO process, to meet the data input requirements for the ecological risk assessment. Meeting project sensitivity requirements is most important in usefulness of chemical data. The historical range of chemicals of interest will be reviewed and discussed in the ecological risk assessment report for this project.

10. *Identify the decision errors and choose the main hypothesis*

11. *Define both types of decision errors and establish the true state of nature for each decision error.* The two types of decision error are 1) concluding that there is no risk when there is, and 2) concluding that there is a risk when there is not.

12. *Specify and evaluate the potential consequences of each decision error.* The first error could result in risks being left unaddressed thereby jeopardizing the health of the environment. The second error could result in expenditures that do not have a measurable benefit and which could even result in additional ecological harm.

To minimize decision errors, the highest level of data quality is defined for target compound data generated in support of risk assessment. Analyses will be performed in strict accordance with the USEPA methods defined in Section 7 of this QAPP. All of the chemical data for use in the ecological risk assessment and the fish fillet data for use in the human health risk assessment will be validated using USEPA data validation guidance (National Functional Guidelines for Organic Data Review, USEPA) and the Quality Assurance/Quality Control (QA/QC) requirements described in this QAPP. Specific DQOs for QA/QC, to support the ecological risk assessment chemical measurements, have been defined for the QA/QC parameters of accuracy, precision, sensitivity, representativeness, completeness, and comparability. These project-specific DQOs are summarized in Tables 3-1 through 3-8.

4.0 ECOLOGICAL ASSESSMENT FIELD SAMPLING PLAN

This section constitutes the Field Sampling Plan (FSP) for the Ecological Risk Assessment. It describes the conceptual approach for a Reconnaissance Survey and how data from the Mississippi River, to be used in the Ecological Risk Assessment (ERA), will be collected during the Main Sampling Event. It also describes planned stations for collection of surface water, sediment, and fish samples, collection procedures, number of samples to be collected, container and preservation requirements, and holding times. In this document, the terms "station" and "location" are used interchangeably to describe the discrete points at which samples will be collected. Further field documentation procedures required for the FSP, including labeling and chain-of-custody requirements, are included in Section 5 of this QAPP. The FSP procedures to collect fish fillets support the Human Health Risk Assessment for ingestion of fish as a potential exposure pathway. Field sampling personnel will follow the procedures documented in this Section 4.0 and subsequent Section 5.0, the associated field sampling SOPs included in Appendix A, and the Ecological Risk Assessment Health and Safety Plan (HASP), attached as Volume 3C. The field sampling procedures may be refined as a result of the reconnaissance survey.

4.1 STUDY AREA

The Site encompasses approximately 344 acres situated adjacent to the Mississippi River. The Site, found within the villages of Sauget and Cahokia, Illinois, is roughly bounded to the west by the Mississippi River, to the north by the MacArthur bridge railroad tracks, to the east by Illinois State Highway 3, and to the south by Cargill Road. The Site fronts approximately 8,000 feet of the Mississippi River.

The Sauget Area 2 Site includes five former disposal areas, Sites O, P, Q, R and S, adjacent, or in close proximity, to the Mississippi River. These five disposal areas were given letter designations by the Illinois Environmental Protection Agency (IEPA) in the 1980s. Two of these sites, Sites Q and R, are located on the wet side of the floodwall and levee which is operated and maintained by the US Corps of Engineers and the Metro East Sanitary District. The floodwall is designed to protect the City of East St. Louis and the Villages of Sauget and Cahokia from flooding. Sites O, P and S are located on the dry side of the floodwall and levee.

Site O is located on Mobile Avenue in Sauget and occupies approximately 20 acres northeast of the American Bottoms Regional Wastewater Treatment Facility (ABRTF). Site P is located between the

Illinois Central Gulf Railroad and the Terminal Railroad and is north of Monsanto Avenue in the Village of Sauget. Site P occupies approximately 20 acres of land. Site Q occupies approximately 90 acres and is bordered by the Sauget Site R and the old Union Electric Power Plant on the north, the Illinois Central Gulf Railroad and the U.S. Corps of Engineers flood control levee on the east, and the Mississippi River on the west. The two ponds created by the borrow pit operations are located at the southern end of Site Q. Site R is located adjacent to the Mississippi and has had a temporary cap placed on it. Site S is a small drum disposal area west-southwest of Site O.

The ecological assessment will focus on portions of the Mississippi River downgradient of Areas O, P, Q, R, and S, shown in Figure 4-1. The aquatic sampling study area includes the near-shore portion of the Mississippi River where facility groundwater may be discharging to surface water, referred to here as "plume discharge area" (PDA) and sites upstream of the discharge area (UDA) and downstream of the discharge area (DDA). The UDA and DDA sites have not yet been identified (see Section 4.2.1 below) and as such are not shown on the figure. The plume discharge area is defined based on the terrestrial boundaries of Sites P through Q. A description of sampling sites and site photos will be provided after the reconnaissance survey described in Section 4.2.1.

4.2 Field Sampling Rationale and Sampling Locations

The Ecological Risk Assessment FSP consists of two separate sampling field events: a Reconnaissance Survey and the Main Sampling Event.

4.2.1 Reconnaissance Survey Objectives

A Reconnaissance Survey (Survey) will be conducted to evaluate physical conditions at the planned sampling locations and to refine the Field Sampling Program. The field observations made during the Survey are used to provide a verbal and photographic description of the site, finalize sampling locations, procedures, and determine the number of aquatic samples that can be realistically collected during the main sampling event. The objectives and justification for the Survey activities are described below:

- **Photodocument Areas O, P, Q, R, and S**

- **Locate aquatic sampling locations** for the collection of water, sediment, and biota samples during the Main Sampling Event. This will be done by collecting a series of sediment samples for the analysis of volatile organic compounds (VOC) (by method 8260B), and by making qualitative observations of the sediments including the presence of any benthic invertebrates and grain size. Sediment samples will be collected using a ponar sampler near the bank of the Mississippi River, from the northernmost end of site P to the southernmost end of site Q. Samples will be collected every 500 ft (a total of approximately 28 samples) on a line approximately 50 ft from the bank.

- **Select two upstream / downstream sites** for comparison of ecological impact in the plume discharge area to areas where no facility groundwater discharge occurs. These sites will include an Upstream (UDA) and Downstream (DDA) site, with nine sampling stations within each site. One set of samples will be collected at each of these stations during the Main Sampling Event. The UDA sampling stations will be selected, to the extent practicable, in areas of similar grain size and habitat as the sampling sites adjacent to areas O, P, Q, R, and S. The UDA sites will be located as far north as River mile 190 (and will not be north of Lock and Dam 26). The DDA stations will be supported by a review of river currents and bathymetric data maintained by the U.S. Army Corps of Engineers and other agencies to verify the downstream samples can all be located along the eastern shore of the Mississippi River. To the extent practicable, DDA stations will be selected in depositional environments that have potentially been impacted by erosion and subsequent deposition of sediments from the plume discharge area. A minimum of four downstream samples will be located in depositional areas. These sampling stations will be investigated for site-related impacts during the Main Sampling Event. Overall, the upstream / downstream sites should be reasonably comparable to the plume discharge area in terms of habitat and sediment characteristics. These characteristics will be limited to those that can be visually observed in the field (e.g. adjacent stream bank condition, depth of water, approximate velocity, sediment grain size, color, odor, presence of organic matter, and presence of benthic macroinvertebrates).

- **Finalize the list of the selected representative receptor species** that are using the Mississippi River habitats and represent assessment endpoints for the baseline ERA.

- **Conduct a fish habitat evaluation** for the planned sampling locations in the Mississippi River. The purpose of this evaluation will be to determine if these portions of the Mississippi River

support the types of fish that are planned for collection to represent different trophic levels for the ERA and game fish (for ingestion pathway) for the human health risk assessment. This evaluation will include discussions with a professional fisherman and an aquatic scientist familiar with the river.

- **Perform a habitat assessment for benthic invertebrates** by documenting the physical and environmental conditions for the planned ERA sampling locations, including the upstream / downstream sites. Observations of the bottom substrate, available cover, estimation of embeddedness, estimation of the flow or velocity and depth regime, channel morphology, and riparian and bank structure (such as bank stability, bank vegetation, and streamside cover) will be documented to evaluate the benthic habitat.
- **Determine the most appropriate sampling techniques** for water, sediment and fish based on Survey observations. Due to the expected swift current of the Mississippi River, and the anticipated coarse texture of the sediments, different sampling techniques will be evaluated to ensure that the Main Sampling Event will generate representative samples for use in the ERA.
- **Evaluate the need for modifications of analytical preparation methods** based on visual observations of the "wetness" (percent moisture) of the sediments to evaluate the need for freeze-drying techniques to increase the solids content as a means to achieve adequate chemical detection levels.

After the reconnaissance survey is completed, a technical memorandum, summarizing the results of the survey will be prepared and submitted to US EPA. This memo will summarize the chemical results of screening sample analysis, present observations made during the reconnaissance survey, and confirm the sampling locations to collect aquatic and terrestrial samples during the main sampling event. Relevant information from the Survey will be incorporated into the final planning of the Main Sampling Event.

4.2.2 Main Sampling Objectives

The Main Sampling Event, which is scheduled for July-August 2001, will consist of collection of surface water, sediment and fish samples from the Mississippi River and analyses of these samples to determine the concentration of site-related constituents in these three media and assess their impact, if any. The sampling locations and rationale are provided in the next section (4.2.3). The sampling activities will consist of the following collections:

- **Sediment samples for chemical and physical analyses**: at 45 locations (nine sampling stations at each of the three plume discharge area sites and two upstream / downstream sites) for use in the ERA as part of the sediment triad approach.

- **Sediment samples for toxicity testing**: at 45 locations (nine sampling stations at each of the three plume discharge area sites and two upstream / downstream sites) for chronic toxicity tests and bioaccumulation tests for use in the ERA and as part of the sediment triad approach. Chronic toxicity testing will use two indicator species, *Hyalella azteca* and *Chironomus tentans*; a total of 90 sediment toxicity tests will be performed (two species x 45 locations). Also, for two stations for each toxicity test species, the sediment will be examined for VOC concentrations at the beginning and the end of the bioassay to assess VOC concentration changes over the bioassay period.

- **Sediment samples for bioaccumulation testing**: at 15 locations (on sediment samples collected at the center sampling station on each transect in each of the three plume discharge area sites and two upstream / downstream sites) for bioaccumulation tests for use in the ERA and as part of the sediment triad approach. The bioaccumulation test will use *Corbicula fulminea* (Asiatic clam) to assess the potential for contaminant bioaccumulation in the tissue of sediment-dwelling organisms; a total of 15 bioaccumulation tests will be performed (15 locations).

- **Benthic invertebrates samples**: at each of the 9 of the upstream and 9 downstream sampling sites, and at each of the 27 site-related sampling sites (9 sampling sites within each of the three sampling sites) = 45 samples for analysis of community structure (species identification to the lowest practical taxon, species richness, and biomass) for use in the ERA and as part of the sediment triad approach.

- **Surface water samples for chemical analyses**: at 45 locations (nine sampling stations at each of the three PDA sites and two upstream / downstream sites) for chemical analysis for use in the ERA. In addition to the unfiltered surface water samples collected, a filtered surface water sample will be collected at each sampling location for trace metals analysis. Filtered sample results are required in order to compare to ambient water quality criteria.

- **Surface water samples for toxicity:** at 45 locations for chronic surface water toxicity (bioassay) tests using two indicator species, the cladoceran *Ceriodaphnia dubia* and the fathead minnow, *Pimephales promeles,* for use in the ERA. A total of 90 surface water toxicity tests will be performed (two species x 45 locations).

- **Fish samples for chemical analysis:** at three locations within the plume discharge area and at upstream and downstream sites for three trophic levels (bottom feeder, forager, and predator) for use in the ERA and for predator/game fish for use in the Human Health Risk Assessment. The weight, length, and width of each fish will be recorded. The stomach contents of a representative subset will be observed. The general health (and any visual anomalies) will be assessed by a qualified aquatic biologist.

- **Field observations** of the fish community and their habitat for use in the ERA, as necessary.

- **QC samples**: including field duplicates, field equipment rinsate blanks, trip blanks, and additional sample volumes for matrix QC of matrix spike/matrix spike duplicates at the frequencies defined in Section 3 of this QAPP and detailed, per media and parameter type, in Tables 4-1 through 4-6 at the end of this FSP.

4.2.3 Sampling Locations

Surface water, sediment, and fish samples will be collected along three transects running parallel to the river bank at five stations: 1) downgradient of Site P, 2) downgradient of Sites O, R, S and the northern end of Site Q, 3) downgradient of the southern end of Site Q, 4) a reference site upstream of Sauget Area 2, and 5) a site downstream of Sauget Area 2 (upstream and downstream sampling sites are to be selected based on results of the reconnaissance survey). Transects will be located 50, 150 and 300 feet from shore. Each transect will consist of three sampling stations; thus, there are nine sampling locations for each of the five sites listed above (a total of 45 sampling locations). The general locations of PDA transects are shown on Figure 4-1 and generalized sampling locations within each sample site are shown in Figure 4-2. Actual sampling sites will be finalized based on visual observations, sediment physical characteristics, and VOC analysis of sediment samples collected during the reconnaissance survey. Coordinates for all final sampling locations will be

established in the field using a differential Geographical Positioning System (GPS) as well as by line of sight.

Reconnaissance survey analytical data, collected as part of the May 2000 W.G. Krummrich Accelerated Corrective Action AOC, indicate that these sampling transects will be located in the likely plume discharge area. During this survey, which was conducted in September 2000 by Menzie-Cura & Associates, sediment samples were collected in the Mississippi River downgradient of the Krummrich plant along three transects running from the bank toward center of the river. Analytical results from the reconnaissance survey are summarized below:

	Distance from Bank, feet								
Total VOCs (ppb)	**50**	**200**	**300**	**400**	**500**	**600**	**700**	**1000**	**1400**
North Transect	644	NS	854	ND	ND	NS	ND	ND	ND
Center Transect	1300	ND	NS	NS	ND	NS	NS	NS	NS
South Transect	45	NS	473	NS	NS	1	NS	NS	NS

Surface water, sediment, and fish samples collected during the main sampling event will be analyzed for chemicals of concern (target analytes) listed in Tables 4-1 through 4-7 as presented in Section 1.0 of the QAPP. Specific numbers of samples, types of samples to be collected, container specifications, etc. are described below. Analyses of chemicals in fish fillets will be used in the Human Health Risk Assessment to evaluate human exposure due to ingestion. Fillets of buffalo will be analyzed and used in human health risk assessments. Composites of Channel Catfish will be analyzed and used in ERA evaluation of risks to Piscivorous animals. Benthic community structure will be evaluated to provide data for sediment triad evaluation. Bioassays will be conducted on surface water and sediment samples to determine the toxicity, if any, of these environmental media to sensitive organisms.

Fish will be sampled in three sites in the Mississippi River: 1) downgradient of Site P, 2) downgradient of Sites O, R, S and the northern end of Site Q, and 3) the southern end of Site Q. A food source approach will be used to select fish for fish tissue analysis:

Primary Food Source	Fish	Trophic Level	Endpoint Organism
Detritus, benthic invertebrates, piscines	Channel Catfish	Bottom feeder / Predator	Piscivorous animals
Plankton	Shad (Large)	Forager	Osprey
Plankton	Shad (Small)	Forager	Heron
Aquatic insects, piscines	Buffalo	Predator/Gamefish	Recreational fisher

These fish tissue samples, collected in the plume discharge area, will be used to determine the impact, if any, of groundwater discharge on higher trophic level organisms. Fish will also be sampled from sites upstream and downstream of Sauget Area 2.

Information collected as part of the Surface Water, Sediment and Aquatic Biota Sampling Plan will be used in the Human Health Risk Assessment and the Ecological Risk Assessment. With five disposal sites located adjacent to or near the east bank of the Mississippi River, the primary ecological exposure pathway is groundwater discharge to surface water. Other exposure pathways include terrestrial organism exposure to site soils and aquatic organism exposure to water and sediments in on-site ponded areas. These exposure pathways will be included in the conceptual site model section of the Ecological Risk Assessment Workplan. Additional exposure pathways include surface runoff from the site to the Mississippi River and seeps along the riverbanks. Surface runoff sampling will be conducted by URS as part of the Remedial Investigation, and is described in a separate document.

All of the above sampling and analysis activities will be performed under the direction of AMEC field team leader. Samples to be collected from the various media and the chemical, physical, and toxicity analyses to be performed in support of the Main Sampling Event for Sauget Area 2 investigations are summarized in Table 4-1. Analytical methods to be used for analyses are presented in Section 7 of the QAPP.

4.3 SURFACE WATER SAMPLING

4.3.1 Water Sample Collection

Surface water samples will be collected at the 45 sampling locations identified in Figure 4-1 and Figure 4-2. Surface water samples will be collected at the sediment-water interface (within one foot of the bottom) at all stations. Surface water samples will be collected using the standard operating procedure, *Surface Water Sampling* (Appendix A). Note that this SOP may be modified after the reconnaissance survey and during actual sampling based on logistics of surface water sampling in the Mississippi River. Because of the volumes required and sampling limitations due to the fast currents, a peristaltic pump system will be utilized. Decontaminated tubing will be used at each location. A decontaminated Niskin sampler may also be utilized to collect water sample at depth. All chemical analysis in surface water samples will be conducted in unfiltered samples. For trace metals analysis, in addition to unfiltered samples, a filtered water sample will also be collected using disposable 0.45 um filters connected in-line to the pump.

In all cases, the performance criteria for sampling are to collect water samples in a manner that prevents contamination from the sediments, minimizes the potential for contamination by the sampling system, and provides a representative sample of the water column above the sediments. Potential contamination of surface water samples by sediments will also be minimized by either conducting all surface water sampling prior to the sediment sampling event or by performing surface water sampling prior to any sediment sampling at a location (if the sampling events are combined). Surface water samples will be collected in the following locations:

Site P - Surface water samples will be collected along three transects running parallel to the bank from the north to south end of Site P. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling

stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One surface water sample will be collected at each sampling station and analyzed for hardness, metals (both dissolved and total), VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Surface water samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 4-2) will be analyzed for dioxins. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Ceriodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

Sites O, R, S and Northern End of Site Q - Surface water samples will be collected along three transects running parallel to the bank from the north to south end of Site R. Sites O and S and the northern end of Site Q are located upgradient of Site R. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One surface water sample will be collected at each sampling station and analyzed for hardness, metals (both dissolved and total), VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Surface water samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 4-2) will be analyzed for dioxins. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Ceriodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

Southern End of Site Q - Surface water samples will be collected along three transects running parallel to the bank from the south end of Site R to the south end of Site Q. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on

each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One surface water sample will be collected at each sampling station and analyzed for hardness, metals (both dissolved and total), VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Surface water samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 4-2) will be analyzed for dioxins. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Ceriodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

Upstream Reference Site - Surface water samples will be collected at nine locations upstream of Sauget Area 2. Sampling locations will be selected during a reconnaissance survey conducted prior to sample collection.

One surface water sample will be collected at each sampling station and analyzed for hardness, metals (both dissolved and total), VOCs, SVOCs, dioxins, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Cerodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

Downstream Sampling Site - Surface water and sediment samples will be collected at nine locations in the Mississippi River downstream of Sauget Area 2. Sampling locations will be selected during a reconnaissance survey conducted one month prior to sample collection.

One surface water sample will be collected at each sampling station and analyzed for metals (both dissolved and total), VOCs, SVOCs, dioxins, pesticides, herbicides and PCBs to determine the concentration of these constituents in surface water. Samples will be collected just above the sediment/surface water interface. Bioassays, using *Cerodaphnia* and *Pimephales*, will be performed on each surface water sample to determine surface water toxicity.

4.3.2 Water Sample Analytes, Containers, and Shipment Requirements

All surface water samples will be analyzed for the target compounds in the following categories: hardness, metals, VOCs, SVOCs, pesticides, herbicides, dioxin and PCBs. In addition, field measurements of temperature, pH, dissolved oxygen, and conductivity and laboratory measurements during bioassays will be conducted to characterize the water quality, as described in Section 4.3.3, below. In addition to collecting an unfiltered water sample, water samples collected for trace metals analysis will be filtered in the field using a disposable 0.45 um filter. The VOC samples will be collected by directly filling 40-mL VOC vials from the peristaltic pump tubing to minimize VOC loss and avoid preservative loss. Table 4-2 summarizes the number of samples, sample container types, preservation, and holding time requirements for all compounds in surface water samples. Field duplicates, field blanks and MS/MSD samples for QC will be collected at frequencies as described in Section 3 of this document. QC samples are also listed in Table 4-1 as a summary of all the types of samples planned for collection and analysis.

All surface water samples will be appropriately labeled with location, date, sampler's initials, test type and preservative and stored on ice, maintained at 4°C and delivered to the appropriate laboratory within 24 hours of collection via FedEX or similar overnight carrier for next day delivery. The chain-of-custody (COC) documentation will accompany all samples from the field to the laboratory (see Section 5 of this QAPP).

4.3.3 Supporting Measurements for Surface Water Quality

At each location, the following water quality parameters will be measured in the field: turbidity, dissolved oxygen, specific conductivity, temperature, and pH. Measurements will be made at the surface (top), middle, and bottom portion of the water column, consistent with the surface water sample collection protocols. Depending on water flow conditions, water quality parameter measurement may be restricted to surface waters only. Water quality measurements will be made using a Horiba U-10 meter for turbidity, dissolved oxygen (DO), specific conductivity, temperature, and pH. Calibration of field equipment will be performed as described in the field measurement SOP included in Appendix A of the QAPP.

4.4 SEDIMENT SAMPLING

4.4.1 Sediment Sample Collection

Sediment samples will be collected at each of the 45 sediment locations using a Van Veen grab sampler (3.2 L or 11.1 L). The equipment is deployed from a davit along the side of the boat and collected using winch assistance. Prior to sampling at a location, the grabs and all other sampling devices such as spoons are decontaminated in accordance with the: SOP, *Equipment Decontamination* (Appendix A). Table 4-3 lists the number of sediment samples to be collected, container and preservation specifications and holding time requirements for the chemical analysis to be performed on the sediment samples. Table 4-4 and 4-5 list the number of sediment samples, container and preservation specifications and holding time requirements for the benthic community evaluation and bioassays, respectively, to be performed in support of the ERA.

The goal of the sampling program is to collect representative sediment from the upper few inches (5-6 cm). This depth interval was selected as the zone most relevant to exposures of ecological receptors. The sediments will be collected and analyzed for the chemicals and sediment parameters presented in Tables 4-1 through 4-7 of the QAPP. Several grab samples may need to be collected at each location to provide sufficient sample volume for all the parameters to be tested including the target compound chemical analyses (metals, VOCs, SVOCs, pesticides, herbicides, dioxin and PCBs), the non-target analysis of pH, total organic carbon (TOC) and grain size, collection of benthic invertebrates for community evaluation, and for the sediment toxicity (bioassay) tests. Section 4.5 discusses collection of benthic invertebrate samples and Section 4.6 discusses collection of samples for bioassay toxicity testing.

Samples for VOCs will be obtained from the first grab. All other samples will be drawn from a composite made up of the upper few inches of sediment from additional grab samples. The VOC sample will be collected using a 5 mL modified syringe/plunger. The plunger tube will be inserted into the sediment below the surface and removed with care taken to prevent loss. The plunger will then be utilized to push the sample into the bottle. This method is the only feasible method for collecting samples in sediments that are very coarse-grained. These samples will be released into 1) laboratory water preservative for low-level analysis, 2) methanol preservative for high-level

analysis. The number of sediment samples to be collected for VOC analysis, container specifications, preservation, and holding time requirements are listed in Table 4-3.

For all other parameters, the sediment (~5-6 cm) will be removed from the grab and homogenized in a stainless steel bowl using a stainless steel spoon or scoop. Sufficient sediment will be collected and mixed in the bowl so that sub-samples can be taken for chemical analysis of SVOCs, pesticides, herbicides, metals, dioxins, PCBs and TOC, physical analysis of grain size distribution, and sediment toxicity (bioassay) testing. Each of these sub-samp1es will be taken using either a stainless steel spoon and placed in the appropriate container (see Section 4.4.2, below).

Site P - Sediment samples will be collected along three transects running parallel to the bank from the north to south end of Site P. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Sediment samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 4-2) will be analyzed for dioxins. Bioassays, using the amphipod *Hyallela azteca* and the midge larvae *Chironomus tentans*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

Sites O, R, S and Northern End of Site Q – Sediment samples will be collected in the Mississippi River along three transects running parallel to the bank from the north to south end of Site R. Sites O and S and the northern end of Site Q are located upgradient of Site R. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Sediment samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 4-2) will be analyzed for dioxins. Bioassays, using *Hyalella* and *Chironomus*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

Southern End of Site Q - Sediment samples will be collected in the Mississippi River along three transects running parallel to the bank from the south end of Site R to the south end of Site Q. Transects will be located 50 feet, 150 feet and 300 feet from the bank. Three sampling stations will be located on each transect resulting in nine sampling stations within the plume discharge area. One sampling station will be located at the center point of each transect. Another sampling station will be located half way between the center station and the upstream end of each transect. A third sampling station will be located half way between the center station and the downstream end of each transect.

One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Sediment samples from five of the sampling locations (stations 6,5,4,1, and 7 in Figure 4-2) will be analyzed for dioxins. Bioassays, using *Hyalella* and *Chironomus*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

Upstream Reference Site - Sediment samples will be collected at nine locations in the Mississippi River upstream of Sauget Area 2. Sampling locations will be selected during a reconnaissance survey conducted one month prior to sample collection. One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, dioxins, metals, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Bioassays, using *Hyalella* and *Chironomus*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

Downstream Sampling Site - Sediment samples will be collected at nine locations in the Mississippi River downstream of Sauget Area 2. Sampling locations will be selected during a reconnaissance survey conducted one month prior to sample collection. One sediment sample will be collected at each sampling station and analyzed for metals, VOCs, SVOCs, dioxins, metals, pesticides, herbicides and PCBs to determine the concentration of these constituents in sediments. Bioassays, using *Hyalella* and *Chironomus*, will be performed on each sediment sample to determine sediment toxicity. Bioaccumulation tests will also be performed on these sediment samples.

4.4.2 Sediment Sample Analytes, Containers, and Shipment Requirements

Required sample sizes are listed in Table 4-3 along with sample container types, preservation, and holding times. Sediment samples will be analyzed for VOCs, SVOCs, metals, pesticides, herbicides, dioxin, PCBs, TOC, pH, and grain size using methods listed in Section 7 of the QAPP. Field QC samples, including field duplicates, field rinsate blanks and trip blanks, and matrix QC, described in Section 3 of this QAPP will be collected at the frequencies summarized in Table 4-1.

Each sample container will be labeled to identify sample location, analysis required and preservation, date, and the initials of the collector. All sediment samples will be kept on ice within coolers maintained at 4°C and delivered to the appropriate laboratory within 24 hrs of collection via FedEx or similar overnight carrier. The chain-of-custody (COC) documentation will accompany all samples from the field to the laboratory (see Section 5 of the QAPP).

4.5 Benthic Invertebrate Sample Collection

Benthic invertebrates will be collected to evaluate the composition and abundance of the benthic community as part of the triad approach in the ERA. Information on the composition and abundance of benthic invertebrates will be used to evaluate potential effects of target compounds on the benthic invertebrate community. The data will be analyzed for taxa richness, abundance, percent dominant taxon/taxa, and community composition (see Section 7 of QAPP). Table 4-1 summarizes the number of samples to be collected at each location and Table 4-4 specifies the numbers of samples from each location, container specification, preservation, and holding time requirements for the benthic invertebrate samples for use in support of the ERA.

4.5.1 Benthic Invertebrate Collection for Community Evaluation

One sediment grab sample will be collected at each of the nine sampling stations in each site (three sites in the PDA; one UDA and one DDA). This will yield a total of 45 benthic invertebrate samples for community evaluation. Community analyses will be done for species identification to the lowest practical level taxon, species richness, and biomass.

Sediment samples will be collected with a Van Veen or similar large volume grab sampler using techniques described in the standard operating procedure, *Sediment Sampling* (Appendix A of the QAPP). Benthic community samples will be taken after sub-sampling for VOCs and prior to homogenization for sediment chemistry sub-sampling. Samples will be labeled using the sample identification scheme in associated site documents. Sample locations, time and date of collection, and initials of the collector will be on each sample label and the chain of custody form. This information will also be documented in a field notebook or log sheet. The samples will be preserved with 70% isopropyl alcohol and transported to the laboratory for analysis.

4.5.2 Assessment of Habitat Conditions at Benthic Sampling Location

Habitat conditions at each of the 45 sample stations (nine sample stations at each of the five sites) will be evaluated in the field and by several of the analytical measurements made in the laboratory. Qualitative field observations of sediment type, water depth, river flow velocity, width of water body, shoreline characteristics, and bordering vegetation will be recorded in a field notebook or log sheet. These observations for the Habitat Assessment will be guided by the *Physical Characterization/Water Quality Field Data Sheet and the Habitat Assessment Field Data Sheet, USEPA Rapid Bioassessment Protocols,* 1999 (Forms 1 and 2, respectively, in Appendix A-4 of the QAPP).

4.6 Bioassay Toxicity Tests

4.6.1 Surface Water Toxicity Tests

Samples for the surface water toxicity (bioassay) tests will be taken from all 9 sample stations at each of the five sites (Tables 4-1 and 4-5). At each of the 45 locations, 2.5 gallons of water (at depth) will be collected and placed into plastic bottles, labeled, and placed on ice in a cooler.

Samples will be labeled using the sample identification scheme in associated Site documents. Sample locations, time and date of collection, and initials of the collector will be on each sample label and the chain of custody form which will accompany each shipment to the laboratory. Samples will be provided to the laboratory within 24 hours of collection via FedEx or similar overnight carrier. At the laboratory, the surface water samples for toxicity testing will be refrigerated to 4° C and protected from light prior to use in testing to maintain the integrity of the original sample (Table 4-5).

The surface water toxicity tests will be used to evaluate whether chemicals in surface water within the plume discharge area, upstream of this area, and downstream of this area are toxic to benthic invertebrates. Chronic toxicity tests will be conducted at all 45 sampling stations with two indicator species, the cladoceran *Ceriodaphnia dubia* and the fathead minnow *Pimephales promeles*. The laboratory will monitor the samples and if after acute time periods 50% or more of the animals survive, chronic testing will continue for these two species; chronic testing will not continue if >50% mortality is observed. This eliminates the need to set up and run long term tests for water in which the organisms cannot survive. The acute and chronic tests are summarized in Table 4-5 and the test methods are described in Section 7 and Appendix C of the QAPP.

4.6.2 Sediment Toxicity Tests

Samples for the sediment toxicity (bioassay) tests will be taken from the same homogenized composite sediment sample that is collected from all nine sample stations at each of the five sites (Tables 4-1 and 4-5) for chemical and physical analyses. This homogenized sample will consist of the upper few inches of sediment from several grabs. A clean scoop will be used to transfer the sediment sample from the mixing bowl to the sample container. Unrepresentative material *(e.g.,* stones, wood chips) will be removed from the sample at the discretion of the field sampler and will be documented in the field log.

Approximately one gallon of sediment will be collected from each of the 45 sample stations and placed into plastic bottles, labeled, and placed on ice in a cooler. The one gallon sample will be sufficient for the bioassays for both indicator species. Samples will be labeled using the sample identification scheme in associated Site documents. Sample locations, time and date of collection, and initials of the collector will be on each sample label and the chain of custody form which will accompany each shipment to the laboratory. Samples will be provided to the laboratory within 24

hours of collection via FedEx or similar overnight carrier. At the laboratory, sediment samples for toxicity testing will be refrigerated to 4° C and protected from light prior to use in testing to maintain the integrity of the original sediment (Table 4-5).

The sediment toxicity tests will be used to evaluate whether chemicals in sediments within the plume discharge area, upstream of this area, and downstream of this area are toxic to benthic invertebrates. Chronic toxicity tests will be conducted at all 45 sampling stations with two indicator species, the amphipod *Hyalella azteca* and the midge larvae *Chironomus tentans*. The sediment toxicity tests are summarized in Table 4-5 and the test methods are described in Section 7 and Appendix C of the QAPP. In order to monitor VOC concentration changes during the test periods, sediment samples will be collected by the laboratory following the aforementioned VOC collection SOP at the beginning and the end of the bioassay for a subset of two *Hyallela* and two *Chironomus* tests.

4.6.3 Sediment Bioaccumulation Test

Samples for the sediment bioaccumulation test will be taken from the same homogenized composite sediment sample (described above) that is collected from the center sampling station on all three transects at each of the five sites (i.e., 15 samples; Tables 4-1 and 4-5) for chemical and physical analyses. Approximately one gallon of sediment will be collected from each of the 15 center sample stations and placed into plastic bottles, labeled, and placed on ice in a cooler. Samples will be labeled using the sample identification scheme in associated Site documents. Sample locations, time and date of collection, and initials of the collector will be on each sample label and the chain-of-custody form which will accompany each shipment to the laboratory. Samples will be provided to the laboratory within 24 hours of collection via FedEx or similar overnight carrier. At the laboratory, sediment samples for bioaccumulation testing will be refrigerated to 4° C and protected from light prior to use in testing to maintain the integrity of the original sediment (Table 4-5).

The bioaccumulation test provides an estimate of contaminant uptake by benthic fauna, providing information on the potential for trophic transfer of bioaccumulative contaminants to higher organisms. The bioaccumulative contaminants which will be analyzed in the test organisms include PCBs, chlorinated pesticides, and dioxins. The bioaccumulation test involves exposing the test organism, *Corbicula fulminea*, for a 28-day period.

4.7 FISH SAMPLE COLLECTION

Fish sampling will be performed at the three sites in the plume discharge area as well as the sites located upstream and downstream of plume discharge. The objectives of the fish sampling program are summarized below:

- Identify the composition and general abundance of fish in the five sites to determine if the plume discharge area is depauperate in species composition or relative abundance in comparison to the upstream and downstream locations. The upstream location may be used as a reference site;
- Examine the age structure and the weight and length relationships for species in the five locations; examine the overall health of the fish in the plume discharge area and upstream / downstream sites;
- Evaluate the habitat quality of the sampling locations with respect to supporting different fish species;
- Determine the potential of the plume discharge area for supporting recreational (game) fishing;
- Analyze the gut contents of fish in different trophic levels to document food sources of the fish species;
- Measure body burdens of chemicals in fish tissues of different trophic levels and species, for use in the ecological and human health risk assessments; and
- Examine fish for gross histopathological anomalies and compare these between the plume discharge area and upstream and downstream locations.

4.7.1 Habitat Assessment

Field assessments of the fish species and habitat present at each of the five sample sites will be made during both the Reconnaissance Event and the Main Sampling Event. General field observations made during the Reconnaissance Event will be used to finalize sample locations and methods during the Main Sampling Event.

Habitat assessment will be performed by the Ecological Project Manager and Field Team Leader in

cooperation with a qualified aquatic ecologist or fisheries biologist. These field observations of habitat and fish will be conducted in accordance with the *Fish Collection* and *Collection and Treatment of Fish Field Data* standard operating procedures and using the *Fish Sampling Field Data Sheet* provided in Appendix A of the QAPP. This assessment will be qualitative and based on a combination of experience in similar water bodies, a qualitative assessment of the physical and water quality conditions provided by the habitat, and qualitative assessment of recreational fishing potential. Information pertinent to fish populations includes: water depth, physical sediment characteristics, type and extent of submerged and emergent vegetation, area, nature of contiguous aquatic environments, general water quality conditions (turbidity, DO, conductivity, pH, and temperature), and characteristics of the shoreline. These will be documented in the field by recording observations in log books or field data sheets and by taking photographs.

The evaluation of whether the plume discharge area can support recreational fishing will be based on the results of the fish collection and discussions with commercial fishermen and qualified scientists. For example, successful collection of adult edible game fish species such as white bass would be an indication that the sampling locations contain fish that are sought for recreational fishing.

4.7.2 Fish Collection for Tissue Analysis

To support the ERA, fish exposed through different feeding strategies (trophic levels) will be collected. In addition, game fish will be collected to support the human health risk assessment pathway of ingestion (fillets). The categories will include fish from three trophic levels (bottom feeder, forager, and predator) and a game fish, which may be an equivalent species to the predator fish. Bottom fish, such as channel catfish, are in the most intimate contact with sediments and are therefore useful species to examine potential effects of sediments. Forager fish may also be impacted by sediments and bioaccumulation effects of chemicals. Predators and game fish such as buffalo are those most likely to be eaten by humans and wildlife species. Some species of fish found in the Mississippi River may represent more than one category of fish. For example, the buffalo fish represents a bottom feeder fish and might also represent a game fish for human health consumption. Fish will be collected to represent components of the food chain as follows:

Trophic Level	Target Species (size)	End Point Organism
Bottom feeder / predator	Channel catfish (10-12")	Piscivorous animals
Small forage fish	Small shad (4-8")	Heron
Large forage fish	Large shad (>8")	Osprey
Bottom Feeder / Gamefish	Buffalo (10-12")	Recreational Fishermen

The fish samples of the bottom feeder and forage fishes will be composite samples consisting of the whole bodies of three to five fish. Tissue samples from the game fish will be composites of fillets of the edible portions of one to two fish. The goal of the sampling effort is to collect three composite samples from each target group at each of the five sample sites. For each site and trophic level, each of these composite samples will undergo tissue analysis for metals, SVOCs, pesticides, herbicides, and PCBs content. One composite sample from each site will be analyzed for dioxin content. The remaining composite samples will undergo tissue analysis for QA/QC analysis (MS/MSD and QC Field Duplicates)

This fish collection and analysis scheme is tabulated at the end of this section in Tables 4-1 and 4-6. It is recognized that this goal may be difficult to meet because some of the trophic categories may not be present or fewer than three fish may be collected. In this case, the effort will focus on those fish that are collected within each of the target categories. Substitutions may be made for the expected species in each category, as necessary. The final decision on which fish to analyze will be made after the collections are complete.

Based on expected swift currents in the Mississippi River sampling locations, fish will be collected using either hoop nets (standard commercial fishing gear for the river) or other techniques used by local fishermen. Once fish are identified, length, weight, sex, species, and any gross abnormalities (e.g., lesions, tumors, and deformities) will be recorded on a fish log. Grossly deformed specimens will be photographed, preserved, labeled, and retained in a voucher collection as archived specimen. If necessary, duplicates of the voucher specimens will be collected and sent to recognized fish biology expert(s) for taxonomic confirmation. Fish samples for tissue analyses will be placed in

sealable bags and stored on ice for overnight shipment via FedEx or other shipper to the analytical laboratory where they will be maintained frozen until analysis. The SOP for filleting game fish is presented in Appendix A.

Fish stomach contents will be removed from a representative subset of collected fish in each target group; this subset will include at least one fish from each trophic level from each of the five sites. The collected stomach contents will be labeled, preserved with 70% isopropyl alcohol and transported to the laboratory for analysis.

Tables 4-2 through 4-7 in the QAPP presents the target compound lists for pesticide, dioxin, and PCB in tissue and corresponding reporting limits. The lipid content of each fish composite will be determined to provide a basis for normalizing tissue concentrations for organic constituents. In addition to the field Fish Collection Log, a Sample Processing Record for Fish compositing will be completed by the laboratory prior to sample preparation and analysis. Table 1-6 lists the number of fish samples planned for collection, and container, preservation, and holding time requirements.

5.0 SAMPLE CUSTODY

Chain-of-Custody (COC) procedures for the collection of samples in support of the Ecological Risk Assessment will follow custody protocols as described in "NEIC Policies and Procedures", *EPA-330/9-78DDI-R,* Revised June 1985. This custody is compliant with USEPA Region 5 requirements for sample custody and is divided into three parts: field-specific sample collection, laboratory custody, and final evidence files.

A sample or evidence file is under your custody if:

- the item is in your possession;
- the item is in your view, after being in your possession;
- the item is in your possession and you place it in a secured location; or
- the item is in a designated secure area.

5.1 Field Chain of Custody Procedures

The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain of custody intact.

Field Procedures

(a) The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. To preserve the integrity of the samples, as few people as possible should handle the samples.

(b) All bottles will be identified with unique sample numbers and locations on secure bottle labels. The labels will include a unique sample identification number, location, date of collection, time of collection, and type of analysis required.

(c) Sample labels are to be completed for each sample using waterproof ink. A properly completed COC form (Figure 5-1) will accompany samples. The sample numbers and locations will be listed on the COC. See Section 5.1.2 and 5.1.3 for further field custody documentation and transfer procedures.

5.1.1 Field Logbooks/Documentation

A field logbook will provide the means of recording data collecting activities performed. As such,

entries will be described in as much detail as possible so that persons going to the site could re-construct a particular situation without reliance on memory. Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel, but will stored in the document control center when not in use. The project-specific document number will identify each logbook.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned;
- Logbook number;
- Project name;
- Project start date; and
- End date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station, which includes compass and distance measurements, shall be recorded. T he number of the photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

The procedure and equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected (if applicable), amount and number of containers. Sample identification number will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

Figure 5-1 is an example COC that will be completed in the field during collection of samples. Figure 5-2 is an example Fish Log to be completed in the field for the collection of fish samples. Figure 5-3 is an example of a Sample Processing Record for Fish Compositing that will be used in the

laboratory in this form or an equivalent. Figure 5-4 is an example of the Sample Identification Label that will be used on all sample containers to identify the samples collected for the ERA.

5.1.2 Transfer of Custody and Shipment Procedures

The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the COC intact.

- A properly completed chain-of-custody (COC) form must accompany samples. The sample numbers and locations will be listed on the COC form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.
- Samples will be properly packaged for shipment, including ice to preserve the samples at ≤4°C and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and custody seals for shipment to the laboratory.
- The Chain of Custody Record identifying the contents will accompany all shipments. The original record will accompany the shipment, and copies of the COC will be retained by the field sampler for documentation. It is recommended that a copy of the COC be faxed to the laboratory on the date of collection to give the laboratory forewarning of the shipment and analytical requirements.
- If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

5.2 Laboratory Chain of Custody Procedures

Laboratory custody procedures for sample receiving and log-in; sample storage; tracking during sample preparation and analysis; and storage of data are described in the laboratory's Quality Assurance Plan (reference STL-Savannah Labs QAP dated January 2000).

5.3 Final Evidence Files Custody Procedures

The final evidence files for the data supporting the Ecological Risk Assessment will be maintained by the Facility Program Manager at Solutia. The contents of the evidence file will include, at a minimum, all relevant records, reports, correspondence, logs, field logbooks, laboratory sample preparation and analysis raw data, original laboratory data packages, pictures, subcontractor's reports including data validation reports, assessment reports, progress reports, and chain of custody records/forms. The (evidence file will be under custody of the Site Program Manager in a locked, secured area.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

All instruments used to perform chemical measurements must be properly calibrated prior and during use to ensure acceptable and valid results. The accuracy and traceability of all calibration standards used must be properly documented. This section describes the procedures necessary for maintaining the accuracy of all the instrumentation used in the field tests and the laboratory analyses. The procedures described herein are to be used in conjunction with specific instrument manufacturer's instructions, applicable analytical methodology requirements, and specific laboratory/field procedures for instrument operation.

6.1 FIELD INSTRUMENTS/EQUIPMENT

The field measurements defined for this project will include the following instrumentation: Horiba U-10 meter to measure turbidity, pH, temperature, specific conductance, and dissolved oxygen.

All field equipment for quantitative measurements will be calibrated using verified standards (traceable to appropriate NIST standards where possible), at least daily prior to initiating field activities or at the frequencies recommended by the manufacturer, whichever is more frequent. Calibrations will be checked based on the manufacturer's recommendations, following the daily calibration process. Specific procedures for field measurements are described in the Field SOPs included in Appendix A.

Balances, if used, will be calibrated with class C weights and inspected by a certified technician at least annually or at the start of the field program. Daily checks will confirm balance calibration. All instruments will be maintained and repaired in accordance with the manufacturer's specifications. Calibration procedures, including date of calibration, type of measurement, concentration of standards, and identity of field personnel performing measurement, should be recorded in the field log.

6.2 LABORATORY INSTRUMENTS

The methodologies selected for use in this investigation specify the types and frequency of calibrations. For all analytical procedures, the lowest calibration standard analyzed must be at or below the project required reporting limit for the specific media being tested to ensure accurate reporting limit determinations. The specific methods to be used are provided in Section 7.0. Appendix B contains the laboratory's SOPs detailing specifics on instrumentation and calibration

procedures. The laboratory maintains a logbook for each instrument which will contain, at a minimum, the following: instrument identification, serial number, date of calibration, analyst, identity of calibration standards run, and the samples associated with the calibration.

Accessory analytical equipment such as refrigerators, balances and ovens required for the storage and preparation of samples must be calibrated using manufacturer's instruction with the following guidelines:

- Calibration of equipment must be checked daily and these records kept in a logbook or calibration-specific log.
- The laboratory must document clearly the acceptance criteria for all such equipment (e.g., refrigerator temperature must be 4°C ±2°C and corrective actions must be taken for any out-of-control situation as described in the laboratory's quality assurance plan or manual. The equipment must not be used after corrective action until it has been recalibrated or verified through the successful analysis of a check standard.
- Calibrations of other miscellaneous analytical equipment (e.g., automatic pipettes) must be performed according to manufacturer's recommendations.

Implementation of the laboratory calibrations will be the responsibility of the Laboratory Director and the analysts performing the procedures.

7.0 ANALYTICAL PROCEDURES

This section provides a brief overview of the analytical methodologies to be used during the Sauget Area 2 sampling and analysis activities in support of the Ecological and Human Health Risk Assessments.

7.1 FIELD ANALYTICAL PROCEDURES

Field measurements of surface waters will be conducted in accordance with the methods described in Section 4 and the field measurement SOPs included in Appendix A.

At each location, water quality measurements of turbidity, dissolved oxygen, specific conductivity, temperature, and pH will be made at the surface, at mid-depth, and just above (within one foot of) the bottom. Operation and calibration of field equipment will be performed as described in Section 6.0 and the appropriate field measurement SOPs included in Appendix A.

7.2 LABORATORY ANALYTICAL PROCEDURES

Laboratory analyses will be performed by Severn Trent Laboratories - Savannah (STL-Savannah) for VOCs, SVOCs, metals, herbicides, pesticides, and PCBs and by Triangle Laboratory, for dioxin/dibenzofuran analysis. Bioassay toxicity testing of surface water and sediment will be performed by AMEC's San Diego Bioassay Laboratory and benthic invertebrate community composition will be performed by Pennington & Associates. Table 7-1 summarizes the target and non-target parameters, appropriate laboratory SOP references, and USEPA methods to be used in this investigation. Appendices B and C contain the relevant SOPs from STL - Savannah, Triangle, and Aquatec Biological Sciences, which will be employed in this investigation.

The laboratory reporting limits will be supported by a low-level standard in their calibration curves for all target compounds. Sample-specific reporting limits (i.e., RLs that include all of the sample-specific preparation factors, analysis dilution factors, and dry-weight conversion for sediments) will be reported for all non-detected target compounds. For those compounds that the laboratory reports down below the RLs down to the MDLs, the results will be flagged with a "J" indicating an estimated value. This approach will generate the lowest level reporting, using the laboratory protocols and USEPA methods described, to support the Ecological and Human Health Risk Assessment activities. However, it is anticipated, that even using the approach of reporting down to the laboratory MDLs for detected results, the achievable levels of detection in some media for some

compounds may not meet the risk-based levels of concern. The Ecological Project Manager and the human health risk assessor will ultimately decide on the usability of non-detected results at elevated reporting limits on a case-by-case basis.

A complete listing of project target compounds and project-reporting limits (RL) by matrix can be found in the tables in Section 1.0 of this QAPP. Appendix B contains the Method Detection Limits (MDLs) from STL - Savannah to support the project RLs defined for this program.

The remainder of this section describes project-specific requirements for analysis in support of the ERA and human health risk assessments. Analytical protocols are not reiterated herein, as they are detailed in the SOPs and the USEPA methods being performed.

7.2.1 Surface Water Analysis of Target Parameters

Surface water samples will be analyzed for the target compound lists for VOCs, SVOCs, metals, herbicides, pesticides, PCBs, and dioxin using the USEPA SW-846 preparation (extraction) and analysis methods listed in Table 7-1. The STL-Savannah SOPs for metals, VOC, SVOC, herbicides, pesticides, and PCBs and the Triangle Laboratory SOP for dioxins are included in Appendix B. Results for VOC, SVOC, herbicides, pesticides, and PCB target compounds in surface water will be reported in units of µg/L. Results of dioxin analysis will be reported in units of pg/L.

7.2.2 Surface Water Analysis of Non- Target Parameters

Surface water samples will be analyzed for the non-target water quality parameters in the field as described in Section 7.1 above.

7.2.3 Surface Water Bioassay (Toxicity) Methods

Surface water bioassay (toxicity) testing will be performed using surface water collected from the plume discharge area and at the reference locations upstream and downstream of this area as described in Section 4. Toxicity testing will be performed on the two indicator species: 1) *Ceriodaphnia dubia* and 2) the fish *Pimephales promeles* (Fathead Minnow).

Toxicity tests will be conducted at all 13 sampling locations with the two indicator species, in accordance with USEPA Methods presented in the SOPs (Appendix C). For stations where the results of acute toxicity tests indicate survival of 50% or more of the animals, chronic tests will also be conducted for these two species. Sequential testing (acute followed by chronic) will eliminate the need to set up and run long-term tests for surface water in which the organisms cannot survive.

7.2.4 Sediment Analysis of VOC Target Compounds

Table 7-1 lists the SOPs and USEPA methods that will be used for the preparation and analysis of sediments for target VOCs.

As discussed in Section 4.0 of this QAPP, a modified syringe/plunger-sampling device will be used for collection of the sediment samples for VOCs. For each location, a minimum of three VOC samples will be collected for use as a low-level (two samples) and high-level volatile (one sample) aliquot. The low-level samples are collected in a pre-weighed vial containing 5-mL reagent grade water and a Teflon-coated stir bar and are frozen upon receipt by STL -Savannah. The other sample (high-level) will be placed in a pre-weighed vial containing 5-mL methanol. The laboratory will ensure that the preservative completely covers the sample and will determine the weights of the vials after addition of the sample so that the exact weight of the sediment undergoing analysis is determined (this equals the initial weight of the sediment for concentration calculations). This procedure is a modification of USEPA method 5035 for collection and preservation of VOCs in soil/sediment samples.

The laboratory will analyze and report the results of the low-level volatile sample aliquots unless the samples are shown, through analysis, to contain volatiles at levels above the instrument calibration range. If volatile analytes are present in the low-level analysis at concentrations exceeding the calibration range (> 200 µg/Kg), the high-level (methanol-preserved) aliquot will also be analyzed. The laboratory must report both sets of data, low-level and high-level, if both VOC aliquots are analyzed. The data validator will determine which results represent the definitive results for use in the risk assessment in cases where both a low-level and high-level VOC analysis of sediment is performed. Results for target VOCs in sediment will be reported in units of µg/Kg dry weight.

7.2.5 Sediment Analysis of SVOCs, Metals, Herbicides, Pesticides, PCBs, and Dioxin

Table 7-I lists the SOPs and USEPA methods that will be used for the preparation and analysis of sediments for target SVOCs, metals, herbicides, pesticides, PCBs, and dioxin. As necessary, Gel Permeation Chromatography (GPC, USEPA SW-846 Method 3640A) will be used to cleanup the extracts for analysis to remove interferences from the sample matrix that may adversely impact the detection of the analytes of concern at the project reporting limits required. All target compound results in sediments must be reported on a dry-weight basis in units of µg/kg for SVOCs, metals, herbicides, pesticides, and PCBs and in ng/kg for dioxin.

7.2.6 Analysis of Non-Target Parameters in Sediment

Table 7-1 lists the USEPA methods that will be used for the analysis of TOC and grain size distribution in sediment. Percent solids will be performed based on the protocols in the USEPA methods in SW-846. TOC results will be reported on a dry-weight basis using the results of the percent solids determinations to perform the result calculations. Results for TOC in sediment will be reported in units of mg/Kg or in %TOC and grain size will be reported in %distribution of the various grain size categories.

7.2.7 Sediment Bioassay (Toxicity) Methods

Sediment bioassay (toxicity) testing will be performed using sediments collected from the plume discharge area and at the reference locations upstream and downstream of this area as described in Section 4. Toxicity testing will be performed on the two benthic indicator species: 1) the amphipod *Hyalella azteca* and 2) the midge larvae *Chironomus tentans.*

Sediment will be prepared following procedures cited in the laboratory SOPs (Appendix C).

Chronic toxicity tests (Appendix C) will be conducted for these two species unless 50% of the organisms do not survive acute exposure. This testing scheme will eliminate the need to set up and run long-term tests for sediments in which the organisms can not survive.

7.2.8 Fish Processing and Filleting Methods

All fish collected in support of the ERA will be processed as whole fish in the laboratory. Game fish will be filleted and only the edible fillet will be analyzed for input into the human health risk assessment. Procedures for filleting and processing fish tissue are included in Appendix A.

7.2.9 Fish Tissue Analysis Methods

Analysis of fish will be conducted by STL - Savannah and Triangle Laboratories in accordance with the USEPA methods summarized in Table 7-1 and the SOPs included in Appendix B. The target compounds for SVOCs, metals, herbicides, pesticides, PCBs, and dioxin in fish tissue and the project-required reporting limits in support of risk assessment are listed in tables associated with Section 1 of this QAPP. Additional guidance is provided as follows.

- Sample preparation for fish samples prior to solvent extraction will include homogenization of each sample using a tissuemizer or blender at the laboratory. This procedure will ensure a uniform sample aliquot for analysis.

- Samples that have significant matrix interferences will require specialized cleanup procedures with a reporting limit at or closer to the project required reporting limits. Gel-Permeation Chromatography (GPC USEPA SW-846 Method 3640A) may be used on solvent extracts for organic compounds prior to analysis to remove high molecular weight fatty acids and lipids. Additional cleanup procedures may be required and, if needed, will be drawn from the procedures given in USEPA SW -846, 3rd Edition. Any matrix interferences that result in elevated reporting limits without positive results for target analytes must be documented in a narrative by the laboratory.
- Fish samples will also be analyzed for lipid content, since there is a documented correlation between bioaccumulation of certain organic contaminants and the lipid content of fish.
- Results for target compounds in fish tissue will be reported on a wet-weight basis in units of µg/kg for SVOCs, metals, herbicides, pesticides, and PCBs and in units of ng/kg for dioxin.

7.2.10 Benthic Invertebrate Community Composition

The field-preserved benthic invertebrate grab samples collected for evaluation of benthic community composition will be sorted at the laboratory using techniques described in the SOP for processing of benthic invertebrate samples for taxonomic identification and community evaluation included in Appendix C. Benthic invertebrates will be identified to the lowest practical taxonomic level and counted. A voucher collection of the identified animals will be maintained. The data will be analyzed for taxa richness, abundance, percent dominant taxon/taxa, and community composition.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Field Quality Control Checks

Field Quality Control (QC) samples are collected in the field to verify the performance of sampling activities. These field QC checks are submitted to the laboratory to demonstrate the overall effectiveness of the sampling protocols. The type and frequency of field-generated QC samples are described in Section 3.0 of this QAPP. Collection of the samples will be in accordance with the applicable SOPs in Appendix A and the field sampling procedures described in Section 4 of this QAPP. Primarily, equipment rinsate blanks, trip blanks, and field duplicates are employed to verify the field sampling approach.

8.2 Laboratory Quality Control Checks

Laboratory QC checks include the analysis of initial and continuing calibration checks, internal standard responses (for organics), blanks, matrix spike (MS) and matrix spike duplicates (MSD), laboratory control samples (LCS) and standard reference materials (SRM), surrogates (organic analyses only), and cleanup check standards (for organics if cleanup is employed). Brief descriptions of these QC check samples are given below. Criteria that the laboratory must meet for these are based on the specific analytical methods used and are summarized in Tables 3-I through 3-3. Laboratory QC will be checked against the analytical methods and data usability criteria during the data generation and review process.

8.2.1 Calibration Criteria

Calibration checks will be performed according to the method-specific requirements as summarized below. The specifics for the calibrations are detailed in the individual analytical methods.

VOC and SVOC Analyses

- Instrument tuning of the GC/MS systems will be performed every I2 hours using the method-appropriate tuning standard and acceptance criteria.
- Multilevel initial calibrations (≥5-level) will be performed to establish the instrument's response to the targets of interest across a range of concentrations (calibration curves). The lowest level calibration standard must be at or below the project required reporting limit.

- Calibration verification will be performed at least once every 12 hours of gas chromatograph/mass spectrometer (GC/MS) analysis.

Calibration criteria for all other methods for analysis of target compounds including metals, herbicides, pesticides, PCBs and dioxins/dibenzofurans are described in the USEPA methods listed in Table 7-1 and in the laboratory SOPs included in Appendix B.

8.2.2 Internal Standard Responses

Internal Standards (ISs) are added to each organic sample or sample extract (for VOCs and SVOCs, respectively) just before sample analysis. The ISs used are method-specific and are either brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds. The ISs are used to normalize one sample analysis (or injection) to another and serve as a quality control indicator of the overall GC/MS instrument performance. The area response and retention time of the ISs in the calibration standards are measured and all ISs in all samples analyzed following this calibration are monitored to ensure that the responses and retention times are within method criteria.

8.2.3 Blanks

The laboratory generates method blanks that are processed along with the field samples. These method blanks are analyte-free matrices that are processed using all of the reagents and procedures that are used on the field samples to evaluate whether or not contamination occurred during sample preparation and analysis. Method blanks will be analyzed at a minimum of one per 20 field samples per matrix per preparation batch. Contamination found in the method blank and similarly in the field samples may be an indication of cross-contamination and may not be indicative of the samples taken from the field. Additional method blanks, such as cleanup method blanks, may be generated to independently verify the cleanup technique, if used. Criteria for acceptance of method blanks are method-specific and are included in Tables 3-I through 3-8.

8.2.4 Matrix Spikes and Matrix Spike Duplicates

Matrix Spike (MS) samples are prepared by spiking known concentrations of target analytes into an aliquot of field sample. The MS is processed in exactly the same manner as all other field samples. The percent recovery of a target spike compound is an indication of the ability of the methods of analysis and of the laboratory to accurately quantitate the target analyte in the sample that was spiked. The recovery of the MS may aid the analyst in determining whether a matrix effect or interference exists in the analysis of the unspiked sample. For organic analyses in particular, the

recovery of the MS does not necessarily reflect the ability to accurately determine the target analyte, or analytes of similar chemical nature, in other field samples. MS target compounds and criteria are method specific and are summarized in Tables 3-I through 3-8.

Matrix Spike Duplicates (MSD) are prepared for organic analyses and are handled in the same exact manner as the MS. The relative percent difference (RPD) is a measure of comparability between the MS and MSD and provides a measure of analytical precision. For all organic analyses, an MS/MSD pair will be prepared and analyzed at a frequency of one per 20 samples per matrix per analytical batch. RPD acceptance criteria for the MS/MSD are analyte and method specific and are summarized in Tables 3-1 through 3-8.

8.2.5 Laboratory Control Samples and Standard Reference Material

Laboratory Control Samples (LCS) are prepared by spiking known concentrations of target analytes into analyte-free matrices (blank matrices). Standard Reference Material (SRM) contains the analytes of interest in a matrix of interest and is purchased from a standard's vendor and will be used as a QC measure of accuracy for SVOCs in fish tissue. LCS and SRM QC samples are prepared and analyzed concurrently with the field samples. The recovery of the target compounds from the LCS or SRM is a measure of the ability of the preparation and analysis methods to accurately quantitate target compounds in the absence of matrix effects or interferences. LCS will be analyzed at a minimum of one per 20 field samples per matrix per preparation batch. LCS criteria are analyte and method specific and are summarized in Tables 3-1 through 3-8. The SRM criteria are based on the manufacturer's accuracy limits.

8.2.6 Surrogate Spikes

All samples, including field and QC samples, analyzed for organic components will have surrogates added to the samples during the preparation procedures. The surrogates used are method-specific and are similar in chemica1 nature to the targets of interest; however, they are not normally found in environmental samples. The recoveries of the surrogate compounds assist the analyst and data user in the determination of the accuracy of the measurements for the target compounds of interest. Tables 3-1 through 3-8 summarize the surrogate identities and criteria by method.

8.2.7 Cleanup Check Samples

Whenever a cleanup technique *(e.g.,* gel permeation chromatography (GPC), alumina column cleanup, *etc.)* is employed to eliminate interferences which may prevent accurate determination of

the targets of interest at the project required reporting limit, the cleanup procedure must be verified through the analysis of check standards. A standard containing some or all of the target compounds must be processed through the cleanup procedure and analyzed. The recovery of the target compounds in this check will indicate if the cleanup procedure was effective in elimination of interferences without undo elimination of the targets of interest. Cleanups may be needed to accurately quantitate SVOCs in sediments and tissues that have complex matrices.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

All data generated by the laboratories shall be reduced, reviewed, and validated prior to use in the Ecological Risk Assessment using the following procedures,

9.1 DATA REDUCTION

9.1.1 Field Data Reduction Procedures

Field measurements for quantitative analyses include turbidity, pH, temperature, specific conductance, and dissolved oxygen. In addition, field activities include observations and sample collection information. Raw data consist of instrument responses in the form of meter recorder, or printer output. The technician/operator performing the analysis will enter the data in a field logbook or form for each parameter. All reductions of data must follow the procedures and equations provided in the respective testing protocols (see Table 7-1). The reduction of field data will consist of summarizing the raw field data, which may be presented in the form of tables, logs, illustrations, and graphs, as deemed appropriate.

9.1.2 Laboratory Data Reduction Procedures

Laboratory data reduction procedures will be performed according to the general protocols and laboratory-specific protocols described in the laboratory Quality Assurance Plan. All raw analytical data will be recorded and documented using laboratory standard procedures. Laboratory data will include, at a minimum, the unique sample identification number, analytical method used, name of analyst, date of analysis, matrix sampled, reagent and standard concentrations, and instrument settings. The Lab QA Manager shall perform periodic review of laboratory notebooks (logbooks) and data reports.

For this project, analytical results for sediment samples will be reported on a dry-weight basis. Results for tissue (biota) analyses will be reported on a wet-weight basis. QC data (e.g., laboratory control samples, field duplicates, surrogates, MS/MSDs) will be compared to the acceptance criteria defined in this QAPP in Sections 3 and 7. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits, and any other anomalous conditions encountered during sample analysis. After the laboratory submits the laboratory data package to the Site Program Manager, the data are considered approved by the laboratory and ready for third party data review and validation.

9.2 DATA VALIDATION

Formal data validation, using standard USEPA protocols for evaluating the technical and regulatory validity of environmental data, shall be performed for the chemical data for target VOCs, SVOCs, metals, pesticides, herbicides, dioxins/dibenzofurans and PCBs generated in support of ERA and human health risk assessments. For field activities, the Field Team Leader and Ecological Risk Assessment Project Manager will review the field measurements and perform a qualitative review of observations recorded as a result of field activities. The non-target parameters in sediment, including TOC and grain size, and percent lipids in tissues, will not be formally validated.

9.2.1 Procedures Used to Validate Field Data

The procedures to evaluate field information for the Environmental Risk Assessment include checking for transcription errors and review of field logbooks. Historical data from previous investigations may be compared to the data generated during this assessment as part of the verification process. The Field Team Leader and Ecological Risk Assessment Project Manager will perform these reviews.

9.2.2 Procedures Used to Validate Laboratory Data

Procedures to validate laboratory data will follow as closely as possible the USEPA's *National Functional Guidelines for Organic Data Review* (October 1999). The data validator will modify these protocols to include the criteria in this project QAPP as listed in Sections 3 and 8.

Briefly, the validation includes a review of all technical holding times, instrument performance check sample results, initial and continuing calibration results, and all batch and matrix QC including field blanks, field duplicates, MS/MSD, surrogate recoveries, method blanks, laboratory control samples, standard reference material results, and the identification and quantitation of the target compounds of interest. One hundred percent of the analytical data shall be validated in support of using the data in the Ecological Risk Assessment.

Additionally, the analytical laboratory will perform method detection limit studies (MDL) for all chemicals of concern in all representative media. These MDLs must support the project reporting limit requirements and have been performed within one year of the analysis of samples collected for the Ecological Risk Assessment. The laboratory shall follow the MDL procedures as outlined in the Federal Register, Vol. 49, no.209, October 26, 1984, pp.198-199. Appendix B contains current MDLs for the analytes and matrices of interest from the analytical laboratory.

In addition to the precision, accuracy, and sensitivity criteria as defined in Sections 3 and 8 of this QAPP, the overall completeness of the data package will also be evaluated by the data validator. Completeness checks will be administered on all data to determine whether deliverables specified in the QAPP (Section 9.3, below) are present. The reviewer will determine whether all required items are present and request copies of missing deliverables using resubmittal request documentation via facsimile or email. Such documentation will be included in the data validation reports. Additionally, the Data Validator will determine the overall completeness of generation of valid chemical data for use in risk assessment activities.

9.3 DATA REPORTING

9.3.1 Field Data Reporting

Field data reporting for measurements are described in Sections 4.0, 5.0, 6.0, and Appendix A of this QAPP.

9.3.2 Laboratory Data Reporting

The Laboratory will provide at least two hard copies of each laboratory data report, an original and a copy for data validation, to the Site Program Manager. Electronic deliverables may be required for the project database. Specific formats for electronic deliverables shall be determined by the Site Program Manager, the Ecological Risk Assessment Project Manager, and the analytical laboratory prior to the start of the program.

The laboratory data reports shall consist of the following, at a minimum:

1. Case Narrative
    - Date of issuance
    - Laboratory analysis performed
    - Any deviations from intended analytical strategy
    - Laboratory batch number
    - Numbers of samples and respective matrices
    - QC procedures utilized and also references to the acceptance criteria

- Laboratory report contents
- Project name and number
- Condition of samples 'as-received'
- Discussion of whether or not sample holding times were met
- Discussion of technical problems or other observations which may have created analytical difficulties
- Discussion of any laboratory QC checks which failed to meet project criteria
- Signature of the Laboratory QA Manager and/or Laboratory Director or designee

2. Chemistry Data Package

- Summary page indicating dates of analyses for samples and laboratory QC checks
- Cross referencing of laboratory sample to project sample identification numbers
- Description of laboratory data qualifiers used
- Sample preparation and analyses dates and methods used for samples
- Sample results with units clearly labeled
- Raw data for sample results and laboratory QC samples
- Results of (dated) initial and continuing calibration checks, and GC/MS tuning results
- MS/MSD recoveries, laboratory control samples/standard reference, method blank results
- Calibration check compounds, system performance check results, surrogate recoveries
- Chromatograms/spectra or other raw data of sample results and QC checks
- Example result calculations

The data package submitted will be a "CLP-like" data package consisting of all the information presented in a CLP data package, including CLP-like reporting forms to facilitate data validation.

9.4 Data Reconciliation with Ecological Risk Assessment Requirements for Usability

The goal of this project is to produce an Ecological Risk Assessment. As such, the data generated must meet the risk assessor's needs as defined in Section 3 of this QAPP. In summary from Section 3, the primary objectives for assessing the usability of the data for Ecological Risk Assessment are (1) to collect data that are representative of site conditions and comparable with prior data; (2) to produce data that meet the project reporting limit requirements for Ecological Risk Assessment; (3) to produce data of the highest quality possible in order to accurately and precisely characterize the ecological conditions.

The Data Validator will apply the standard data validation qualifiers to data to indicate the level of uncertainty in the associated result. In general, for the purposes of the Ecological Risk Assessment, data that are left unqualified, data qualified "U" (non-detected), data qualified "J" (detected as an estimated result), and data qualified "UJ" (non-detected at an estimated detection reporting limit) are considered valid and usable for project objectives. Data that are qualified "R" (rejected), due to severe exceedances of QC requirements, will be considered invalid and unusable for the Ecological Risk Assessment.

In environmental sampling and analysis, some data may be lost due to sampling location logistics, field or laboratory errors, or matrix effects that may cause the rejection of results for some compounds. The overall completeness of collection of valid data, as defined in Section 3 of this QAPP, is 90%. The Data Validator will assess the completeness of the overall data generation against the project goal of producing 90% of the planned data as valid and usable results for the Ecological Risk Assessment. If this goal is not met, data gaps may exist that may compromise the risk assessment.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities may be conducted to verify that sampling and analysis are performed in accordance with the procedures established in this QAPP and the Ecological Risk Assessment Work Plan. The audits of field and laboratory activities, if performed, will include two independent parts: internal and external audits.

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

10.1.1 Internal Field Audit Responsibilities, Frequency, and Procedures

Internal audits of field activities including sampling and field observations, if performed, will be conducted by the Ecological Project Manager/Field Team Leader. These audits will verify that all established procedures are being followed.

Internal field audits may be conducted at least once at the beginning of the facility sample collection activities and potentially during the course of sampling activities if problems in the field are encountered.

Internal field audits will include examination of field sampling records, field observation records, sample collection, handling and packaging in compliance with the established procedures, maintenance of QA procedures, chain of custody, and any other procedures defined in this QAPP Sections 4 and 5. These audits may occur at the onset of the project to verify that all established procedures are followed. Follow-up audits may be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the project.

10.1.2 External Field Audit Responsibilities, Frequency, and Procedures

An external audit may be conducted by appropriate oversight QA staff of Region 5 and/or contractor. External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of USEPA Region 5.

External field audits, if performed, will be conducted according to the field activity information presented in this QAPP in Sections 4 and 5 and field activities described in the Ecological Risk Assessment Work Plan. The external field audit process may include (but not be limited to): sampling equipment decontamination procedures, sample bottle preparation procedures, sampling

procedures, examination of field sampling and safety plans, sample vessel cleanliness and QA procedures, procedures for verification of field duplicates, sample preservation and preparation for shipment, as well as field measurement procedures, as applicable.

10.2 LABORATORY PERFORMANCE AND SYSTEM AUDITS

10.2.1 Internal Laboratory Audit Responsibilities, Frequency, and Procedures

The internal laboratory audit may be conducted by the laboratory QA Officer. The internal system audits will be done on an annual basis while the internal performance audits may be conducted on a quarterly basis according to the laboratory QAPP procedures.

The internal system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc. The auditor should ensure that all Standard Operating Procedures (SOPs) and Method Detection Limits (MDL) are current and appropriate for the matrices and analyses being conducted for the project. The laboratory internal auditor will follow procedures described in the laboratory QA Plan for internal system audits.

The performance audits may involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The laboratory QA Officer will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance. The laboratory auditor will follow procedures for the performance audits as described in the laboratory QA Plan.

Data package review, as discussed in Section 10.2.2, below, may also be performed.

10.2.2 External Laboratory Audit Responsibilities, Frequency, and Procedures

An external laboratory audit may be conducted as required, by appropriate QA staff of USEPA Region 5 and/or its contractor. External laboratory audits may be conducted any time during the analytical operations. These audits may or may not be announced and are at the discretion of USEPA Region 5 oversight staff. External audits may include: review of laboratory analytical procedures; laboratory on-site visits; and results of performance evaluation samples submitted to the laboratory for analysis. Failure of any or all audit procedures chosen can lead to laboratory disqualification, and the requirement that another suitable laboratory is chosen.

An external on-site review can consist of: sample receipt procedures, custody and sample security

and log in procedures, sample storage procedures, review of instrument calibration records, instrument logs and statistics (number and type), review of QA procedures, log books, sample preparation procedures, analytical Standard Operating Procedure (SOP) review, Method Detection Limit (MDL) review, instrument reviews, personnel interviews, review of glassware preparation procedures, and corrective action protocols.

It is common practice when conducting an external laboratory audit to review one or more data packages from sample lots recently analyzed by the laboratory. This review will most likely include but not be limited to:

- Comparison of resulting data to the SOP or method, including deviations.
- Verification of initial and continuing calibrations within control limits.
- Verification of surrogate recoveries and instrument timing results, where applicable.
- Review of extended quantitation reports for comparisons of library spectra to instrument spectra, where applicable.
- Recoveries on laboratory control samples and/or SRM analyses.
- Review of run logs with run times, ensuring proper order of runs.
- Review of spike recoveries/QC sample data.
- Review of suspected manually integrated GC/MS data and its cause, where applicable.
- Assurance that samples are run within holding times.

Ideally, the data should be reviewed while on the premises, so that any data called into question can be discussed with the staff.

11.0 PREVENTATIVE MAINTENANCE

11.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

All field equipment and supplies will be routinely maintained, stocked and cared for by the field personnel. An inventory of equipment, including model and serial number, quantity, and condition will be maintained. Routine checks will be made on the status of the equipment, and spare parts will be stocked. Preventive maintenance of the equipment, as outlined in the manufacturer's instructions will be followed.

Field sampling personnel will be familiar with the calibration, operation, and maintenance of the equipment, and will perform the prescribed field operating procedures outlined in the manufacturer's instructions, in Section 4, and the field SOPs in Appendix A of this QAPP. All equipment will be inspected at least twice, once at the beginning of each day of sampling and again at the end of the days sampling. All preventive maintenance will be documented in the field logbook.

11.2 LABORATORY INSTRUMENT PREVENTATIVE MAINTENANCE

As part of the laboratory QA Plan, a routine preventative maintenance program is conducted by the laboratory to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees regularly perform routine scheduled maintenance and repair of (or coordinate with the vendor for the repair of) all instruments. All laboratory instruments are maintained in accordance with manufacturer's specifications. The preventive maintenance program should include:

- An inventory of replacement and spare parts for instruments that are maintained.
- Maintenance log books for each instrument will be kept along with information on routine and non-routine procedures. The logbook records must include the instrument number, date of maintenance activity, and the type of activity performed.
- Training of laboratory staff in the maintenance requirements of the instruments used in this project. Preventive maintenance schedules and activities will be outlined in the laboratory's SOPs and will be adhered to.

11.2.1 Inductively Couple Plasma Spectroscopy

The Inductively coupled Plasma (ICP) spectrometer should be maintained under service contract with the manufacturer. Routine preventive maintenance should include:

- Inspect pump tubing and replacement, as necessary
- Inspect nebulizer for even spray and clean, as necessary.
- Inspect torch for plasma height and shape and clean, as necessary
- Inspect photomultiplier sensitivity and replace as necessary.

11.2.2 GC/MS Instruments

The GC/MS systems will be maintained on a service contract or undergo in-house maintenance to provide routine preventive maintenance. Spare parts for the GC and GC/MS systems should include: filaments, electron multiplier, source parts, o-rings, ferrules, septa, injection port liners, and columns. Routine preventive maintenance for the systems should include:

- Checking the data systems (disk drives, tape readers, etc.) and servicing, as necessary.
- Changing oil and traps on mechanical and turbo pumps.
- Servicing the MS source through cleaning, replacement of filaments and other source parts, as necessary.
- Replacement of Injection port septa and liners, as necessary.
- Clipping front end of GC column or replacement of GC column, as necessary.

11.2.3 Thermometers

Thermometers for refrigerators and ovens are calibrated yearly against National Institute of Standards and Technology (NIST) certified thermometers. The laboratory QA manager will be responsible for the safekeeping of the NIST thermometers and for the documentation asserting the accuracy of their measurements.

11.2.4 Analytical Balances

Virtually every analytical procedure requires the use of side-loading and/or top-loading balances. Many of these requirements involve standards preparation and are, therefore, crucial to accurate

determination. Balances should be maintained on a service contract. A calibration status label is affixed to each balance after calibration during servicing.

12.0 SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The purpose of this section is to indicate the methods by which it will be ensured that the data collected for this investigation conforms with the data quality objectives (DQOs) as described in Section 3 of this QAPP. To meet these DQOs, a combination of statistical procedures and qualitative evaluations will be used to check the quality of the data. These procedures will be used by the laboratory, in generating the data, and by the data validator, in the validation of the results for ultimate use in the Ecological Risk Assessment.

Results for QC samples, including field and laboratory blanks, spikes, and duplicates as previously described in Sections 3, 6, and 8 of this QAPP, will be evaluated using the equations described below to determine the validity and usability of the data. In addition, the data will be reviewed for indications of interferences to results caused by sample matrices, contamination during sampling, contamination in the laboratory, and sample preservation and storage anomalies (i.e., samples holding time or analytical instrument problems).

12.1 PRECISION ASSESSMENT

The relative percent difference (RPD), as a measure of variability, between the matrix spike and matrix spike duplicate for organics (VOCs, SVOCs, metals, pesticides, herbicides, dioxins/dibenzofurans, and PCBs) and field duplicate pair results for VOC, SVOC, TOC, and grain size analyses will be calculated to compare to precision and representativeness DQOs. The RPD of duplicate measurements is calculated according to the following formula:

$$RPD = \frac{(\text{Result in sample 1} - \text{Result in sample 2}) \times 100}{[(\text{Result in sample 1} + \text{Result in sample 2}) / 2]}$$

where:

Sample 1 = initial sample or spiked sample result

Sample 2 = duplicate sample or duplicate spiked sample result.

12.2 ACCURACY ASSESSMENT

Accuracy, as a measure of bias, will be evaluated based on the percent recoveries of the matrix spike sample, matrix spike duplicate sample, surrogates (organics), internal standards, laboratory

control samples and/or standard reference materials, initial and continuing calibration check samples. These QC results will be compared to the project DQOs for accuracy.

The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. Percent recoveries for spiked samples and QC are determined using the following equation:

$$\%\ R = \frac{(\text{Result in spiked sample} - \text{Result in original (unspiked) sample}) \times 100}{\text{Known amount of spike added}}$$

Percent recoveries for LCS and SRM are determined using the following equation:

$$\%\ R = \frac{\text{Result for compound in LCS or SRM} \times 100}{\text{Verified amount of compound in LCS or SRM}}$$

Additionally, field and laboratory blanks will be used to evaluate whether field or laboratory procedures represent a possible source of contamination in the samples. Unmonitored contamination can allow false positive results to be reported and treated as true sample components when, in fact, they are not. This type of error will adversely affect the accuracy of the reported results. Several types of blanks, including field blanks, method blanks, and instrument blanks, will be used in this project as described in Sections 3,6, and 8.

Specific DQOs for blanks have been defined for this program in Sections 3, 6, and 8. In general, the procedure for assessing blank samples for potential contamination is as follows:

- Tabulate blank compound results.
- Identify blank samples for which compounds are reported above the project-required reporting limits.
- If no compounds are detected above the reporting limits in any blanks, the associated data are reported unqualified and no blank actions are taken.
- If compounds are detected above the reporting limits in the blanks, the associated sample compounds will be qualified during data validation. This qualification may result in the negation of results at raised reporting limits due to blank actions.

12.3 COMPLETENESS ASSESSMENT

Completeness is the ratio of the number of valid sample results to the total number of results planned for collection. Following completion of the sampling, analysis, and data validation, the percent completeness will be calculated and compared to the project DQO of ≥ 90% (Section 3 of this QAPP) using the following equation:

$$\% \text{ Completeness} = \frac{\text{number of valid (usable) results obtained}}{\text{number of valid/usable results planned}} \times 100$$

12.4 OVERALL ASSESSMENT OF ECOLOGICAL DATA

Data assessment will involve Data Validation and usability to determine if the data collected are of the appropriate quality, quantity and representativeness to support the Ecological Risk Assessment. The effect of the loss of data deemed unacceptable for use, for whatever reason, will be discussed and decisions made on corrective action for potential data gaps. The QC results associated with each analytical parameter for each sample will be compared to the objectives presented in Sections 3, 6, and 8 of this QAPP. Only data generated in association with QC results meeting these objectives and the data validation criteria will be considered usable for the Ecological Risk Assessment.

Factors to be considered in the overall data assessment based on the DQOs in this QAPP and the data validation by the third-party validator will include, but not necessarily be limited to, the following.

- Were all samples obtained using the methodologies and SOPs proposed in the QAPP?
- Were all proposed analyses performed according to the SOPs provided in the QAPP?
- Were samples obtained from all proposed sampling locations planned?
- Do any analytical results exhibit elevated detection limits due to matrix interferences or contaminants present at high concentrations?
- Were all laboratory data validated according to the validation protocols, including project-specific QC objectives as defined in this QAPP?
- Which data sets were found to be unusable (qualified as "R") based on the data

validation results?

- Which data sets were found to be usable as estimated data, (qualified as "J" or "UJ") based on the data validation results?
- Has sufficient data of appropriate quality been generated to support the Ecological Risk Assessment?
- Were all issues requiring corrective action, if any, fully resolved?
- Have any remaining data gaps been identified and summarized in the final report?

13.0 CORRECTIVE ACTIONS

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out of QC performance which can affect data quality and usability. Corrective actions may be required for two classes of problems: analytical and equipment problems and noncompliance problems. Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrumental analysis, and data review.

For noncompliance problems (for example, non-compliance with USEPA methods or QC defined in this QAPP), a formal corrective action will be implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Site Project Manager. A description of the problem and the corrective action implemented will be confirmed in writing via email, facsimile, or technical memorandum. Any nonconformance with the established quality control procedures in this QAPP will be identified and corrected. Corrective actions in the field will be implemented and documented in the field record book.

13.1 FIELD CORRECTIVE ACTIONS

Technical staff and field project personnel will be responsible for reporting all suspected technical or QA nonconformance or suspected deficiencies of any field collection or observation activity by reporting the situation to the Site Program Manager or designee. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the field personnel.

The Site Program Manager is ultimately responsible for ensuring that corrective action for nonconformance are initiated by:

- evaluating all reported nonconformance.
- controlling additional work on nonconforming items.
- determining disposition or action to be taken.
- maintaining a log of nonconformances.
- reviewing nonconformance reports and corrective actions taken.
- ensuring nonconformance reports are included in the final site documentation in project

files. If appropriate, the Site Program Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

If a corrective action warrants a change in the program protocols, this change will be documented and signed by the Field Team Leader for the Ecological Risk Assessment and the Site Program Manager.

13.2 LABORATORY CORRECTIVE ACTIONS

The laboratory participating in this program is required to have written SOPs specifying corrective actions to be taken when an analytical error is discovered or the analytical system is determined to be out of control. The SOP requires documentation of the corrective action and notification by the analyst about the errors and corrective procedures.

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is dependent on the analysis and the event. Laboratory corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy.
- Blanks contain compounds of interest, as listed in tables in Section 1 of this QAPP, above acceptable levels.
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits.
- Deficiencies are detected by the Laboratory QA Department during internal or external audits or from the results of performance evaluation samples.
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA department.

Corrective actions may include:

- Re-analyzing the samples, if holding time criteria permits.
- Resampling and analyzing.
- Evaluating and amending analytical procedures.
- Accepting data and acknowledging the level of uncertainty as documented in the laboratory data package case narrative.

If resampling is deemed necessary due to laboratory problems, the Site Program Manager must identify the necessary approach, including cost recovery, for the additional sampling effort.

13.3 DATA VALIDATION AND DATA ASSESSMENT CORRECTIVE ACTIONS

During the data validation process, the data validator may determine the need for corrective action based on the observed usability of the data. Any situation which results in rejected (qualified as "R" during validation) data will be brought to the immediate attention of the Site Program Manager to determine if critical data gaps exist which may warrant resampling and/or, if within holding time, reanalysis of the samples. Corrective action may also be deemed necessary based on the overall assessment of the data collected and not related to rejection of analytical results *(e.g.,* inability of the laboratory to perform all of the analytical tests required for a particular sample causing a critical data gap). The Site Program Manager will be responsible for approving the implementation of corrective action, including resampling, during the data assessment. All corrective actions of this type will be documented.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The deliverables associated with the tasks identified in the Ecological Risk Assessment Workplan will contain an uncertainty evaluation in which data quality information collected during the task is summarized. The Ecological Risk Assessment report will include the results of the data validation of the samples as a documentation of the quality of the data collected for assessing ecological risk.

The Ecological Risk Assessment report will contain information generated during the project on the achievement of project-specific DQOs, uncertainties in the data, the effect of these uncertainties on the risk assessment, and a summary of corrective actions implemented, as necessary.

15.0 REFERENCES

USEPA. 1994. Contract laboratory program : National functional guidelines for organic data review. EPA-540/R-94/082.

US EPA. 1994. Guidance for the data quality objectives process. EPA QA/G-4.

USEPA. 1996. Biological Criteria: Technical Guidance for Streams and Small Rivers - Revised Edition. EPA-822/B-96-001.

USEPA. 1999. Data Validation Standard Operating Procedures for Contract Laboratory Program. Routine Analytical Services, Revision 2.1. July, 1999.

Table 1-1. Target Volatile Organic Compound Analytical Parameters and Reporting Limits in Support of the Ecological Risk Assessment

Target Compound List VOCs	CAS Number	Surface Water RL ug/L (ppb)	Sediment RL[1] ug/kg (ppb)
Chloromethane	74-87-3	2	2*
Bromomethane	74-83-9	2	2*
Vinyl Chloride	75-01-4	2	2
Chloroethane	75-00-3	2	2
Methylene Chloride	75-09-2	5	5
Acetone	67-64-1	25	25
Carbon Disulfide	75-15-0	2	2
1,1-Dichloroethylene	75-35-4	2	2
1,1-Dichloroethane	75-34-3	2	2*
cis-1,2-Dichloroethene	156-59-4	2	2
Trans-1,2-Dichloroethene	156-60-5	2	2
Chloroform	67-66-3	2	2
1,2-Dichloroethane	107-06-2	2	2
2-Butanone	78-93-3	10	10
I,1,1-Trichloroethane	71-55-6	2	2
Carbon Tetrachloride	56-23-5	2	2
Bromodichloromethane	75-27-4	2	2*
1,2-Dichloropropane	78-87-5	2	2
cis-1,3-Dichloropropene	10061-01-5	2	2
Trichloroethene	79-01-6	2	2
Dibromochloromethane	124-48-1	2	2
1,1,2-Trichloroethane	79-00-5	2	2
Benzene	71-43-2	2	2
Trans-1,3-Dichloropropene	10061-02-6	2	2
Bromoform	75-25-2	2	2
4-Methyl-2-pentanone	108-10-1	10	10
2-Hexanone	591-78-6	10	10
Tetrachloroethene	127-18-4	2	2
1,1,2,2-Tetrachloroethane	79-34-5	2	2
Toluene	108-88-3	2	2
Chlorobenzene	108-90-7	2	2
Ethylbenzene	100-41-4	2	2*
Styrene	100-42-5	2	2
Xylenes (total)	1330-20-7	2*	2

[1]Sediments must be reported on a dry-weight basis. The project reporting limits (RLs) are nominal limits assuming 100% solids, Sample-specific RLs will vary based on the % solids of the sediment sample. The reporting limit for sediments is dependent upon the sample preservation technique used. Low-level Method 5035 is planned; RL = 2-5 µg/kg. If High-level preservation is used for any samples, the RL would be in 50 times higher than shown (100-250 µg/kg).

*Project Reporting Limit is higher than the Region 5 Ecological Data Quality Level (EOQL) or other Ecological RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data. Wherever possible, the laboratory will report estimated results for detected compound between their MDL and the RL.

Table 1-2. Target Semivolatile Organic Analytical Parameters and Reporting Limits in Support of the Ecological and Human Health Risk Assessments

Target Compound List SVOCs	CAS Number	Surface Water RL ug/L (ppb)	Sediment RL[1] ug/kg (ppb)	Biota RL[2] ug/kg (ppb)
Phenol	108-95-2	5	170*	1000
bis-(2-Chloroethyl)ether	111-44-4	5	170	1000**
2-Chlorophenol	95-57--8	5	170*	1000
1,3-Dichlorobenzene	541-73-1	5	170	1000
1,4-Dichlorobenzene	106-46-7	5	170	1000**
1,2-Dichlorobenzene	95-50-1	5	170	1000
2-Methylphenol	95-48-7	5	170*	1000
2,2'-oxybis(1-chloropropane)	108-60-1	5	170*	1000**
4-Methylphenol	106-44-5	5	170*	1000
N-Nitroso-di-n-propylamine	621-64-7	5	170*	1000**
Hexachloroethane	67-72-1	5	170	1000
Nitrobenzene	98-95-3	5	170	1000
Isophorone	78-59-1	5	170	1000
2-Nitrophenol	88-75-5	5	170*	1000
2,4-Dimethylphenol	105-67-9	5	170	1000
bis(2-Chloroethoxy)methane	111-91-1	5	170	1000
2,4-Dichlorophenol	120-83-2	5	170.	1000
1,3,5-Trichlorobenzene	108-70-3	5	170	1000
Naphthalene	91-20-3	5	170*	1000
4-Chloroaniline	106-47-8	10	330	2000
Hexachlorobutadiene	87-68-3	5*	170	1000**
4-Chloro-3-methylphenol	59-50-7	5	170	1000**
2-Methylnaphthalene	91-57-6	5	170*	1000
Hexachlorocyclopentadiene	77-47-4	5	170	1000
2,4,6-Trichlorophenol	88-06-2	5*	170.	1000
2,4,5-Trichlorophenol	95-95-4	5	170*	1000
2-Chloronaphthalene	91-58-7	5*	170	1000
2-Nitroaniline	88-74-4	25	830*	5000
Dimethylphthalate	131-11-3	5	170*	1000
Acenaphthylene	208-96-8	5	170*	1000
2,6-Dinitrotoluene	606-20-2	5	170*	1000
3-Nitroaniline	99-09-2	25	830*	5000
Acenaphthene	83-32-9	5	170*	1000
2,4-Dinitrophenol	51-28-5	25*	830*	5000
4-Nitrophenol	100-02.7	25	830*	5000
Dibenzofuran	132-64-9	5	170	1000
2,4-Dinitrotoluene	121-14-2	5	170*	1000
Diethylphthalate	84-66-2	5*	170*	1000
4-Chlorophenylphenylether	7005-72-3	5	170	1000
Fluorene	86-73-7	5*	170*	1000
4-Nitroaniline	100-01-6	25	830*	5000
4,6-Dinitro-2-methylphenol	534-52-1	25*	830*	5000
N-Nitrosodiphenylamine	86-30-6	5	170*	1000

Table 1-2. Semivolatile Organic Analytical Parameters and Reporting Limits in Support of the Ecological and Human Health Risk Assessments *(continued)*

Target Compound List SVOCs	CAS Number	Surface Water RL ug/L (ppb)	Sediment RL[1] ug/kg (ppb)	Biota RL[2] ug/kg (ppb)
4-Bromophenylphenylether	101-55-3	5*	170	1000
Hexachlorobenzene	118-74-1	5*	170*	1000**
Pentachlorophenol	87-86-5	25*	830	5000**
Phenanthrene	85-01-8	5*	170*	1000
Anthracene	120-12-7	5*	170*	1000
Carbazole	86-74-8	5	170	1000**
Di-n-butylphthalate	84-74-2	5*	170*	1000
Fluoranthene	206-44-0	5	170*	1000
Pyrene	129-00-0	5*	170*	1000
Butylbenzylphthalate	85-68-7	5	170	1000
3,3'-Dichlorobenzidine	91-94-1	10	660*	2000**
Benzo (a) anthracene	56-55-3	5*	170*	1000**
Chrysene	218-01-9	5*	170*	1000
bis(2-Ethylhexyl)phthalate	117-81-7	5*	170	1000
Di-n-octylphthalate	117-84-0	5	170	1000
Benzo (b) fluoranthene	205-99-2	5	170	1000**
Benzo(k)fluoranthene	207-08-9	5*	170	1000**
Benzo (a) pyrene	50-32-8	5*	170*	1000**
Indeno (1,2,3-cd) pyrene	193-39-5	5*	170	1000**
Dibenzo (a,h) anthracene	53-70-3	5*	170*	1000**
Benzo (g,h,i) perylene	191-24-2	5	170	1000

[1]Sediments must be reported on a dry-weight basis. The project reporting limits (RLs) are nominal limits assuming 100% solids using Method 8270C in the EI mode of operation (full scan mode). Sample-specific RLs will vary based on the % solids of the sediment sample.

[2]Biota results must be reported on a wet-weight basis. Percent solids will not be performed on tissue samples. The project reporting limits (RLs) are nominal limits using Method 8270C in the EI mode of operation (full scan mode). These values are revised based on information obtained from the lab on 5/16/01.

*Project Reporting Limit is higher than the Region 5 Ecological Data Quality Level (EDQL) or other Ecological RBC; however. This is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data. Wherever possible, the laboratory will report estimated results for detected compound between their MDL and the RL.

**Project Reporting Limit is above the Human Health RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data.

Table 1-3. Pesticide Parameters and Reporting Limits in Support of the Ecological and Human Health Risk Assessments

Target Compound List Pesticides	CAS Number	Surface Water RL ug/L (ppb)	SedimentRL[1] ug/kg (ppb)	Biota RL[2] ug/kg (ppb)
alpha-BHC	319-84-6	0.025	0.85	5**
beta-BHC	319-85-7	0.025	0.85	5**
delta-BHC	319-36-8	0.025	0.85	5**
gamma-BHC (lindane)	58-89-9	0.025*	0.85	5**
Heptachlor	76-44-8	0.05*	1.7*	5**
Aldrin	309-00-2	0.5	1.7	5**
Heptachlor epoxide	1024-57-3	0.05*	1.7*	5**
Endosulfan I	959-98-8	0.05*	1.7*	5
Dieldrin	60-57-1	0.1*	3.3*	5**
4,4'-DDE	72-55-9	0.1*	3.3*	5
Endrin	72-20-8	0.1*	3.3*	5
Endosulfan II	33213-65-9	0.1*	3.3*	5
4,4'-DDD	72-54-8	0.1*	3.3	5
Endosulfan sulfate	1031-07-8	0.1	3.3	5
4,4'-DDT	50-29-3	0.1*	3.3*	5
Methoxychlor	72-43-5	0.5*	17*	20
Endrin Ketone	53494-70-5	0.1	3.3	5
Endrin Aldehyde	7421-36-3	0.1	3.3*	5
alpha-Chlordane	5103-71-9	0.05	1.7	5
gamma-Chlordane	5103-74-2	0.05	1.7	5
Toxaphene	8001-35-2	5*	170*	170**

[1]Sediments must be reported on a dry-weight basis. The project reporting limits (RLs) are nominal limits assuming 100% solids. Sample-specific RLs will vary based on the % solids of the sediment sample.
[2]Biota results must be reported on a wet-weight basis. Percent solids will not be performed on tissue samples.
*Project Reporting Limit is higher than the Region 5 Ecological Data Quality Level (EDQL) or other Ecological RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data. Wherever possible, the laboratory will report estimated results for detected compound between their MDL and the RL.
**Project Reporting Limit is above the Human Health RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data.

Table 1-4. Polychlorinated Biphenyl Parameters and Reporting Limits in Support of the Ecological and Human Health Risk Assessments

Target Compound List PCBs	CAS Number	Surface Water RL ug/L (ppb)	Sediment RL[2] ug/kg (ppb)	Biota RL[2] ug/kg (ppb)
Monochlorobiphenyls	NA	0.1	3.3	10
Dichlorobiphenyls	NA	0.1	3.3	10
Trichlorobiphenyls	NA	0.1	3.3	10
Tetrachlorobiphenyls	NA	0.2	6.7	20
Pentachlorobiphenyls	NA	0.2	6.7	20
Hexachlorobiphenyls	NA	0.2	6.7	20
Heptachlorobiphenyls	NA	0.3	10	30
Octachlorobiphenyls	NA	0.3	10	30
Nonachlorobiphenyls	NA	0.5	17	50
Decachlorobiphenyls	NA	0.5	17	50

[1]PCBs will be analyzed by US EPA method 680 (Preparation method 3510/3520)
[2]Sediments must be reported on a dry-weight basis The project reporting limits (RLs) are nominal limits assuming 100% solids. Sample-specific RLs will vary based on the % solids of the sediment sample.
*Project Reporting Limit is higher than the Region 5 Ecological Data Quality Level (EDQL) or other Ecological RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data. Wherever possible, the laboratory will report estimated results for detected compound between their MOL and the RL.
**Project Reporting Limit is above the Human Health RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data.

Table 1-5. Herbicide Parameters and Reporting Limits in Support of the Ecological and Human Health Risk Assessments

Target Compound List Herbicides	CAS Number	Surface Water RL ug/L (ppb)	Sediment RL[1] ug/kg (ppb)	Biota RL[2] ug/kg (ppb)
2,4-D	94-75-7	0.5	8.3*	25
2,4-DB	94-82-6	0.5	8,3	25
2,4,5-TP (Silvex)	93-72-1	0.5	8.3	25
2,4,5-T	93-76-5	0.5	8.3	25
Dalapon	75-99-0	120	2000	6000
Dicamba	1918-00-9	1.2	20	60
Dichloroprop	120-36-5	6	100	300
Dinoseb	88-85-7	6*	100*	300
MCPA	94-74-6	120	2000	6000**
MCPP	93-65-2	120	2000	6000**

[1]Sediments must be reported on a dry-weight basis. The project reporting limits (RLs) are nominal limits assuming 100% solids. Sample-specific RLs will vary based on the % solids of these sediment sample.
[2]Biota results must be reported on a wet-weight basis. Percent solids will not be performed on tissue samples.
*Project Reporting Limit is higher than the Region 5 Ecological Data Quality Level (EDQL) or other Ecological RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data. Wherever possible, the laboratory will report estimated results for detected compound between their MDL and the RL.
**Project Reporting Limit is above the Human Health RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data.

Table 1-6. Dioxin and Dibenzofuran Parameters and Reporting Limits in Support of the Ecological and Human Health Risk Assessments

Target Compound List Dioxins and Dibenzofurans	CAS Number	Surface Water RL[3] pg/L (ppq)	Sediment RL[1,3] ng/kg (ppt)	Biota RL[2,3] ng/kg (ppt)
2,3,7,8-TCDD	1746-01-6	10*	1	1**
1,2,3,7,8-PeCDD	40321-76-4	50	5	5**
1,2,3,4,7,8-HxCDD	39227-28-6	50	5	5**
1,2,3,6,7,8-HxCDD	57653-85-7	50	5	5**
1,2,3,7,8,9-HxcDD	19408-74-3	50	5	5**
1,2,3,4,6,7,8-HpCDD	35822-46-9	50	5	5**
1,2,3,4,5,6,7,8-0CDD	3268-87-9	100	10	10**
2,3,7,8-TCDF	51207-31-9	10	1*	1**
1,2,3,7,8-PeCDF	57117-41-6	50	5	5**
2,3,4,7,8-PeCDF	57117-31-4	50	5	5**
1,2,3,4,7,8-HxCDF	70648-26-9	50	5	5**
1,2,3,6,7,8-HxCDF	57117-44-9	50	5	5**
1,2,3,7,8,9-HxCDF	72918-21-9	50	5	5**
2,3,4,6,7,8-HxCDF	60851-34-5	50	5	5**
1,2,3,4,6,7,8-HpCDF	67562-39-4	50	5	5**
1,2,3,4,7,8,9-HpCDF	55673-89-7	50	5	5**
1,2,3,4,5,6,7,8-0CDF	39001-02-0	100	10	10**
Total TCDD	41903-57-5	10	1	1
Total PeCDD	36088-22-9	50*	5*	5
Total HxCDD	34465-46-8	50	5	5
Total HpCDD	37871-00-4	50	5	5
Total TCDF	55722-27-5	10	1	1
Total PeCDF	30402-15-4	50	5	5
Total HxCDF	55684-94-1	50	5	5
Total HpCDF	38988-75-3	50	5	5

[1]Sediments must be reported on a dry-weight basis. The project reporting limits (RLs) are nominal limits assuming 100% solids. Sample-specific RLs will vary based on the % solids of the sediment sample.
[2]Biota results must be reported on a wet-weight basis. Percent solids will not be performed on tissue samples. Percent Lipids determination will be performed as part of the Dioxin analysis, Method 8290.
[3]For the Dioxin and Dibenzofuran analyses, Triangle Laboratory will report estimated detection limits (EDLs) for non-detected analytes through measurement of the signal noise level at the quantitation ions for each analyte and calculate an EDL based on these measurements. These EDLs are generally much lower than the RLs given above, which are based on the calibration standards analyzed.
*Project Reporting Limit is higher than the Region 5 Ecological Data Quality Level (EDQL) or other Ecological RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data. Wherever possible, the laboratory will report estimated results for detected compound between their MDL and the RL.
**Project Reporting Limit is above the Human Health RBC; however, this is the lowest level of reporting possible for the method of analysis. As a conservative estimate of risk, one-half of the reporting limit will be used in the risk assessment for non-detected data.

Table 1-7. Inorganic (Metals) Parameters and Reporting Limits in Surface Water, Sediment, and Biota in Support of the Ecological Risk Assessment

Target Compound List Inorganics	CAS Number	Surface Water RL[3] ug/L (ppb)	Sediment RL[1,3] mg/kg (ppm)	Biota RL[2,3] mg/kg (ppm)
Aluminum	7429-90-5	200	20	20
Antimony	7440-36-0	20	2.0	5.0
Arsenic	7440-38-2	10	1.0	1.0
Beryllium	7440-41-7	4.0	0.4	0.4
Cadmium	7440-43-9	5.0	0.5	0.5
Chromium, trivalent	7440-47-3	10	1.0	1.0
Chromium, hexavalent		10	0.4	-
Copper	7440-50-8	20	2.0	2.0
Iron	7439-89-6	50	5.0	5.0
Lead	7439-92-1	5.0	0.5	0.5
Nickel	7440-02-0	2.0	4.0	4.0
Selenium	7782-49-2	10	1.0	1.0
Silver	7440-22-4	10	1.0	1.0
Zinc	7440-66-6	20	2.0	2.0

[1]Sediments must be reported on a dry-weight basis. The project reporting limits (RLs) are nominal limits assuming 100% solids. Sample-specific RLs will vary based on the % solids of the sediment sample.
[2]Biota results must be reported on a wet-weight basis.
[3]RLs are from Savannah Lab QC manual, dated 01/31/00.

Table 1-8. Water Quality and Other Non-Target Parameters and Sample Media in Support of the Ecological Risk Assessment

	Media for Environmental Analyses		
Parameter	**Surface Water**	**Sediment**	**Biota (Fish)**
Turbidity	X	NA	NA
Conductivity	X	NA	NA
Dissolved Oxygen	X	NA	NA
pH	X	X	NA
Temperature	X	NA	NA
Percent Solids	NA	X	NA
Grain Size	NA	X	NA
Total Organic Carbon (TOC)	NA	X	NA
Percent Lipids	NA	NA	X
Benthic Community Evaluation	NA	X	NA
Bioassay Toxicity Tests (Acute and Chronic with two indicator species per medium)	X	X	NA
Hardness	X	NA	NA

NA-Measurement of this parameter is not applicable to this media.

Table 3-1. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Volatile Organic Compound Analyses of Surface Water Samples

Parameter	Matrix	QC Compounds	Field /Matrix Duplicate Precision (RPD)[a]	MS/MSD Precision (RPD)	Blanks	MS[a,b] & MSD Accuracy (% Recovery)	Surrogate Accuracy[a,b] (% Recovery)
Volatile Organic Compounds	Surface Water	All analytes	≤ 30		≤ 5x RL for acetone, methylene chloride, and 2-butanone; <RL for all other analytes		
		1,1-dichloroethene		≤ 30		46-147	
		trichloroethene		≤ 35		56-143	
		benzene		≤ 37		62-135	
		toluene		≤ 33		68-131	
		chlorobenzene		≤ 22		72-127	
		toluene-d_8					77-122
		bromofluorobenzene					74-126
		1,2-dichloroethane-d_4					70-130
Volatile Organic Compounds	Sediments	All analytes	≤ 50				
		1,1-dichloroethene		≤ 46	≤ 5x RL for acetone, methylene chloride, and 2-butanone; <RL for all other analytes	40-164	
		trichloroethene		≤ 34		51-146	
		benzene		≤ 42		49-142	
		toluene		≤ 32		38-158	
		chlorobenzene		≤ 34		66-135	
		toluene-d_8					64-136
		bromofluorobenzene					63-135
		1,2-dochloroethane-d_4					

Notes:

RL = Project Reporting Limit

[a]Provisions for wider acceptance limits near the RL may be based on professional judgment during data review/validation.

[b]Limits are based on those from STL-Savannah, effective 01/31/00.

Table 3-2a. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Semi-volatile Organic Compound Analyses of Surface Water Samples

Parameter	QC Compounds	Field /Matrix Duplicate Precision[a,c] (RPD)	MS/MSD Precision (RPD)	Blanks	MS[a,b] & MSD Accuracy (% Recovery)	Surrogate Accuracy[a,b] (% Recovery)
Semivolatile Organic Compounds	All analytes	≤ 50		≤ 5x RL for phthalates ≤ RL for all others		
	phenol		≤ 36		33-122	
	2-chlorophenol		≤ 34		38-115	
	1,4-dichlorobenzene		≤ 31		27-130	
	N-nitroso-di-n-propylamine		≤ 30		31-138	
	1,2,4-trichlorobenzene		≤ 28		28-110	
	p-chloro-m-cresol		≤ 31		34-126	
	acenapthene		≤ 35		36-121	
	4-nitrophenol		≤ 44		12-143	
	2,4-dinitrotoluene		≤ 32		37-129	
	pentachlorophenol		≤ 33		19-148	
	pyrene		≤ 42		31-139	
	nitrobenzene-d_5					34-130
	2-fluorobiphenyl					36-124
	terphenyl-d_{14}					14-148
	phenol-d_5					25-128
	2-fluorophenol					29-121
	2,4,6-tribromophenol					29-143
	2-chlorophenol-d_4					20-130*
	1,2-dichlorobenzene-d_4					20-130*

Notes

General: All method requirements for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Method 8270C), must be followed

* Advisory Limits Only

[a] Provision for wider acceptance limits near the RL may be based on professional judgement during data review/validation

[b] Limits are based on those from STL-Savannah, effective 01/31/00

[c] Field duplicate precision based on technical judgement and USEPA National Functional Guidelines for duplicate precision

Table 3-2b. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Semi-volatile Organic Compound Analyses of Sediment Samples

Parameter	QC Compounds	Field /Matrix Duplicate Precision (RPD)[a]	MS/MSD Precision (RPD)	Blanks	MS[a,b] & MSD Accuracy (% Recovery)	Surrogate Accuracy[a,b] (% Recovery)
Semivolatile Organic Compounds	All analytes	≤ 50		≤ 5x RL for phthalates ≤ RL for all others		
	phenol		≤ 39		13-115	
	2-chlorophenol		≤ 39		15-111	
	1,4-dichlorobenzene		≤ 31		10-105	
	N-nitroso-di-n-propylamine		≤ 37		11-122	
	1,2,4-trichlorobenzene		≤ 22		10-112	
	p-chloro-m-cresol		≤ 32		24-114	
	acenapthene		≤ 49		18-123	
	4-nitrophenol		≤ 57		15-118	
	2,4-dinitrotoluene		≤ 37		11-120	
	Pentachlorophenol		≤ 55		10-140	
	Pyrene		≤ 42		10-133	
	nitrobenzene-d_5					20-120
	2-fluorobiphenyl					30-120
	terphenyl-d_{14}					30-131
	phenol-d_5					19-114
	2-fluorophenol					16-113
	2,4,6-tribromophenol					23-129

Notes

General: All method requirements for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Method 8270C), must be followed

* Advisory Limits Only

[a] Provision for wider acceptance limits near the RL may be based on professional judgement during data review/validation

[b] Limits are based on those from STL-Savannah, effective 01/31/00.

[c] Field duplicate precision based on technical judgement and USEPA National Functional Guidelines for duplicate precision

Table 3-2c. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Semi-volatile Organic Compound Analyses of Biota Samples.

Parameter	QC Compounds	Field /Matrix Duplicate Precision (RPD)[a]	MS/MSD Precision (RPD)	Blanks	MS[a,b] & MSD Accuracy (% Recovery)	Surrogate Accuracy[a,b] (% Recovery)
Semivolatile Organic Compounds	All analytes	≤ 50		≤ 5x RL for phthalates ≤ RL for all others		
	phenol		≤ 25		17-103	
	2-chlorophenol		≤ 25		23-114	
	1,4-dichlorobenzene		≤ 40		10-125	
	N-nitroso-di-n-propylamine		≤ 35		11-117	
	1,2,4-trichlorobenzene		≤ 28		17-105	
	p-chloro-m-cresol		≤ 25		25-107	
	acenapthene		≤ 25		28-102	
	4-nitrophenol		≤ 45		10-117	
	2,4-dinitrotoluene		≤ 30		26-107	
	Pentachlorophenol		≤ 44		10-120	
	Pyrene		≤ 25		18-136	
	nitrobenzene-d_5					12-125
	2-fluorobiphenyl					24-118
	terphenyl-d_{14}					18-153
	phenol-d_5					10-142
	2-fluorophenol					10-118
	2,4,6-tribromophenol					14-121

Notes:
General: All method requirements for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Method 8270C), must be followed
* Advisory Limits Only
[a] Provision for wider acceptance limits near the RL may be based on professional judgement during data review/validation
[b] As required by EPA SW846 Method 8270C, these criteria limits represent the lab-specific limits for accuracy and precision based on analysis of biota samples.
[c] Field duplicate precision based on technical judgement and USEPA National Functional Guidelines for duplicate precision

Table 3-3. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Pesticide Compound Analyses

Parameter	Matrix	QC Compounds	Field/ Matrix[a] Duplicate Precision (RPD)	MS/MSD Precision (RPD)	Blanks	LCS & MS/MSD[b] Accuracy (% Recovery)	Surrogate[b] Accuracy (% Recovery)
Pesticides	Surface Water	All analytes	≤30		<RL		
		gamma-BHC		≤26		40-139	
		heptachlor		≤26		37-148	
		aldrin		≤25		38-129	
		dieldrin		≤42		34-150	
		endrin		≤25		41-148	
		4,4'-DDT		≤27		50-147	
		tetrachloro-m-xylene					30-150
		decachlorobiphenyl					30-150
Pesticides	Sediments and Biota	All analytes	≤50		≤RL		
		gamma-BHC		≤37		12-138	
		(lindane)		≤38		17-138	
		heptachlor		≤38		10-144	
		aldrin		≤30		28-137	
		dieldrin		≤32		33-149	
		endrin		≤26		29-134	
		4,4'-DDT					
		tetrachloro-m-xylene					30-150
		decachlorobiphenyl					30-150

Notes:

RL=Project Reporting Limit
[a]Provisions for wider acceptance limits near the RL may be based on professional judgment during data review/validation.
[b]Limits are based on those from STL – Savannah, effective 10-31-00.

Table 3-4. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Polychlorinated Biphenyl Compound Analyses

Parameter	Matrix	QC Compounds[c]	Field/ Matrix[a] Duplicate Precision (RPD)	MS/MSD Precision (RPD)	Blanks	LCS & MS/MSD[b] Accuracy (% Recovery)	Surrogate [b] Accuracy (% Recovery)
PCBs	Surface Water	All analytes	≤30		<RL		
		Monochlorbiphenyls		≤50		30-130	
		Dichlorobiphenyls		≤50		30-130	
		Trichlorobiphenyls		≤50		30-130	
		Tetrachlorobiphenyls		≤50		40-140	
		Pentachlorobiphenyls		≤50		40-140	
		Hexachlorobiphenyls		≤50		40-140	
		Heptachlorobiphenyls		≤50		40-140	
		Octachlorobiphenyls		≤50		40-140	
		Decachlorobiphenyl		≤50		30-130	
		Decachlorobiphenyl-$^{13}C_{10}$					30-130
PCBs	Sediments and Biota	All analytes	≤50		≤RL		
		Monochlorbiphenyls		≤50		30-130	
		Dichlorobiphenyls		≤50		30-130	
		Trichlorobiphenyls		≤50		30-130	
		Tetrachlorobiphenyls		≤50		40-140	
		Pentachlorobiphenyls		≤50		40-140	
		Hexachlorobiphenyls		≤50		40-140	
		Heptachlorobiphenyls		≤50		40-140	
		Octachlorobiphenyls		≤50		40-140	
		Decachlorobiphenyl		≤50		30-130	
		Decachlorobiphenyl-$^{13}C_{10}$					30-130

Notes: RL=Project Reporting Limit

[a]Provisions for wider acceptance limits near the RL may be based on professional judgment during data review/validation.

[b]Limits are based on those from STL-Savannah, effective 01/31/00.

[c]A representative congener from each of the homologue series shown is used as co-spike for the MS/MSD analysis. The laboratory reports spike recovery data for the homologue series based on the recovery of the representative spike within the series.

Table 3-5. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Herbicide Compound Analyses

Parameter	Matrix	QC Compounds	Field/ Matrix[a] Duplicate Precision (Rpd)	MS/MSD Precision (RPD)[b]	Blanks	LCS & MS/MSD[b] Accuracy (% Recovery)	Surrogate[b] Accuracy (% Recovery)
Herbicides	Surface Water	All analytes	≤30		<RL		
		2,4-D		≤78		11-154	
		2,4-DB		≤43		55-167	
		2,4,5-TP (silvex)		≤66		10-100	
		dalapon		≤68		26-97	
		dicamba		≤46		38-152	
		DCAA (2,4-dichloro-phenyl acetic acid)					70-130
Herbicides	Sediments and Biota	All analytes	≤30		≤RL		
		2,4-D		≤47		19-153	
		2,4-DB		≤40		20-160	
		2,4,5-TP (silvex)		≤51		27-120	
		dalapon		≤40		10-170	
		dicamba		≤40		20-160	
		DCAA (2,4-dichloro-phenyl acetic acid)					70-130

Notes:
RL=Project Reporting Limit
[a]Provisions for wider acceptance limits near the RL may be based on professional judgment during data review/validation.
[b]Limits are based on those from STL – Savannah, effective 10/16/00.

Table 3-6. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Dioxin and Dibenzofuran Compound Analyses in Surface Water Samples, Sediments, and Biota

Parameter	QC Compounds	Field/Matrix Duplicate Precision (RPD)[a]	MS/MSD Precision (RPD)	Blanks	Internal[a] Standard Accuracy (% Recovery)	Recovery Standard Accuracy
Dioxin and Dibenzo-furan Analysis	All analytes	≤ 25		< RL		
	2,3,7,8-TCDD		≤ 20			
	1,2,3,7,8-TCDF		≤ 20			
	1,2,3,7,8-PeCDD		≤ 20			
	1,2,3,7,8-PeCDF		≤ 20			
	2,3,4,7,8-PeCDF		≤ 20			
	1,2,3,4,7,8-HxCDD		≤ 20			
	1,2,3,6,7,8-HxCDD		≤ 20			
	1,2,3,7,8,9-HxCDD		≤ 20			
	1,2,3,4,7,8-HxCDF		≤ 20			
	1,2,3,6,7,8-HxCDF		≤ 20			
	1,2,3,7,8,9-HxCDF		≤ 20			
	2,3,4,6,7,8-HxCDF		≤ 20			
	1,2,3,4,6,7,8-HpCDD		≤ 20			
	1,2,3,4,6,7,8-HpCDF		≤ 20			
	1,2,3,4,7,8,9-HpCDF		≤ 20			
	OCDD		≤ 20			
	OCDF		≤ 20			
	$^{13}C_{12}$-2,3,7,8-TCDD				40-135	
	$^{13}C_{12}$-2,3,7,8-TCDF				40-135	
	$^{13}C_{12}$-1,2,3,7,8-PeCDD				40-135	
	$^{13}C_{12}$-1,2,3,7,8-PeCDF				40-135	
	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD				40-135	
	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF				40-135	
	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD				40-135	
	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF				40-135	
	$^{13}C_{12}$-OCDD				40-135	
	$^{13}C_{12}$-1,2,3,4-TCDD					Method LCL and UCL criteria met
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD					

Notes:
General: All methods for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW-846 Method 8290), must be followed.
a Provision for wider acceptance limits near the RL may be based on professional judgement during data review/ validation

Table 3-7. Analytical Laboratory Data Quality Objectives for Precision and Accuracy Inorganic Analyses in Water, Sediment and Fish Tissue Samples

Inorganic Analyses (Metals)	QC Compounds	Field Duplicate Precision[a,c] (RPD)	Sample/MD Precision[a] (RPD)	MS Accuracy (% Recovery)[b]	Blanks	LCS/SRM Accuracy (% Recovery)
Media						
Water, Sediments, Fish Tissue	All analytes	≤ 50	<20% RPD for results >5x RL; difference <± RL for results <5x RL	75-125	< ± RL	Manufacturer's Control Limits

Notes:
General: All method requirements for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Methods 6010B), must be followed
[a] Provision for wider acceptance limits near the RL may be based on professional judgement during data review/validation.
[b] Unless the sample concentration exceeds the spike added concentrations by a factor of 4 or more
[c] Field duplicate precision based on technical judgement and USEPA National Functional Guidelines for duplicate precision

Table 3-8. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for TOC and Grain Size Analyses

Parameter	Matrix	Field Duplicate Precision (RPD)[a]	MS Accuracy (% Recovery) MS/MSD Precision	Method and Field Blanks	LCS Accuracy (% Recovery)
Total Organic Carbon	Sediment	<50%	NR	<RL	Not Applicable
Grain Size	Sediment	<50%	NR	<RL	Not Applicable

Notes:
These are the minimum DQOs defined for these tests. The laboratory is expected to perform all method-required QC.
[a] Professional judgment may be used to accept RPD's outside of these criteria, during data validation, for concentration levels near the RLs.
NR=Not Required to support the DQO level defined for this analysis.

Table 3-9. Summary of QC Sample Types, Criteria, and Corrective Action

TYPE	PURPOSE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
Field Blank (Equipment Rinsate Blanks)	Evaluate cleanliness of sample containers, sample handling, and collection procedures	1 per media per 10 field samples collected	all compounds of interest < RL	Qualify Data
Field Duplicate	Evaluate precision and representativeness taking into account variability of sample matrix	1 per media per 10 field samples collected	plus 50% RPD with provisions for wider acceptance limits near the detection limits	Compare to matrix duplicates, check for possible matrix interferences or improper sample collection procedure, Qualify Data
Laboratory Control Sample (LCS) and Standard Reference Material (SRM)	Evaluate laboratory performance (accuracy) using verified standards from an outside source	1 per media per 20 field samples or per laboratory sample batch, whichever is more frequent	Vendor supplied: Within the 95% confidence interval/ vendor supplied limits	Re-prepare and re-analyze associated samples to obtain acceptable LCS/SRM. Check if MS/MSD acceptable to compare for matrix effects.
Calibration Check Sample	Verifies calibration curve	Minimum of 1 per analytical batch per day	90-110% recovery for inorganics; as specified in EPA methods for organics listed in Table 7-1	Recalibrate: check system
Method Blank	Verifies clean reagents instrument systems, and lab procedures	Minimum of 1 per analytical batch or per 20 field samples: whichever is more frequent	All compounds of interest < RL	Reanalyze; if second blank exceeds criteria, clean and recalibrate system; document corrective action
Matrix spikes and matrix duplicates MS/MSD/ MD	Evaluate precision and accuracy taking into account variability of sample matrix	1 set per media per 20 field samples	Recoveries for MS/MSD specified in Tables 3-1 through 3-7 and in laboratory SOPs. RPD for sample/MS in Tables 3-1 through 3-7.	Qualify data for matrix effects if LCS/SRM is acceptable. Qualify sample/MD if precision is not acceptable. If LCS/RSM is not acceptable, see above
Surrogate Standards	Measures recoveries in actual sample matrices	All GC/MS and all GC samples for organic analyses.	Recoveries as specified in Tables 3-1, 3-3, 3-4, 3-5, 3-7, and in laboratory SOPs	Reanalyze samples: Qualify data.
Internal Standards	Provides standard for calculating analyte response and concentrations	All GC/MS and all GC samples for organic analyses.	Recoveries as specified in the EPA methods in Table 7-1.	Reanalyze samples: Qualify data.

Notes:

RL = Reporting Limit
MD = Matrix duplicate sample
LCS = Laboratory control sample
GC = Gas Chromatography
MSD = Matrix spike duplicate sample
MS = Matrix spike sample
RPD = Relative percent difference (between duplicate results)
GC/MS = Gas Chromatography/Mass Spectrometry
SRM = Standard reference material
LCS = Laboratory control sample

Table 4-1. Summary of Proposed Sampling and Analysis Program in Support of the ERA at Each Of the Five Sampling Sites (Three Plume Discharge Area Sites, UDA and DDA Sites)

Sample Matrix	Field Parameters	Lab Parameters[1]	Investigated Samples	QC Field Duplicates[3]	Trip Blank[2]	QC Matrix Spike/MS Duplicates	Equipment Rinse Blank[2]	Total Number of Samples at Each Site (Excluding blanks)
Surface Water	pH, Temperature DO Conductivity Turbidity	Metals	18(9 filtered, 9 unfiltered)	2 (1 filtered and 1 unfiltered)		2	2 for each site (1 for dissolved metals; 1 for all other parameters)	22
		VOCs	9	1	1 Per Cooler	1		11
		SVOCs	9	1		1		11
		Pesticides	9	1		1		11
		Herbicides	9	1		1		11
		PCBs	9	1		1		11
		Dioxin	PDA sites-5 each; UDA/DDA-9 each	1		1		7(each PDA site) 11(each UDA/DDA site)
		Hardness	9	1		NA		10
		Bioassay (2 Species)	18 (9 for each species)					18
Sediment	None	Metals	9	1		1	1 for each site	11
		VOCs	18 (9 high levels and 9 low levels)	2	2 per cooler (DI for low-level; Methanol for high-level)	2		22
		SVOCs	9	1		1		11
		Pesticides	9	1		1		11
		Herbicides	9	1		1		11
		PCBs	9	1		1		11
		Dioxins	PDA sites-5 each; UDA/DDA-9 each	1		1		7(each PDA site) 11(each UDA/DDA site)
		TOC	9	1	NA	NA	NA	10
		pH	9	1	NA	NA	NA	10
		Grain Size	9	1	NA	NA	NA	10
		Benthic Community Structure	9	NA	NA	NA	NA	9
		Bioaccumulation Tests	3	1	NA		NA	4
		Bioassay (2 Species)	18 (9 for each species)	1	NA		NA	19

Table 4-1. Summary of Proposed Sampling and Analysis Program in Support of the ERA at Each of the Five Sampling Sites (Three Plume Discharge Area Sites, UDA and DDA Sites) *(continued)*

Sample Matrix	Field Parameters	Lab Parameters[1]	Investigated Samples	QC Field Duplicates[3]	Trip Blank[2]	QC Matrix Spike/MS Duplicates	Equipment Rinse Blank[2]	Total Number of Samples at Each Site (Excluding blanks)
Fish Composites (Channel Catfish)	Weight, Length, Stomach contents	PCBs	3	1	NA	1	NA	5
		SVOCs	3	1	NA	1	NA	5
		Pesticides	3	1	NA	1	NA	5
		Herbicides	3	1	NA	1	NA	5
		Metals	3	1	NA	1	NA	5
		Percent Lipid	3	1	NA		NA	4
		Dioxins	1	1	NA	1	NA	3
Fish Composites (Large Shad)	Weight, Length, Stomach Contents	PCBs	3	1	NA	1	NA	5
		SVOCs	3	1	NA	1	NA	5
		Pesticides	3	1	NA	1	NA	5
		Herbicides	3	1	NA	1	NA	5
		Metals	3	1	NA	1	NA	5
		Percent Lipid	3	1	NA		NA	4
		Dioxins	1	1	NA	1	NA	3
Fish Composites (Small Shad)	Weight, Length, Stomach Contents	PCBs	3	1	NA	1	NA	5
		SVOCs	3	1	NA	1	NA	5
		Pesticides	3	1	NA	1	NA	5
		Herbicides	3	1	NA	1	NA	5
		Metals	3	1	NA	1	NA	5
		Percent Lipid	3	1	NA		NA	4
		Dioxins	1	1	NA	1	NA	3
Fish Fillets (Buffalo)	Weight, Length, Stomach contents	PCBs	3	1	NA	1	NA	5
		SVOCs	3	1	NA	1	NA	5
		Pesticides	3	1	NA	1	NA	5
		Metals	3	1	NA	1	NA	5
		Herbicides	3	1	NA	1	NA	5
		Percent Lipid	3	1	NA		NA	4
		Dioxins	1	1	NA	1	NA	3

(1) USEPA Methods for analysis are listed in Section 7 of this QAPP, analysis for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), Pesticides, Herbicides, metals, and PCBs will be performed by STL Laboratory - Savannah, Dioxins to be performed by Triangle Laboratory. Bioassays and Benthic Community Evaluations to be performed by Pennington & Associates,

(2) Field Blanks include trip blanks (TB) for VOC (water low-level TBs are preserved with HCl and high level sediment TBs preserved with methanol) and field equipment rinse blanks (EB) for all parameters.

(3) Field Duplicates and field equipment blanks for all parameters will be performed at a minimum frequency of 1 set per 10 field samples of the same media as required by USEPA Region 5, MS/MSD samples will be collected at a minimum frequency of 1 set per 20 field samples of the same media. Whole composite fish and fish fillets are considered two different media for the purposes of MS/MSD QC frequency. MS/MSD can be performed on different fish samples than indicated in this table; provided the QC frequency is compliant with QAPP criteria, NA = QC not applicable to this parameter based on data quality needs of RCRA investigation.

Table 4-2. Surface Water Samples: Number of Samples, Preservation, Container Specification and Holding Time Requirements in Support of the ERA

Parameter	Number of Samples	Sample Container(s)[1]	Preservative	Holding Time
Metals	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites – for a total of 5 sites; 9 Stations in each of the five sites; 2 samples at each station (1 filtered + 1 unfiltered) for a total of 90 Samples. 2 QC Field duplicate at each site (2 for metals) = 10 QC duplicates; 2 Equipment rinse blanks for each site = 10 Rinse Blanks 2 MS/MSD for each site = 10 MS/MSD samples Total = 90+10+10 +10 = 120 Samples	(1) 250-mL Nalgene bottles for metals for each sample	Cool, 4°C, Protected from Light HNO_3 Preservative for Metals	Extraction: within 7 days of collection Analysis: within 40 Days of extraction
Hardness SVOCs, Herbicides, Pesticides, and PCBs	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites – for a total of 5 sites; 9 Stations in each of the five sites for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates; 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5 +5 = 60 Samples	(2) 1-L glass bottles for SVOCs, Pesticides, Herbicides, and PCBs	Cool, 4°C, Protected from Light	Extraction: within 7 days of collection Analysis: within 40 Days of extraction
Dioxin	3 Sampling sites (Plume Discharge Area) – 5 Stations in each site + 2 upstream / downstream sites - 9 Stations in each site for at total of 33 samples; 1 QC Field duplicate at each site = 5 QC duplicates; 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD sample at each site = 5 MS/MSD Spike samples Total = 33+5+5 +5 = 48 Samples	(2) 1-L Amber Glass Bottles	Cool, 4°C, Protected from light	Extraction: within 30 Days of collection Analysis: within 45 Days of extraction
VOCs	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites - for a total of 5 sites; 9 Stations in each site for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates; 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5+5 = 60 Samples Plus 1 trip blank per cooler	(3) 40-mL glass vials with Teflon-lined septa	Cool, 4°C, Protected from Light, HCl to pH<2	14 days from sample collection

[1]The sample containers list for each sample and one parameter
SVOCs = Semivolatile Organic Compounds; FD = Field Duplicate Sample; VOCs = Volatile Organic Compounds
MS/MSD = Matrix Spike / Matrix Spike Duplicate lab QC; TB = Trip Blank (VOC only); PCBs = Polychlorinated Biphenyls

Table 4-3. Sediment Samples: Number of Samples, Preservation, Container Specification, and Holding Time Requirements in Support of the ERA

Parameter	Number of Samples	Sample Container(s)[1]	Preservative	Holding Time
Metals SVOCs, Herbicides, Pesticides, and PCBs	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites – for a total of 5 sites; 9 Stations in each of the five sites for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5 +5 = 60 Samples	(1) 250-mL glass jar (1) 250-mL Nalgene container for metals	Cool, 4°C, protected from light	Extraction: within 14 days Analysis: within 40 days of extraction
Dioxin	3 Sampling sites (Plume Discharge Area) – 5 Stations in each site + 2 upstream / downstream sites - 9 Stations in each site for at total of 33 samples; 1 QC Field duplicate at each site = 5 QC duplicates; 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD sample at each site = 5 MS/MSD Spike samples Total = 33+5+5 +5 = 48 Samples	(1) 250-mL glass jar	Cool, 4°C, protected from light	Extraction: within 30 days Analysis: within 45 days of extraction
VOCs	low-level VOC: 3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites – for a total of 5 sites; 9 Stations in each site for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC dups. 1 Equipment rinse blanks for each site = 5 Rinse Blanks 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5+5 = 60 Samples Plus 1 trip blank per cooler	(2) 40-mL VOA vials each with 5 mL reagent grade water and a Teflon-coated stir bar, each filled with 5g of sample [water must cover sample] + 125-mL glass bottle (bulk 8260B screening)	Cool, 4°C, protected from light during transit Upon receipt at the laboratory, samples frozen at < -10°C, Protected from light, until just before analysis	14 days from sample collection
	High-Level VOC: 3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites – for a total of 5 sites; 9 Stations in each site for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC dups. 1 Equipment rinse blanks for each site = 5 RB 1 MS/MSD for each site = 5 MS/MSD samples Total = 45+5+5+5 = 60 Samples Plus 1 trip blank per cooler	(1) 40-mL VOA vial with 5 mL methanol filled with 5g of sample [methanol must cover sample]	Cool, 4°C, protected from light	14 days from sample collection
Total Organic Carbon (TOC)	3 Sampling sites (Plume Discharge Areas) + 2 upstream / downstream sites – for a total of 5 sites; 9 Stations in each of the five sites for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates; Total = 45+5+ = 50 Samples	(1) 4-ounce glass jar	Cool, 4°C	28 days
Grain Size	3 Sampling sites (Plume Discharge Area) + 2 upstream / downstream sites – for a total of 5 sites; 9 Stations in each of the five sites for a total of 45 sampling locations = 45 Samples. 1 QC Field duplicate at each site = 5 QC duplicates; Total = 45+5+ = 50 Samples	(1) 500 mL plastic jar	Cool, 4°C	NA

[1]Number of containers for each sampling point and for each parameter
SVOCs = Semivolatile Organic Compounds
FD = Field Duplicate Sample
Duplicate lab QC; EB = Equipment Rinsate Blank
NA = Not Applicable.

VOCs = Volatile Organic Compounds
MS/MSD = Matrix Spike / Matrix Spike
TB = Trip Blank (VOC only)
PCBs = Polychlorinated Biphenyls

Table 4-4. Samples for Analysis of Benthic Community Composition and Abundance: Number of Samples, Sample Preservation, Container Specification, and Holding Time Requirements in Support of the ERA

Parameter	Number of Samples	Sample Container	Preservative	Holding Time
Benthic Invertebrates [Benthic Community Structure]	9 sampling stations at each of the 5 sample sites 1 sample from each station Total = 45 Samples	1 gallon plastic jars	70% Isopropyl alcohol [preservative must cover specimen]	NA

Note: Each benthic fauna sample will be generated from washing and sieving several sediment grabs at each location.

Table 4-5. Samples for Toxicity (Bioassay) and bioaccumulation Tests: Number of Samples, Preservation, Container Specification, and Holding Time Requirements in Support of the ERA

PARAMETER	Matrix	Number of Samples	Sample Container(s)	Preservative	Holding Time
Pimephales promelas (fathead minnow) – 96-hr acute/7-day chronic toxicity, no dilutions, survival and growth	Surface Water	9 sample stations at each of the 5 sample sites; 1 QC Field Duplicate at each site Total = 50 samples for each species	One 2.5-gallon container per sample station (1 container for both tests)	Cool, 4° C, protected from light	14 days
Ceriodaphnia dubia – 48-hr acute/7-day chronic toxicity, no dilutions, survival and reproduction					
Chironomus Tentans (midge larvae) – 7-day Chronic Toxicity, survival and growth	Sediment	9 sample stations at each of the 5 sample sites Total = 45 samples for each species	One wide-mouth 1-gallon container per sample station (1 container for both tests)	Cool, 4° C, protected from light	14 days
Hyalella azteca – 10-day acute, survival and growth					
Cobicula fulminea (Asiatic clam) – 28-day bioaccumulation analysis	Sediment	3 sample stations at each of the 5 sample sites; 1 QC Field Duplicates at each site Total = 20 samples	One wide-mouth 1-gallon container per sample station	Cool, 4° C, protected from light	14 days

Table 4-6. Fish Tissue Analysis Samples: Number, Sample Preservation, Container Specification, and Holding Time Requirements in Support of the ERA

Parameter	Number of Samples[1]	Sample Container	Preservative	Holding Time
SVOCs, Pesticides, PCBs, Metals Herbicides, and Percent Lipids	3 composites of Channel Catfish at each of the five sampling sites (for a total of 15 composites); 3-5 individual Channel Catfish (Fillets) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
Dioxins	1 composite of Channel Catfish at each of the five sampling sites (for a total of 5 composites); 3-5 individual Channel Catfish (Fillets) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
SVOCs, Pesticides, PCBs, Metals Herbicides, and Percent Lipids	3 composites of Large Shad at each of the five sampling sites (for a total of 15 composites); 3-5 individual Large Shad (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
Dioxins	1 composite of Large Shad at each of the five sampling sites (for a total of 5 composites); 3-5 Large Shads (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
SVOCs, Pesticides, PCBs, Metals Herbicides, and Percent Lipids	3 composites of Small Shad at each of the five sampling sites (for a total of 15 composites); 3-5 individual Small Shads (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
Dioxins	1 composite of Small Shad at each of the five sampling sites (for a total of 5 composites); 3-5 individual Small Shads (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
SVOCs, Pesticides, PCBs, Metals Herbicides, and Percent Lipids	3 fillets of Buffalo at each of the five sampling sites (for a total of 15 composites); 3-5 individual Buffalos (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen
Dioxins	1 fillets of Buffalo at each of the five sampling sites (for a total of 5 composites); 3-5 Buffalos (whole body) for each composite	Clean Ziplock Bags	Maintain on dry ice in field, protected from light; lab store frozen < -10°C	1 year frozen

[1]Whole fish will be prepared and analyzed for target SVOCs, pesticides, PCBs, herbicides, and percent lipids, as input to the ecological risk assessment. Fish fillets will be prepared and analyzed for target SVOCs, pesticides, PCBs, herbicides, percent lipids, and dioxin as input to the human health risk assessment.

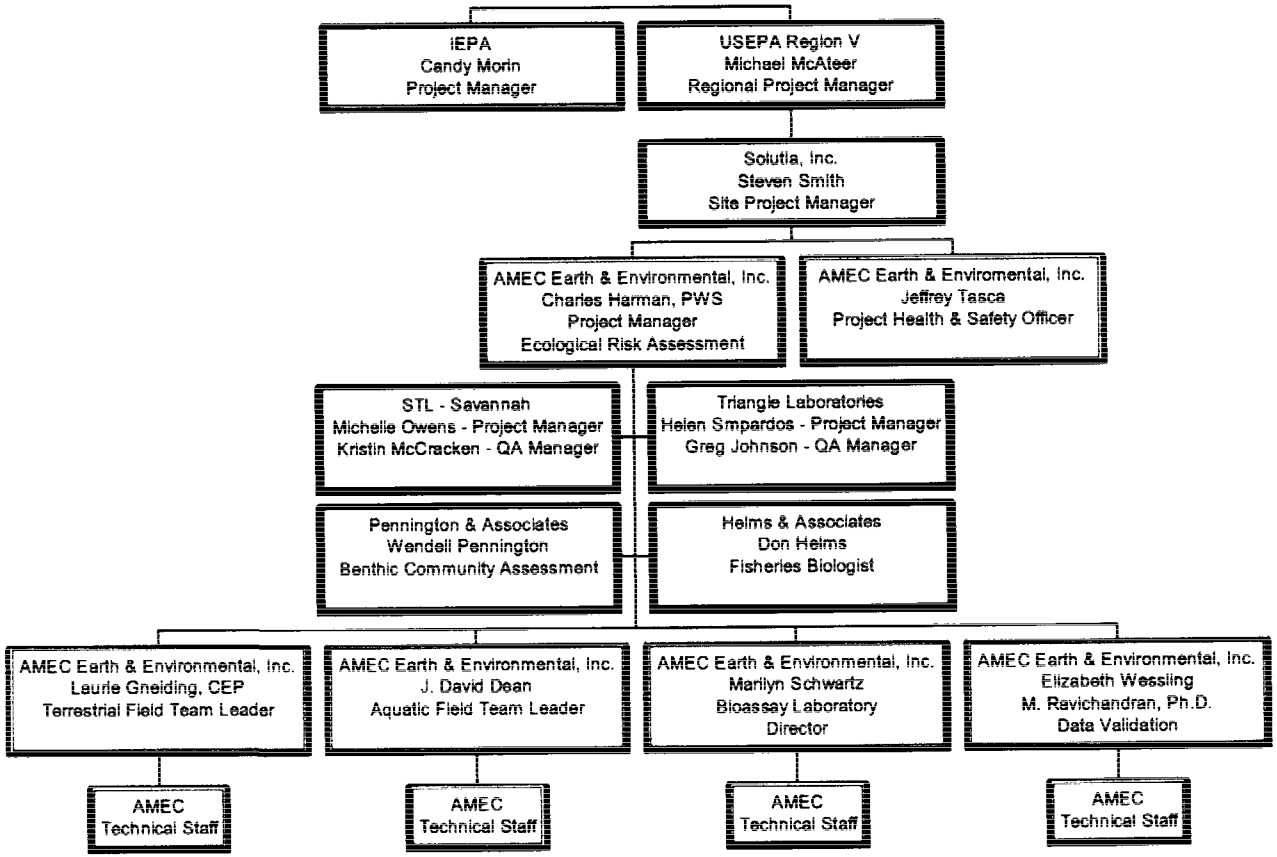
Table 7-1. Laboratory Methods of Analysis in Support of the Ecological and Human Health Risk Assessment

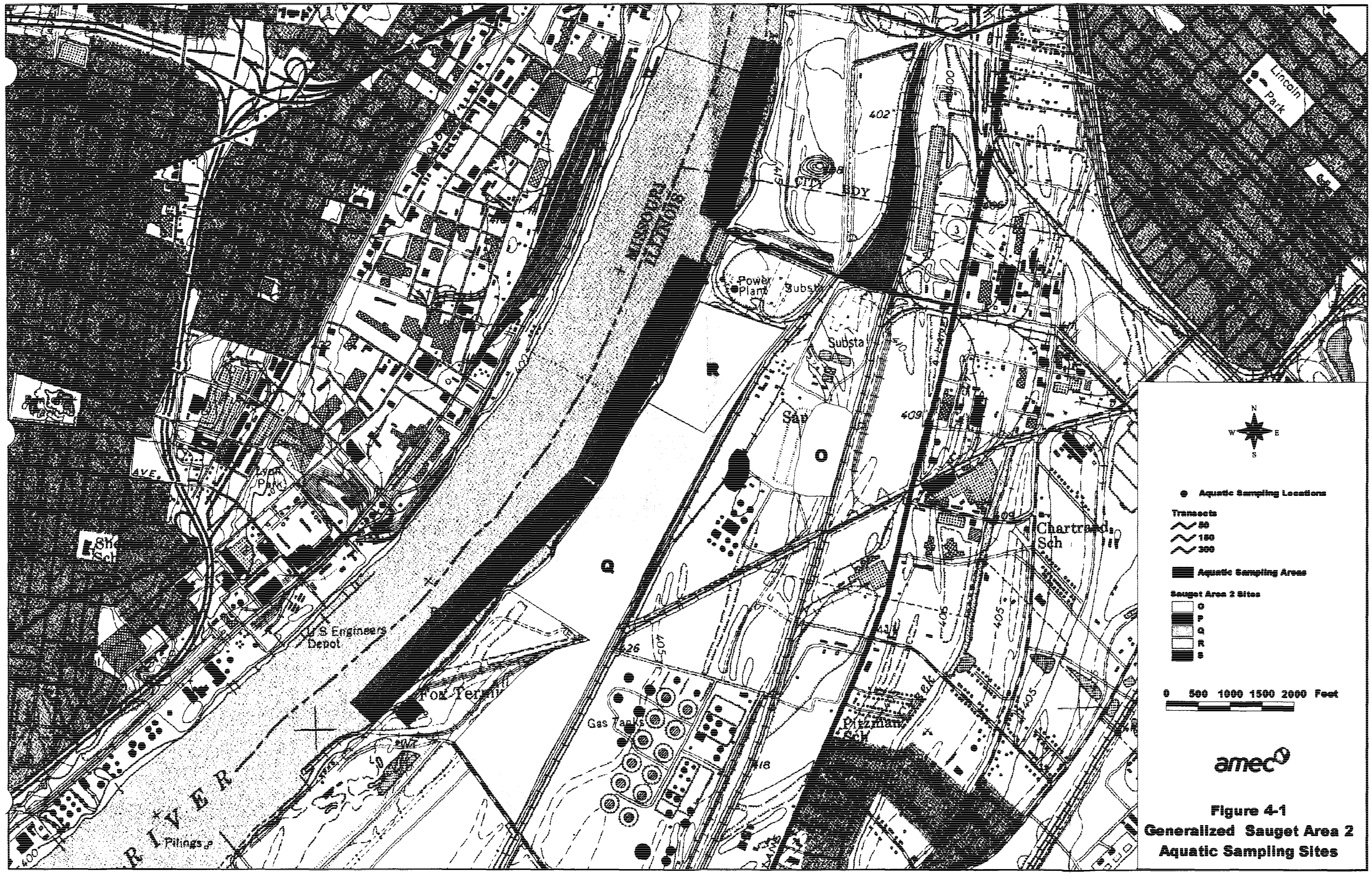
Parameter Test Type	Laboratory SOPs	Method of Analysis Method References
Volatile Organic Compound Analysis	Surface Waters and Sediments STL SOP#s VM20:11.12.99:4	8260B, USEPA SW-846 3rd Edition December 1996
Semivolatile Organic Compound Analysis	Surface Water STL SOP #s: EX30:07.12.99:2 (extraction) SM05:01.07.00:6 (analysis)	3520C, USEPA SW-846, 3rd Edition. December 1996 8270C, USEPA SW-846, 3rd Edition. December 1996
	Sediment STL SOP #s: EX40:02.19.98:1 (extraction) SM05:01.07.00:6 (analysis)	3550B, USEPA SW-846, 3rd Edition. December 1996 8270C, USEPA SW-846. 3rd Edition, December 1996
	Fish Tissue (biota) STL SOP #s: EX41 :01.23.00:0 (extraction) EX61 :07.24.00:0 (cleanup) SM05:01.07.00:6 (analysis)	3550B, USEPA SW-846, 3rd Edition. December 1996 3640A, USEPA SW-846, 3rd Edition, December 1996 8270C, USEPA SW-846. 3rd Edition, December 1996
Pesticides	Surface Water STL SOP #s: EX30:07.12.99:2 (extraction) SG45:01.05.00:6 (analysis)	3520C, USEPA SW-846, 3rd Edition, December 1996 8081A, USEPA SW-846, 3rd Edition, December 1996
	Sediment STL SOP #s: EX40:02.19.98:1 (extraction) SG45:01.05.00:6 (analysis)	3550B USEPA SW-846, 3rd Edition, December 1996 8081A, USEPA SW-846, 3rd Edition. December 1996
	Fish Tissue (biota) STL SOP #s EX41 :01.23.00:0 (extraction) EX61:07.24.00:0 (cleanup) SG45:01.05.00:6 (analysis)	3550B, USEPA SW-846, 3rd Edition, December 1996 3640A, USEPA SW-846, 3rd Edition. December 1996 8081A, USEPA SW-846. 3rd Edition. December 1996
PCBs	Surface Water STL SOP #s: EX30:07.12.99:2 (extraction) SM07:07.12.99:1 (analysis)	3520C, USEPA SW-846, 3rd Edition, December 1996 680, USEPA SW-846, 3rd Edition, December 1996
	Fish Tissue (biota) STL SOP #s: PS15:O8.16.99:3 (extraction) EX61 :07.24.00:0 (cleanup) SM07:O7.12.99:1 (analysis)	3550B, USEPA SW-846. 3rd Edition, December 1996 3640A, USEPA SW-846, 3rd Edition. December 1996 680, USEPA SW-846, 3rd Edition, December 1996
	Sediment STL SOP #s: EX40:02.19.98:1 (extraction) SM07:07.12.99:1 (analysis)	3550B, USEPA SW-846, 3rd Edition, December 1996 680, USEPA SW-846, 3rd Edition, December 1996

Table 7-1. Laboratory Methods of Analysis in Support of the Ecological and Human Health Risk Assessment *(continued)*

Parameter Test Type	Laboratory SOPs	Method of Analysis Method References
Herbicides	Surface Water STL SOP #s: EX45:O6.O3.99:2 (extraction) SG65:O1.12.99:4 (analysis)	8151A, USEPA SW-846. 3rd Edition. December 1996
	Sediment STL SOP #s: EX45:O6.O3.99:2 (extraction) SG65:O1.12.99:4 (analysis)	8151A, USEPA SW-846. 3rd Edition, December 1996
	Fish Tissue (biota) STL SOP#s: EX45:O6.O3.99:2 (extraction) SG65:O1.12.99:4 (analysis)	8151A, USEPA SW-846, 3rd Edition, December 1996
Dioxins	Surface Water Triangle SOP #s: DSP161 (extraction) DHR182 (analysis)	8290, USEPA SW-846, 3rd Edition, December 1996 8290, USEPA SW-846, 3rd Edition. December 1996
	Sediment Triangle SOP #s: DSP1O5 (extraction) DHR182 (analysis)	8290, USEPA SW-846, 3rd Edition, December 1996 8290, USEPA SW-846. 3rd Edition. December 1996
	Fish Tissue (biota) Triangle SOP #s: DSP13O and DSP23O (extraction) DHR182 (analysis)	8290, USEPA SW-846, 3rd Edition, December 1996 8290, USEPA SW-846, 3rd Edition, December 1996
Metals	Surface Water STL SOP#s 3005/3010 (Preparation)	6010B, USEPA SW-846, 3rd Ed., December 1996
	Sediment Sample SOP #s 3050 (Preparation)	6010B, USEPA SW-846, 3rd Ed., December 1996
	Fish Tissue (biota) STL SOP #s 3050 (Preparation)	6010B, USEPA SW-846, 3rd Ed., December 1996
Percent Lipids	Fish Tissue (biota) STL SOP #s:	8290, USEPA SW-846, 3rd Edition. December 1996
Total Organic Carbon (TOC)	Sediment STL SOP #: BA09:O5.O1.98:1	9060 modified for sediments (replicate analyses): USEPA SW-846, 3rd Edition, December 1996
Grain Size	Sediment: Per ASTM Method D-422	ASTM Method D-422
Bioassay Toxicity Testing	Surface Water and Sediment: See Appendix C for complete USEPA method references and SOPs for Acute and Chronic toxicity tests.	Pimephales / Acute EPA/600/4-90/027F; Pimephales / Chronic EPA Method 1000.0; Ceriodaphnia / Acute EPA/600/4-90/027F; Ceriodaphnia / Chronic EPA Method 1002.0; Hyallela / EPA/6OO/R-94/O24; Chironomus / EPA/6OO/R-94/O24; Corbicula / Portland Harbor Sediment Management Plan, June, 1999.

Figure 2.1
Project Team Organization Chart
Sauget Area 2 Sites
Ecological Risk Assessment





amec

Figure 4-1
Generalized Sauget Area 2
Aquatic Sampling Sites

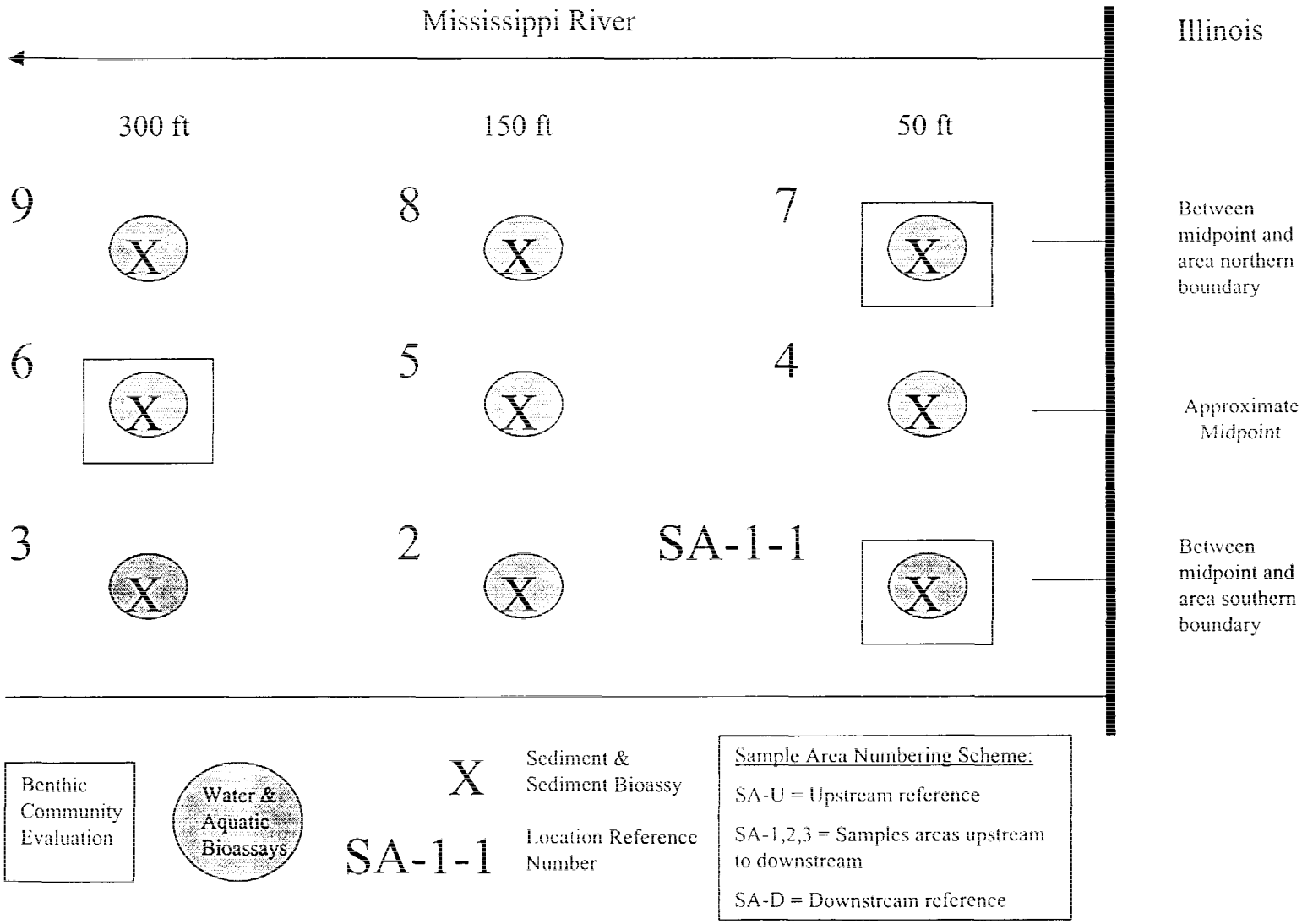


Figure 4-2. Sauget Area 2 Aquatic Sampling Scheme for Plume Discharge Area (PDA) and Reference Sites

Figure 5-1 CHAIN OF CUSTODY RECORD

Project No.	Project Name:	Project Location:
DATE:		Analyses Required
SAMPLERS		

SAMPLE ID	Date	Comp.	Grab	Station Locations	No. of Containers						NOTES/PRESERVATION

Relinquished By: (Signature)	Date	Time	Received By: (Signature)	Date	Time	Remarks:
Relinquished By: (Signature)	Date	Time	Received By: (Signature)	Date	Time	Temp. on receipt: ________ °C
Relinquished By: (Signature)	Date	Time	Received By: (Signature)	Date	Time	
Laboratory:			Phone:			
Contact Person:						

Figure 5-2 Fish Log

AMEC Earth & Environmental, Inc. Fish Collection Log		
Project #	Client:	
Date:		Time:
Species Identification (genus and species):		
Total Length (cm):	Weight (g):	Sex:
Method Collection:	Specimen Identification Number:	
Sample Location (nearest prominent landmark, loran, distance from shore):		
Observations of External Pathology		

Figure 5-3 Sampling Processing Record for Fish Fillet Composites

Project Number: _____ Sampling Date and Time: _____

STUDY PHASE: Screening Study ☐ Intensive Study: ☐ Phase I ☐ Phase II ☐

SITE LOCATION:

Site Name/Number: _____

County/Parish: _____ Lat./Long: _____

Waterbody Name/Segment Number: _____ Waterbody Type: _____

Sample Type (bottom feeder, predator, etc.) _____ Species Name; _____

Composite Sample #: _____ Replicate Number: _____ Number of Individuals: _____

					First Fillet (F1) or Combined Fillets (C)			Second Fillet (F2)		
Fish#	Weight (g)	Scales/Otoliths Removed (✓)	Sex (M,F)	Resection Performed (✓)	Weight (g)	Homogenate Prepared (✓)	Wt. Of Homog. for Composite (g)	Weight (g)	Homogenate Prepared (✓)	Wt. Of Homog. for Composite (g)
001										
002										
003										
004										
005										
006										
007										
008										
009										
010										
Analysis										
Date										

Total Composite Weight(g) (F1 or C) _____ (F2) _____

Notes: _____

Figure 5-4 Example of a Sample Identification Label

Field Samplers Initials	Type of Analyses
Sample ID Number	Sample Location
Sample Date *(d/mo/yr)*	Time (24-h clock)

Appendix A

Field Sampling and Measurement

Standard Operating Procedures

A-1 Equipment Decontamination

A-2 Surface Water Sampling

A-3 Sediment Sampling

A-4 Habitat Assessment Field Data - Sheet Low Gradient Streams

A-5 Fish Collection

A-6 Collection and Treatment of Fish Field Data

A-7 Fish Field and Laboratory Processing

A-8 Horiba U-10 Operation and Calibration Manual

A-1 Equipment Decontamination

EQUIPMENT DECONTAMINATION

1.0 PURPOSE

The standard operating procedure (SOP) describes methods of equipment decontamination for use during site activities at AMEC investigation sites.

2.0 SCOPE

These procedures shall be followed during decontamination of field equipment used to sample environmental media.

This procedure shall serve as management-approved professional guidance for all AMEC personnel and its subcontractors. It is not intended to obviate the need for professional judgment that may arise in unforeseen circumstances. Deviations from this procedure in the planning or execution of activities must be approved by the Project Manager and Technical Director/QA Program Manager.

3.0 DEFINITIONS

None.

4.0 RESPONSIBILITIES

The Field Manager is responsible for ensuring that all field equipment is decontaminated according to this procedure. The Project Manager is responsible for identifying instances of non-compliance with this procedure and ensuring that decontamination activities are in compliance with this procedure.

The Technical Director/QA Program Manager is responsible for ensuring that decontamination activities conducted are in compliance with this procedure.

5.0 PROCEDURES

Decontamination of equipment used in soil/sediment sampling, ground-water monitoring, well drilling and well development, as well as equipment used to sample ground water,

surface water, sediment, waste, wipe, asbestos, and unsaturated zone is necessary to prevent cross-contamination and to maintain the highest integrity possible in collected samples. Planning a decontamination program requires consideration of the following factors:

- The location where the decontamination procedures will be conducted
- The types of equipment requiring decontamination
- The frequency of equipment decontamination
- The cleaning technique and types of cleaning solutions appropriate to the contaminants of concern
- The method for containing the residual contaminants and wash water from the decontamination process
- The use of a quality control measure to determine the effectiveness of the decontamination procedure

This subsection describes standards for decontamination, including the techniques to be used, frequency of decontamination, cleaning solutions, and effectiveness.

5.1 Decontamination Area

An appropriate location for the decontamination area at a site shall be selected on the basis of the ability to control access to the area, the ability to control residual material removed from equipment, the need to store clean equipment, and the ability to restrict access to the area being investigated. The decontamination area shall be located an adequate distance away and upwind from potential contaminant sources to avoid contamination of clean equipment.

5.2 Types of Equipment

Drilling equipment that must be decontaminated includes drill bits, auger sections, drill-string tools, drill rods, split barrel samplers, tremie pipes, clamps, hand tools, and steel

cable. Decontamination of monitoring well development and ground-water sampling equipment includes submersible pumps, bailers, interface probes, water level meters, bladder pumps, air lift pumps, peristaltic pumps, and lysimeters. Other sampling equipment that requires decontamination includes, but is not limited to, hand trowels, hand augers, slide hammer samplers, shovels, stainless steel spoons and bowls, soil sample liners and caps, wipe sampling templates, COLIWASA samplers, and dippers. Equipment with a porous surface, such as rope, cloth hoses, and wooden blocks, cannot be thoroughly decontaminated and shall be properly disposed of after one use.

5.3 Frequency of Equipment Decontamination

Down-hole drilling equipment and equipment used in monitoring well development and purging shall be decontaminated prior to initial use and between each borehole or well. However, down-hole drilling equipment may require more frequent cleaning to prevent cross-contamination between vertical zones within a single borehole. When drilling through a shallow contaminated zone and installing a surface casing to seal off the contaminated zone, the drilling tools shall be decontaminated prior to drilling deeper. Ground-water sampling shall be initiated by sampling ground water from the monitoring well where the least contamination is suspected. All ground-water, surface water, and soil sampling devices shall be decontaminated prior to initial use and between collection of each sample to prevent the possible introduction of contaminants into successive samples.

5.4 Cleaning Solutions and Techniques

Decontamination can be accomplished using a variety of techniques and fluids. The preferred method of decontaminating major equipment such as drill bits, augers, drill string, pump drop-pipe, etc., is steam cleaning. Steam cleaning is accomplished using a portable, high pressure steam cleaner equipped with a pressure hose and fittings. For this method, equipment shall be thoroughly steam washed and rinsed with potable tap water to remove particulates and contaminants.

A rinse decontamination procedure is acceptable for equipment such as bailers, water level meters, new and re-used soil sample liners, and hand tools. The decontamination procedure shall consist of the following: (1) wash with a non-phosphate detergent

(alconox, liquinox, or other suitable detergent) and potable water solution, (2) rinse in a bath with potable water, (3) spray with isopropyl alcohol, (4) rinse in a bath with deionized or distilled water, and (5) spray with deionized or distilled water. If possible, equipment shall be disassembled prior to cleaning. A second wash should be added at the beginning of the process if equipment is very soiled.

Decontaminating submersible pumps requires additional effort because internal surfaces become contaminated during usage. These pumps shall be decontaminated by washing and rinsing the outside surfaces using the procedure described for small equipment or by steam cleaning. The internal surfaces shall be decontaminated by recirculating fluids through the pump while it is operating. This recirculation can be done using a relatively long (typically 4 feet) large diameter pipe (4-inch or greater) equipped with a bottom cap. The pipe shall be filled with the decontamination fluids, the pump placed within the capped pipe, and the pump operated while recirculating the fluids back into the pipe. The decontamination sequence shall include (1) detergent and potable water, (2) potable water rinse, (3) potable water rinse, and (4) deionized water rinse. The decontamination fluids shall be changed after each decontamination cycle.

Solvents other than isopropyl alcohol may be used, depending upon the contaminants involved. For example, if polychlorinated biphenyls (PCBs) or chlorinated pesticides are contaminants of concern, hexane may be used as the decontamination solvent. However, if samples are also to be analyzed for volatile organics, hexane shall not be used. In addition, some decontamination solvents have health effects that must be considered. Decontamination water shall consist of distilled or deionized water. Steam-distilled water shall not be used in the decontamination process as this type of water usually contains elevated concentrations of metals. Decontamination solvents to be used during field activities will be specified in CTO/DO Work Plans or Quality Assurance Project Plans (QAPPs).

Equipment used for measuring field parameters such as pH, temperature, specific conductivity, and turbidity shall be rinsed with deionized or distilled water after each measurement. New, unused soil sample liners and caps will also be washed with a fresh detergent solution and rinsed with potable water followed by distilled or deionized water to remove any dirt or cutting oils that may be on them prior to use.

5.5 CONTAINMENT OF RESIDUAL CONTAMINANTS AND CLEANING SOLUTIONS

A decontamination program for equipment exposed to potentially hazardous materials requires a provision for catchment and disposal of the contaminated material, cleaning solution, and wash water.

When contaminated material and cleaning fluids must be contained from heavy equipment such as drill rigs and support vehicles, the area must be properly floored, preferably with a concrete pad that slopes toward a sump pit. If a concrete pad is impractical, planking can be used to construct solid flooring that is then covered by a nonporous surface and sloped toward a collection sump. If the decontamination area lacks a collection sump, plastic sheeting and blocks or other objects shall be used to create a bermed area for collection of equipment decontamination water. Items such as auger flights, which can be placed on metal stands or other similar equipment, should be situated on this equipment during decontamination to prevent contact with fluids generated by previous equipment decontamination. Clean equipment should be stored in a separate location to prevent recontamination. Decontamination fluids contained within the bermed area shall be collected and stored in secured containers as described below.

Catchment of fluids from the decontamination of lighter-weight drilling equipment and hand-held sampling devices shall be accomplished using wash buckets or tubs. The decontamination fluids shall be collected and stored onsite in secured containers such as DOT-approved drums until their disposition is determined by laboratory analytical results. Containers shall be labeled identifying the site, project number, data, and activity which generated the materials.

5.6 EFFECTIVENESS OF DECONTAMINATION PROCEDURES

A decontamination program must incorporate quality control measures to determine the effectiveness of cleaning methods. Quality control measures typically include collection of equipment rinsate samples or wipe testing. Equipment rinsates consist of analyte-free water that has been poured over or through the sample collection equipment after its final decontamination rinse. Wipe testing is performed by wiping a cloth over the surface of the equipment after cleaning. These quality control measures provide "after-the fact"

information that may be useful in determining whether or not cleaning methods were effective in removing the contaminants of concern.

6.0 RECORDS

The decontamination process shall be described in the field logbook.

7.0 HEALTH AND SAFETY

It is the responsibility of the Onsite Health and Safety Coordinator (OHSC) to set up the site zones (i.e., exclusion, transition, and clean) and decontamination areas. Generally the decontamination area is located within the transition zone, upwind of intrusive activities, and serves as the area where both personnel and equipment are washed to minimize the spread of contamination into the clean zone. For equipment, a series of buckets are set up on a visqueen-lined bermed area. Separate spray bottles containing isopropyl alcohol (or alternative cleaning solvent as described in the Field Sampling Plan) and distilled water are used for final rinsing of equipment. Depending on the nature of the hazards and the site location, decontamination of heavy equipment such as augers, pump drop pipe, and vehicles may be accomplished using a variety of techniques.

Personnel responsible for equipment decontamination must wear the PPE specified in the site- specific Health and Safety Plan (HSP). Generally this includes at a minimum Tyvek® coveralls, steel-toed boots with boot covers or steel-toed rubber boots, safety glasses, ANSI-Standard hard hats, and hearing protection (if heavy equipment is in operation). It should be noted that air monitoring by the OHSC may result in an upgrade to the use of half-face respirators and cartridges in the decontamination area; therefore,

this equipment must be available onsite. If safe alternatives are not achievable, site activities will be discontinued immediately.

In addition to the aforementioned precautions, the following safe work practices will be employed:

Chemical Hazards Associated With Equipment Decontamination

1. Avoid skin contact with and/or incidental ingestion of decontamination solutions and water.

2. Utilize PPE as specified in the site-specific HSP to maximize splash protection.

3. Refer to material safety data sheets (MSDSs), safety personnel, and/or consult sampling personnel regarding appropriate safety measures (i.e., handling, PPE - skin, respiratory, etc.).

4. Take necessary precautions when handling detergents and reagents.

Physical Hazards Associated With Equipment Decontamination

1. To avoid possible back strain, it is recommended that the decontamination area be raised 1 to 2 feet above ground level.

2. To avoid heat stress, over exertion, and exhaustion, it is a recommended that equipment decontamination be rotated among all site personnel.

3. Take necessary precautions when handling field sampling equipment.

8.0 REFERENCES

U.S. EPA Environmental Response Team. 1988. Response Engineering and Analytical Contract Standard Operating Procedures. U.S. EPA, Research Triangle Park, NC.

9.0 ATTACHMENTS

None.

A-2 Surface Water Sampling

SURFACE WATER SAMPLING

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to establish standard protocols for all AMEC field personnel and its subcontractors for use in sampling surface water.

2.0 SCOPE

This procedure shall apply to all surface water sample collection conducted during field investigation activities.

This procedure has been developed to serve as management-approved professional guidance for AMEC personnel and its subcontractors. It is not intended to obviate the need for professional judgment that may arise in unforeseen circumstances. Deviations from this procedure when planning or executing planned activities must be approved by the Project Manager and the Technical Director/QA Program Manager and documented.

3.0 DEFINITIONS

None.

4.0 RESPONSIBILITIES

AMEC field personnel shall follow this SOP when they sample surface water. The Field Manager shall ensure that all field personnel follow this SOP. The Project Manager is responsible for ensuring that all project field personnel follow these procedures when sampling surface water. The Technical Director/QA Program Manager shall evaluate project compliance with these procedures.

5.0 PROCEDURES

Surface water bodies that could be affected by a release from a hazardous waste site may be selected for sampling. This SOP describes sample collection methods for a surface water sampling program.

Proper selection of sampling points and collection methodology are essential to meeting the objectives of a surface water monitoring program. Sampling points should be selected for collection of surface water samples on the basis of characteristics of the body of surface water body to be monitored, the location of the body of surface water, and its hydrologic boundaries with respect to the site. Other considerations include the contaminants of concern, logistical considerations such as access to the surface water body, the direction of flow, and determination of a background location.

Methods of collecting surface water samples vary from hand sampling procedures at a single point to sophisticated, multipoint sampling techniques. The number and type of samples to be collected depends on the characteristics of the body of water, the amount of suspended sediment that a moving body carries, the size of the discharge area at the site, and other factors. Multipoint sampling techniques apply to larger bodies of water; the samples are composited to provide a more representative sample.

A dip or grab sample is appropriate for a small body of water, or for collecting near-surface samples in a larger surface water body. The sampling method involves the filling of a sample container by submerging it either just below the surface, or by lowering the container to a desired depth by using a weighted holder. For shallow bodies of surface water, the sample container shall be held carefully just beneath the water surface to avoid disturbing the streambed and stirring the sediment. The container's mouth should be positioned so that it faces upstream, while the sampling personnel are standing downstream. Any preservative added to the sample should be added after sample collection to avoid loss of preservative. Alternatively, a transfer device may be dipped into the water, then the contents transferred to the appropriate container containing the preservative.

For deeper surface water bodies, either sample containers or transfer devices may be used to collect a sample. A weighted holder that allows either a sample transfer device or a sample container to be lowered, opened for filling, closed, and returned to the surface is suggested for sampling deeper surface water bodies. This is because concentrations of constituents near the surface of a deeper body of surface water may differ from the total concentration distributed throughout the water column cross section and thus a surface sample would not be representative of the water body. An open container that is lowered and raised to the surface at a uniform rate so that the bottle is just filled on reaching the

surface is appropriate for deeper stagnant water bodies, however this method does not collect a truly representative sample in deeper flowing surface water bodies.

Whenever possible, the sampling device, either disposable or constructed of a nonreactive material should hold at least 500 ml to minimize the number of times the liquid must be disturbed, thus reducing agitation of any sediment layers. A 1-liter polypropylene or stainless steel beaker with pour spout and handle works well. Any sampling device may contribute contaminants to a sample. The correct sampling device will not compromise the integrity of the sample and will give the desired analytical results.

Collecting a representative sample from a larger body of surface water is difficult but not impossible. Samples should be collected near the shore unless sampling from a boat is feasible and permitted. If a boat is used, the body of water should be cross sectioned, and samples should be collected at various depths across the water in accordance with the specified sampling plan. For this type of sampling, a weighted-bottle sampler should be used to collect samples at any predetermined depth. The sampler consists of a glass bottle, a weighted sinker, a bottle stopper, and a line that is used to open the bottle and to lower and raise the sampler during sampling. Variations of this sampler are illustrated in ASTM methods D 270 and E 300. It can be either fabricated or purchased. The procedure for use is as follows:

1. Assemble the weighted bottle sampler.
2. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely.
3. Pull out the stopper with a sharp jerk of the sampler line.
4. Allow the bottle to fill completely, as evidenced by the cessation of air bubbles.
5. Raise the sampler and cap the bottle.
6. Wipe the bottle clean. The bottle can also be used as the sample container.

Teflon™ bailers have also been used to collect samples in deep bodies of water. Where cross-sectional sampling is not appropriate, near-shore sampling may be done using a pond sampler.

In this instance, a modification that extends the reach of the sampling technician is most practical. The modification incorporates a telescoping, heavy-duty, aluminum pole with an adjustable beaker clamp attached to the end. A disposable glass or plastic container, or the actual sample container itself, can be fitted into the clamp. If cross contamination is of concern, use a disposable container or the actual sample container.

Another method of extending the reach of sampling efforts is to use a small peristaltic pump. In this method the sample is drawn through heavy-wall Teflon™ tubing and pumped directly into the sample container. This system allows the operator to reach into the liquid body, sample from depth, or sweep the width of narrow streams. However, use of the peristaltic pump is restricted to a maximum depth of 20 to 24 feet due to the physical constraints associated with vacuum pumps.

If medical-grade silicon tubing is used in the peristaltic pump, the system is suitable for sampling almost any analyte, including most organics. Some volatile stripping may occur; however, though the system may have a high flow rate, some material may be lost on the tubing. Therefore, pumping methods should be avoided for sampling volatile organics or oil and grease. Battery-operated pumps of this type are available and can be easily carried by hand or with a shoulder sling. It is necessary in most situations to change both the Teflon™ suction line and the silicon pump tubing between sample locations to avoid cross contamination. This action requires maintaining a sufficiently large stock of material to avoid having to clean the tubing in the field.

When medical-grade silicon tubing is not available or the analytical requirements are particularly strict, the system can be altered. In this configuration, the sample volume accumulates in the vacuum flask and does not enter the pump. The integrity of the collection system can now be maintained with only the most nonreactive material contacting the sample. Some loss in lift ability will result because the pump is now moving air, a compressible gas, rather than an essentially noncompressible liquid. Also, this system cannot be used if volatile compounds are to be analyzed. The potential for losing volatile fractions because of reduced pressure in the vacuum flask renders this method unacceptable for use.

This pump works especially well for sampling large bodies of water when a near-surface sample will not sufficiently characterize the body as a whole. It is capable of lifting water from depths in excess (but not much in excess) of 21 feet. It should be noted that this lift

ability decreases somewhat with higher density fluids and with increased wear on the silicone pump tubing. Similarly, increases in altitude will decrease the pump's ability to lift from depth. When sampling a liquid stream that exhibits a considerable flow rate, it may be necessary to weight the bottom of the suction line.

Samples from various locations and depths can be composited if investigative goals indicate that it is appropriate; otherwise, separate samples will have to be collected. Approximate sampling points should be identified on a sketch of the water body. The following procedures are used for samples collected using transfer devices:

1. Submerge a stainless steel dipper or other suitable device, causing minimal disturbance to the surface of the water. Note the approximate depth and location of the sample source (for example, 1 foot up from bottom or just below the surface).

2. Allow the device to fill slowly and continuously.

3. Retrieve the dipper or device from the surface water with minimal disturbance.

4. Remove the cap from the sample bottle and slightly tilt the mouth of the bottle below the dipper or device edge.

5. Empty the dipper or device slowly, allowing the sample stream to flow gently down the side of the bottle with minimal entry turbulence.

6. Continue delivery of the sample until the bottle is almost completely filled. Check all procedures for recommended headspace for expansion.

7. If necessary, preserve the sample according to guidelines in the sampling plan. In most cases, preservatives should be placed in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.

8. Check that a Teflon™ liner is present in the cap if required. Secure the cap tightly. Tape the cap to the bottle using solvent-free tape; then date and initial the tape. The tape will serve as a custody seal.

9. Label the sample bottle with an appropriate sample tag using a solvent-free marker. Be sure to label the tag carefully and clearly, addressing all the categories or parameters. Record the information in the field logbook and complete the chain-of-custody form.

10. Dismantle the sampler, wipe the parts with terry towels or rags, and store them in plastic bags for subsequent disposal. Follow all instructions for proper decontamination of equipment and personnel.

For samples collected using peristaltic pumps:

1. Install clean, medical-grade silicone tubing in the pump head, per the manufacturer's instructions. Allow sufficient tubing on the discharge side to facilitate convenient dispensation of liquid into sample bottles but only enough on the suction end for attachment to the intake line. This practice will minimize sample contact with the silicone pump tubing. (Some types of thinner Teflon™ tubing may be used.)

2. Select the length of suction intake tubing necessary to reach the required sample depth and attach it to the tubing on the intake side of the pump. If necessary, a small weight composed of relatively inert material, which will not react with anticipated chemicals, may be used to weight the intake tubing. Heavy-wall Teflon™ of a diameter equal to the required pump tubing will suit most applications. (A heavier wall will allow for a slightly greater lateral reach.)

3. If possible, allow several liters of sample to pass through the system before actual sample collection. Collect this purge volume, and then return it to source (i.e., surface water) after the sample aliquot has been collected.

4. Fill necessary sample bottles by allowing pump discharge to flow gently down the side of bottle with minimal entry turbulence. Cap each bottle as filled.

5. Preserve the sample, if necessary, following guidelines in sampling plan. In most cases, preservatives should be placed in sample containers before sample collection to avoid overexposure of samples and overfilling of bottles during collection.

6. Check that a Teflon™ liner is present in the cap, if required. Secure the cap tightly. Tape the cap to the bottle; then date and initial the tape. The tape will serve as a custody seal.

7. Label the sample bottle with an appropriate tag. Be sure to label the tag carefully and clearly, addressing all the categories or parameters. Record the information in the field logbook and complete the chain-of-custody documents.

8. Place the properly labeled sample bottle in an appropriate carrying container.

9. Allow system to drain thoroughly; then disassemble and decontaminate it.

Multipoint sampling techniques that represent both dissolved and suspended constituents and both vertical and horizontal distributions are applicable to larger bodies of water. Subsequent to sample collection, multipoint-sampling techniques may require a compositing and sub-sampling process to homogenize all the individual samples into the number of subsamples required to perform the analyses of interest. Homogenizing samples is discouraged for samples collected for volatile organic analysis, because aeration causes a loss of volatile compounds. If collection of composite samples is required, then the procedure for compositing shall be included in the project-specific Work Plan or FSP.

The sampling devices selected must not compromise sample integrity. Samples must be collected with either disposable devices, or devices constructed of a nonreactive material such as glass, stainless steel, or Teflon™. The device must have adequate capacity to minimize the number of times the liquid must be disturbed, reducing agitation of any sediment layers. Further, the device must be able to transfer the water sample into the sample container without loss of volatile compounds. A single- or double-check valve or stainless steel bailer made of Teflon™ equipped with a bottom discharging device may be utilized.

All equipment used for sample collection must be decontaminated before and after use in accordance with SOP 151, *Equipment Decontamination.*

6.0 RECORDS

During the completion of sampling activities, the sample logbook will be filled out and forms will be transmitted to the Manager for storage in project files.

7.0 HEALTH AND SAFETY

The site-specific Health and Safety Plan shall be adhered to.

8.0 REFERENCES

USEPA. 1988. Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA (EPA USWER Directive 9355 3-01).

USEPA. 1987. A Compendium of Superfund Field Operations Methods.

USEPA. 1992. RCRA Ground-Water Monitoring Technical Enforcement Guidance Document (TEGD).

SOP 151, *Equipment Decontamination*

9.0 ATTACHMENTS

None.

A-3 Sediment Sampling

SEDIMENT SAMPLING

1.0 PURPOSE

This standard operating procedure (SOP) describes the methods by which AMEC personnel will conduct sediment sampling.

2.0 SCOPE

This SOP applies to all AMEC personnel responsible for conducting subaqueous or surface sediment sampling. This SOP shall serve as management-approved professional guidance for field sampling. However, it is not intended to obviate the need for professional judgment that may arise in unforeseen circumstances. Deviations from this procedure in planning or in the execution of planned activities must be approved by the project manager and documented.

3.0 DEFINITIONS

None.

4.0 RESPONSIBILITIES

Project Managers are responsible for selecting the appropriate sampling procedures for use in specific situations. Field Managers and other field personnel are responsible for implementing these procedures in the field.

5.0 PROCEDURE

5.1 METHOD SURVEY

Sediment samples may be recovered using a variety of methods and equipment, depending on the depth of the aqueous layer, the portion and depth of the sediment profile required (surface vs. subsurface), the type of sample required (disturbed vs. undisturbed), and the sediment type.

Sediment is collected from beneath an aqueous layer either directly, using a hand-held device such as a shovel, trowel, or auger, or indirectly using a remotely actuated device such as Ekman or Ponar dredge. Following collection, the sediment is placed into appropriate sample containers.

5.2 Sample Preservation, Containers, Handling, and Storage

5.2.1 Chemical Preservation

Chemical preservation of solids is generally not recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time.

5.2.2 Containers

Wide-mouth glass containers with Teflon™-lined caps are utilized for sediment samples. The sample volume is a function of the analytical requirements and will be specified in the work plan. A sufficient volume should be collected to allow for visual description of a portion of the sample not to be submitted for laboratory analysis.

5.3 Interferences and Potential Problems

Substrate particle size and organic matter content are directly related to water velocity and flow characteristics of a water body. Organic and fine-particle-size contaminants are more likely to be concentrated in sediments typified by fine particle size and a high organic matter content. This type of sediment is most likely to be collected from depositional zones. In contrast, coarse sediments with low organic matter content, do not typically concentrate pollutants and are found in erosional zones. The selection of a sampling location can, therefore, greatly influence the analytical results.

The type of depositional environment, with respect to flow and water velocity, will be a function of the study objectives, and should be specified in the sampling plan.

5.4 Typical Field Sampling Equipment/Apparatus

Sampling Plan
Maps/Plot Plan
Tape measure

Survey stakes, flags, or buoys
Camera and film
Stainless steel, plastic, or other appropriate composition (e.g., Teflon™) bucket
One-quart mason jars w/Teflon™-lined lids
Four-ounce wide-mouth jars with Teflon™-lined lids
Ziploc® plastic bags for samples, and sample jars
Logbook
Labels
Chain-of-Custody Forms
Site Description Forms
Cooler(s)
Ice
Decontamination supplies/equipment
Spade or shovel
Spatula
Scoop
Trowel
Bucket auger
Thin wall auger
Extension rods
T handle
Sampling trier
Sediment coring device:
- tubes
- points
- drive head
- drop hammer
- eggshell check valve devices
- acetate cores

Ponar dredge
Ekman dredge
Nylon rope

5.5 PROCEDURES

5.5.1 Preparation

A. Decontaminate all equipment before sediment sampling (SOP 151, Equipment Decontamination).

B. Perform a general site survey prior to site entry in accordance with the Health and Safety Plan.

C. Evaluate the selection of the sampling device contingent upon the following factors:

1. Depth of water at the sampling location

2. Physical characteristics of the medium to be sampled

5.5.2 Sample Collection

5.5.2.1 Sampling Surface Sediments with Trowels or Scoops from Beneath a Shallow Aqueous Layer

Collection of surface sediment from beneath a shallow aqueous layer can be accomplished with tools such as spades, shovels, and scoops. The surface material can be removed to the required depth; then a stainless steel or plastic scoop should be used to collect the sample.

This method can be used to collect consolidated sediments, but is limited somewhat by the depth of the aqueous layer. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sampling technician. A stainless steel or plastic scoop or lab spoon will suffice in most applications. Care should be exercised to avoid the use of devices plated with chrome or other materials. Plating is particularly common with garden implements such as plotting trowels.

The following procedure will be used to collect the sediment samples:

1. Using a pre-cleaned stainless steel scoop or trowel, remove the desired thickness of sediment from the sampling area.

2. Transfer sample into an appropriate sample container.

5.5.2.2 Sampling Surface Sediments with a Thin-Wall Tube Auger from Beneath a Shallow Aqueous Layer

This system consists of an auger, a series of extensions and a T handle. The auger is driven into the sediment and used to extract a core. A sample of the core is taken from the appropriate depth.

The following procedure will be used for collecting sediment with a thin-walled auger:

1. Insert the auger into the material to be sampled at a 0° to 45° angle from horizontal. This orientation minimizes the spillage of sample from the sampler. Extraction of samples may require tilting of the containers.

2. Rotate the trier once or twice to cut a core of material.

3. Slowly withdraw the trier, making sure that the slot is facing upward.

4. An acetate core may be inserted into the auger prior to sampling if characteristics of the sediments or body of water warrant. By using this technique, an intact core can be extracted.

5. Transfer sample into an appropriate sample or homogenization container.

5.5.2.3 Sampling Deep Sediments with Augers and Thin-Wall Tube Samplers from Beneath a Shallow Aqueous Layer

This systems consists of an auger, a series of extensions and a T handle, and a thin-wall tube sampler. The auger is used to bore a hole to a desired sampling depth and then withdrawn. Next, the auger tip is replaced with a tube core sampler, lowered down the borehole, and driven into the sediment at the completion depth. The core is withdrawn

and the sample collected. This method can be used to collect consolidated sediments, but is somewhat limited by depth of the aqueous layer.

Several augers are available, including bucket and posthole augers. Bucket-type augers are better for direct sample recovery, are easy to use, and provide a large volume of sample. Posthole augers have limited utility for sample collection because they are designed for their ability to cut through fibrous, rooted materials.

The following procedures will be used for collecting sediment samples with the hand auger:

1. Attach the auger bit to a drill rod extension, then attach the T handle to the drill rod.

2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter).

3. During augering, periodically remove any accumulated sediment from the auger bucket.

4. After reaching desired depth, slowly and carefully remove auger from the boring. When sampling directly from the auger, collect the sample after the auger is removed from the boring and proceed to Step 10;

5. Remove the auger tip from the drill rods and replace it with a clean thin-wall tube sampler. Install proper cutting tip.

6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the sediment. Care should be taken to avoid scraping the sides of the borehole. Avoid hammering the drill rods to facilitate coring because the vibrations may cause the boring walls to collapse.

7. Remove the tube sampler and unscrew the drill rods.

8. Remove the cutting tip and remove the core from device.

9. Discard the top of core (approximately 1 inch), because it represents material collected by the tube sampler before penetration of the layer in question.

10. Transfer the sample into an appropriate sample container.

If the borehole will not stay open, sampling techniques detailed in Section 5.5.2.4 may be used.

5.5.2.4 Sampling Surface Sediments from Beneath a Deep Aqueous Layer

This technique consists of lowering a remote sampling mechanism (dredge) to the sediment by a rope, cable, or extended handle. The mechanism is triggered, and the device entraps sediment in spring-loaded jaws, or within lever-operated jaws. The hoisting process can be project specific, but deep samples would typically use an electric motor.

The following procedures will be used for collecting sediments with an Ekman Dredge:

1. Attach a sturdy nylon or stainless steel cable to the hook provided, or secure the extended handle to the bracket with machine bolts.

2. Arrange the Ekman dredge sampler so that the jaws are in the open position and trip cables are positioned over the release studs.

3. Lower the sampler to a point just above the sediment surface.

4. Drop the sampler sharply onto the sediment.

5. Trigger the jaw release mechanism by lowering a messenger down the line, or by depressing the bottom on the upper end of the extended handle.

6. Raise the sampler and slowly decant any free liquid through the top of the sampler by slowly inclining the sampler and collecting the escaping water in a bucket or other suitable container.

7. Open the dredge and transfer the sediment into a stainless steel or plastic bucket. Continue to collect additional sediment until sufficient material has been secured. Transfer the sediment to an appropriate sample container.

8. Collect samples for volatile organic analysis directly from the bucket to minimize volatilization of contaminants.

The following procedures will be used for collecting sediments with a Ponar Dredge or similar type of dredge:

1. Attach a sturdy nylon or steel cable to the hook provided on top of the dredge.
2. Arrange the Ponar dredge sampler in the open position, setting the trip bar so the sampler remains open when lifted from the top.
3. Slowly lower the sampler to a point just above the sediment.
4. Drop the sampler sharply into the sediment while jerking up on the line, thus releasing the trip bar and closing the dredge.
5. Raise the sampler to the surface and slowly decant any free liquid through the screens on top of the dredge.
6. Open the dredge and transfer the sediment to a stainless steel or plastic bucket. Continue to collect additional sediment until sufficient material has been gained. Transfer to an appropriate sample container. and
7. Collect samples for volatile organic analysis directly from the bucket to minimize volatilization of contaminants.

5.5.2.5 Sampling Subsurface Sediments from Beneath a Deep Aqueous Layer

This methodology describes the use of a core sampler to collect subsurface sediments. It consists of a coring device, handle, and acetate core:

1. Assemble the coring device by inserting the acetate core into the sampling tube.
2. Insert the "egg shell" check valve mechanisms into the tip of the sampling tube with the convex surface positioned inside the acetate core.
3. Screw the coring point onto the tip of the sampling tube.
4. Screw the handle onto the upper end of the sampling tube and add extension as needed.

5. This sampler may be used with either a drive hammer for firm, consolidated sediments, or a T handle for soft sediments.

6. Place the sampler in a perpendicular position on the material to be sampled.

7. If the T handle is used, place downward pressure on the device until the desired depth is reached. Rotate the sampler to shear off the core of the bottom and proceed to step 15.

8. If the drive hammer is selected, insert the tapered handle (drive head) of the drive hammer through the drive head.

9. With left hand holding tube, drive the sampler into the material to the desired depth. Do not drive the tube further than the tip of the hammer's guide.

10. Record the length of the tube that penetrated the sample material, and the number of blows required to attain this depth.

11. Remove the drive hammer and fit the keyhole-like opening on the flat side of the hammer onto the drive head. In this position, the hammer serves as a handle for the sampler.

12. Rotate the sampler at least two revolutions to shear off the sample at the bottom.

13. Lower the sampler handle (hammer) until it just clears the two ear-like protrusions on the drive head, and rotate 90°.

14. Withdraw the sampler by pulling the handle (hammer) upwards and dislodging the hammer from the sampler.

15. Unscrew the coring point and remove the eggshell check valve.

16. Slide the acetate core out of the sampler tube. The acetate core may be capped at both ends. The sample may be used in this fashion, or the contents may be transferred to a stainless steel or plastic bucket.

17. Samples for volatile organic analysis will be collected directly from the bucket to minimize volatilization of contaminants.

5.6 POST OPERATION

5.6.1 Field

Decontaminate all equipment, both prior to and following all sampling events.

5.6.2 Office

Finalize field notes and/or transfer logging information into report format.

6.0 RECORDS

Keep records of all sampling activities in the field notebook. Sample custody should be documented on the chain-of-custody forms.

7.0 HEALTH & SAFETY

A site-specific health and safety plan shall be prepared by the manager or their designee.

8.0 REFERENCES

Barth, D.S. and B.J. Mason. 1984. Soil Sampling Quality Assurance User's Guide. EPA-600/4-84-043.

de Vera, E.R., B.P. Simmons, R.D. Stephen, and D.L. Storm. 1980. Samplers and Sampling Procedures for Hazardous Waste Streams. EPA-600/2-80-018.

Emergency Response Team. 1991. Manual of Aquatic Sediment Sampling. Boca Raton: Lewis Publishers.

Mason, B.J. 1983. Preparation of Soil Sampling Protocol: Technique and Strategies. EPA-600/4-83-020.

Mudroch, Alena. 1995. Compendium of ERT Surface Water and Sediment Sampling Procedures: Interim Final. Washington, DC: Emergency Response Team, Emergency Response Division, Office of Emergency and Remedial Response, U.S. Environmental Protection Agency.

USEPA. 1984. Characterization of Hazardous Waste Sites - A Methods Manual: Volume II, Available Sampling Methods, Second Edition.

9.0 ATTACHMENTS

None.

A-4 Habitat Assessment Field Data - Sheet Low Gradient Streams

PHYSICAL CHARACTERIZATION/WATER QUALITY FIELD DATA SHEET (FRONT)

STREAM NAME	LOCATION	
STATION #_____ RIVERMILE_____	STREAM CLASS	
LAT _____ LONG _____	RIVER BASIN	
STORET #	AGENCY	
INVESTIGATORS		
FORM COMPLETED BY	DATE _____ TIME _____ AM PM	REASON FOR SURVEY

WEATHER CONDITIONS	**Now** storm (heavy rain) rain (steady rain) showers (intermittent) ____% %cloud cover clear/sunny	**Past 24 hours** ____%	**Has there been a heavy rain in the last 7 days?** Yes No **Air Temperature**_____ °C Other_____
SITE LOCATION/MAP	**Draw a map of the site and indicate the areas sampled (or attach a photograph)**		
STREAM CHARACTERIZATION	**Stream Subsystem** Perennial Intermittent Tidal **Stream Origin** Glacial Spring-fed Non-glacial montane Mixture of origins Swamp and bog Other_____		**Stream Type** Coldwater Warmwater **Catchment Area**_____km^2

PHYSICAL CHARACTERIZATION/WATER QUALITY FIELD DATA SHEET (BACK)

WATERSHED FEATURES	**Predominant Surrounding Landuse** Forest Commercial Field/Pasture Industrial Agricultural Other _____ Residential **Local Watershed NPS Pollution** No evidence Some potential sources Obvious sources **Local Watershed Erosion** None Moderate Heavy
RIPARIAN VEGETATION *(18 meter buffer)*	**Indicate the dominant type and record the dominant species present** Trees Shrubs Grasses Herbaceous **dominant species present** _____
INSTREAM FEATURES	**Estimated Reach Length** _____m **Estimated Stream Width** _____m **Sampling Reach Area** _____m^2 **Area in km^2 (m^2x1000)** _____km^2 **Estimated Stream Depth** _____m **Surface Velocity** _____m/sec **(at thalweg)** **Canopy Cover** Partly open Partly shaded Shaded **High Water Mark** _____m **Proportion of Reach Represented by Stream Morphology Types** Riffle_____% Run_____% Pool_____% **Channelized** Yes No **Dam Present** Yes No
LARGE WOODY DEBRIS	**LWD** _____m^2 **Density of LWD** _____m^2/km^2 **(LWD/ reach area)**
AQUATIC VEGETATION	**Indicate the dominant type and record the dominant species present** Rooted emergent Rooted submergent Rooted floating Free floating Floating Algae Attached Algae ***dominant species present*** _____ **Portion of the reach with aquatic vegetation** _____%
WATER QUALITY	**Temperature**_____° C **Specific Conductance**_____ **Dissolved Oxygen** _____ **pH** _____ **Turbidity** _____ ***WQ Instrument Used*** _____ **Water Odors** Normal/None Sewage Petroleum Chemical Fishy Other_____ **Water Surface Oils** Slick Sheen Globs Flecks None Other_____ **Turbidity (if not measured)** Clear *Slightly turbid* *Turbid* Opaque Stained Other_____
SEDIMENT/ SUBSTRATE	**Odors** Normal Sewage Petroleum Chemical Anaerobic None Other_____ **Oils** Absent Slight Moderate Profuse **Deposits** Sludge Sawdust Paper fiber Sand Relict shells Other_____ **Looking at stones which are not deeply embedded, are the undersides black in color?** Yes No

INORGANIC SUBSTRATE COMPONENTS (should add up to 100%)			**ORGANIC SUBSTRATE COMPONENTS (does not necessarily add up to 100%)**		
Substrate Type	**Diameter**	**% Composition in Sampling Reach**	**Substrate Type**	**Characteristic**	**% Composition in Sampling Area**
Bedrock			Detritus	sticks, wood, coarse plant materials (CPOM)	
Boulder	> 256 mm (10")				
Cobble	64-256 mm (2.5"-10")		Muck-Mud	black, very fine organic (FPOM)	
Gravel	2-64 mm (0.1"-2.5")				
Sand	0.06-2mm (gritty)		Marl	grey, shell fragments	
Silt	0.004-0.06 mm				
Clay	< 0.004 mm (slick)				

Source: USEPA. 1999. *Form 1.*

HABITAT ASSESSMENT FIELD DATA SHEET LOW-GRADIENT STREAMS (FRONT)

STREAM NAME	LOCATION	
STATION # _____ RIVERMILE _____	STREAM CLASS	
LAT _____ LONG _____	RIVER BASIN	
STORET #	AGENCY	
INVESTIGATORS		
FORM COMPLETED BY	DATE _____ TIME _____ AM PM	REASON FOR SURVEY

	Habitat Parameter	Condition Category			
		Optimal	Suboptimal	Marginal	Poor
Parameters to be evaluated in sampling reach	1. Epifaunal Substrate/ Available Cover	Greater than 50% of substrate favorable for epifaunal colonization and fish cover; mix of snags, submerged logs, undercut banks, cobble or other stable habitat and at stage to allow full colonization potential (i.e., logs/snags that are not new fall and not transient).	30-50% mix of stable habitat; well-suited for full colonization potential; adequate habitat for maintenance of populations; presence of additional substrate in the form of newfall, but not yet prepared for colonization (may rate at high end of scale).	10-30% mix of stable habitat; habitat availability less than desirable; substrate frequently disturbed or removed.	Less than 10% stable habitat; lack of habitat is obvious; substrate unstable or lacking.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
	2. Pool Substrate Characterization	Mixture of substrate materials, with gravel and firm sand prevalent; root mats and submerged vegetation common.	Mixture of soft sand, mud, or clay; mud may be dominant; some root mats and submerged vegetation present.	All mud or clay or sand bottom; little or no root mat; no submerged vegetation.	Hard-pan clay or bedrock; no root mat or vegetation.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
	3. Pool Variability	Even mix of large-shallow, large-deep, small-shallow, small-deep pools present.	Majority of pools large-deep; very few shallow.	Shallow pools much more prevalent than deep pools.	Majority of pools small-shallow or pools absent.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
	4. Sediment Deposition	Little or no enlargement of islands or point bars and less than <20% of the bottom affected by sediment deposition.	Some new increase in bar formation, mostly from gravel, sand or fine sediment; 20-50% of the bottom affected; slight deposition in pools.	Moderate deposition of new gravel, sand or fine sediment on old and new bars; 50-80% of the bottom affected; sediment deposits at obstructions, constrictions, and bends; moderate deposition of pools prevalent.	Heavy deposits of fine material, increased bar development; more than 80% of the bottom changing frequently; pools almost absent due to substantial sediment deposition.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
	5. Channel Flow Status	Water reaches base of both lower banks, and minimal amount of channel substrate is exposed.	Water fills >75% of the available channel; or <25% of channel substrate is exposed.	Water fills 25-75% of the available channel, and/or riffle substrates are mostly exposed.	Very little water in channel and mostly present as standing pools.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0

HABITAT ASSESSMENT FIELD DATA SHEET—LOW-GRADIENT STREAMS (BACK)

Parameters to be evaluated broader than sampling reach

Habitat Parameter	Condition Category			
	Optimal	Suboptimal	Marginal	Poor
6. Channel Alteration	Channelization or dredging absent or minimal; stream with normal pattern.	Some channelization present, usually in areas of bridge abutments; evidence of past channelization, i.e., dredging, (greater than past 20 yr) may be present, but recent channelization is not present.	Channelization may be extensive; embankments or shoring structures present on both banks; and 40 to 80% of stream reach channelized and disrupted.	Banks shored with gabion or cement; over 80% of the stream reach channelized and disrupted. Instream habitat greatly altered or removed entirely.
SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
7. Channel Sinuosity	The bends in the stream increase the stream length 3 to 4 times longer than if it was in a straight line. (Note - channel braiding is considered normal in coastal plains and other low-lying areas. This parameter is not easily rated in these areas.)	The bends in the stream increase the stream length 1 to 2 times longer than if it was in a straight line.	The bends in the stream increase the stream length 1 to 2 times longer than if it was in a straight line.	Channel straight; waterway has been channelized for a long distance.
SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
8. Bank Stability (score each bank)	Banks stable; evidence of erosion or bank failure absent or minimal; little potential for future problems. <5% of bank affected.	Moderately stable; infrequent, small areas of erosion mostly healed over. 5-30% of bank in reach has areas of erosion.	Moderately unstable; 30-60% of bank in reach has areas of erosion; high erosion potential during floods.	Unstable; many eroded areas; "raw" areas frequent along straight sections and bends; obvious bank sloughing; 60-100% of bank has erosional scars.
SCORE ___ (LB)	Left Bank 10 9	8 7 6	5 4 3	2 1 0
SCORE ___ (RB)	Right Bank 10 9	8 7 6	5 4 3	2 1 0
9. Vegetative Protection (score each bank) Note: determine left or right side by facing downstream.	More than 90% of the streambank surfaces and immediate riparian zone covered by native vegetation, including trees, understory shrubs, or nonwoody macrophytes; vegetative disruption through grazing or mowing minimal or not evident; almost all plants allowed to grow naturally.	70-90% of the streambank surfaces covered by native vegetation, but one class of plants is not well-represented; disruption evident but not affecting full plant growth potential to any great extent; more than one-half of the potential plant stubble height remaining.	50-70% of the streambank surfaces covered by vegetation; disruption obvious; patches of bare soil or closely cropped vegetation common; less than one-half of the potential plant stubble height remaining.	Less than 50% of the streambank surfaces covered by vegetation; disruption of streambank vegetation is very high; vegetation has been removed to 5 centimeters or less in average stubble height.
SCORE ___ (LB)	Left Bank 10 9	8 7 6	5 4 3	2 1 0
SCORE ___ (RB)	Right Bank 10 9	8 7 6	5 4 3	2 1 0
10. Riparian Vegetative Zone Width (score each bank riparian zone)	Width of riparian zone >18 meters; human activities (i.e., parking lots, roadbeds, clear-cuts, lawns, or crops) have not impacted zone.	Width of riparian zone 12-18 meters; human activities have impacted zone only minimally.	Width of riparian zone 6-12 meters; human activities have impacted zone a great deal.	Width of riparian zone <6 meters: little or no riparian vegetation due to human activities.
SCORE ___ (LB)	Left Bank 10 9	8 7 6	5 4 3	2 1 0
SCORE ___ (RB)	Right Bank 10 9	8 7 6	5 4 3	2 1 0

Total Score _____

Source: USEPA. 1999. Appendix A1-Form 3

A-5 Fish Collection

STANDARD OPERATING PROCEDURE
FISH COLLECTION

1.0 PURPOSE

This method is used to qualitatively assess the fish community and available habitat as well as to collect fish samples in a semi-quantitative manner. Note that the procedures described in this SOP may be modified following the observations made during Reconnaissance Survey activities, to optimize the fish collection protocols for this program.

2.0 EQUIPMENT DESCRIPTIONS

The equipment used to sample fish includes: eloctroshocking equipment, scientific gill nets, hoop nets, other gear utilized by local fishermen, ruler, scale, forceps, aluminum foil and/or zip-loc bags, and pens and labels.

3.0 PROCEDURE

Each type of equipment may be deployed from a boat. Different types of electroshocking equipment are used whether used from a boat or while wading. Electroshocking should be performed according to the instructions of the equipment manufacturer. Gill nets are deployed usually at dawn or dusk and left in the water for 1 to 2 hours.

Each type of equipment should be employed for a standard length of the water body or for a standard amount of time. These lengths of time are decided in the field based on field conditions and will be optimized following the Reconnaissance Survey activities.

Field sampling personnel will record observations such as fish species identification, age, length, weight, and presence of gross histopathological anomalies. Habitat assessment will be performed by the Ecological Project Manager and Field Team Leader in cooperation with the commercial fisherman. This assessment will be qualitative and based on a combination of experience in similar water bodies, a qualitative assessment of the physical and water quality conditions provided by the habitat, and qualitative assessment of recreational fishing potential. Information pertinent to fish populations will be recorded on the Fish Sampling Field Data Sheet (Attachment 1) or a field log book.

The species of each fish caught is determined in the field, and each fish is measured (body length including and excluding fins) and weighed. Other observations may be made as necessary (removal of a scale or spine for aging, determination of sex, and/or observations of overall health). These observations will also be recorded on the Fish Sampling Field Data Sheet (Attachment 1) or a field log book.

Live fish may be kept in aerated water from the water body being studied and later released if not required for chemical analysis.

4.0 DOCUMENTATION

Sample locations, time and date of collection, and initials of the collector will be on each sample label. This information will also be documented in a field note book or log sheet in addition to species, length, weight, and any other observations. Documentation will be consistent with the QAPP.

5.0 REFERENCES

USEPA. 1999. *Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates and Fish, Second Edition.* EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C. **Authors:** Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling.

USEPA. 1998. *Lake and Reservoir Bioassessment and Biocriteria: Technical Guidance Document.* EPA Office of Water, EPA 841-B-98-007

ATTACHMENT 1
FISH SAMPLING FIELD DATA SHEET (FRONT)

STREAM NAME	LOCATION
STATION #________ RIVERMILE________	STREAM CLASS
LAT ____________ LONG ____________	RIVER BASIN
STORET #	AGENCY
GEAR	INVESTIGATORS
FORM COMPLETED BY	DATE ______ TIME ______ AM PM REASON FOR SURVEY

SAMPLE COLLECTION	**How were the fish captured?** back pack tote barge other ____________ **Block nets used?** YES NO **Sampling Duration** Start time ________ End time ________ Duration ________ **Stream width (in meters)** Max________ Mean________
HABITAT TYPES	**Indicate the percentage of each habitat type present** Riffles_____% Pools_____% Runs_____% Snags_____% Submerged Macrophytes_____% Other ( )_____%
GENERAL COMMENTS	

SPECIES	**TOTAL (COUNT)**	**OPTIONAL: LENGTH (mm)/WEIGHT (g) (25 SPECIMEN MAX SUBSAMPLE)**					**ANOMALIES**							
							D	**E**	**F**	**L**	**M**	**S**	**T**	**Z**

ANOMALY CODES: D = deformities; E = eroded fins; F = fungus; L = lesions; M = multiple DELT anomalies; S = emaciated; Z = other

FISH SAMPLING FIELD DATA SHEET (BACK)

SPECIES	TOTAL (COUNT)	OPTIONAL: LENGTH (mm)/WEIGHT (g) (25 SPECIMEN MAX SUBSAMPLE)					ANOMALIES*							
							D	E	F	L	M	S	T	Z

* ANOMALY CODES: D = deformities; E = eroded fins; F = fungus; L = lesions; M = multiple DELT anomalies; S = emaciated; Z = other

Source: USEPA. 1999. Appendix A4-Form 1

A-6 Collection and Treatment of Fish Field Data

STANDARD OPERATING PROCEDURES
COLLECTION AND TREATMENT OF FISH FIELD DATA

1.0 Purpose and Applicability

This procedure describes the efficient collecting and recording of fish field data.

2.0 Definitions

Total length - total length is the maximum length from the tip of the anterior-most portion of the fish with the jaws closed to the posterior-most portion of the caudal fin with the labels appressed.

Fork length - fork length is the distance from the anterior-most portion of the fish with the jaws closed to the deepest incision of the fork of the tail.

3.0 Health and Safety Considerations

Health and Safety considerations are dependent on site logistics and the possible presence of hazardous chemicals or wildlife. All field personnel must wear proper clothing for the environmental conditions present. At least one member of the field team must be trained for basic first aid techniques such as heat stress prevention and CPR. A first aid kit must be provided as standard equipment for all field trips.

4.0 Quality Assurance Planning Considerations

The appropriate equipment and methods will be selected for each project on the basis of circumstances, objectives, and requirements of that project. The provisions of this SOP will be adapted to these project specific requirements in the QAPP.

5.0 Responsibilities

One or more members of the field team are assigned responsibility for collecting and recording field data. Responsibilities include assuring that proper gear and supplies are in working order and ready for transport to the field and performing the tasks indicated in this SOP.

6.0 Training/Qualifications

Each employee who collects and handles fish data will have previous experience with the procedures or be trained by an experienced ecologist in the specific procedure used. Training and experience includes cognizance of the literature covering methods, limitations for each species, and knowledge of results, which can be expected.

7.0 Materials

Weighing scales
Scale envelopes
Scaler
Forceps Scalpel
Scissors
Measuring board
Zip-Loc Bags Labels
Taxonomic fish keys
Pencils
Data sheets
First aid kit

8.0 Methods

8.1 Recording Data

All particulars of field sampling are recorded on data forms (see Fish Collection SOP) or in the field log book. All data sheets for each sampling effort (e.g., each gill net, each shocking run, each seine haul, etc.) are securely fastened together for transport to the laboratory.

If a mixed collection of small fish or fish of doubtful identification are taken for laboratory analysis, an additional data sheet is made up with a description of the sample in the comments section. This sheet is included with the fish data sheets. Another label with all pertinent data and the sample identification is inserted into the sample container.

When project requirements do not necessitate individual length-weight measurements, fish are tallied by species in length groups.

8.2 Weight and Length Measurements

If required, individuals of each fish species are weighed and measured and the data are recorded on data sheets (see *Fish Collection SOP*). If more than one sheet is used, the sheets are identified as sheet 1 of x, sheet 2 of x, etc. Measuring board and scales used in the field are calibrated according to manufacturer's instructions and other appropriate methods. If fish have been covered with sand or other foreign materials in the process of capture, this foreign material is removed before weighing the fish. The units of measurement used for weight and length will be carefully recorded.

8.3 Unusual Specimens

Anomalies (e.g., tail or tail-fin deformed, parasitized, part of a fish protruding from the mouth of a slightly larger fish, emaciated condition, etc.), are noted on the data sheet.

8.4 Uncertain Field Identifications

Any specimen that cannot be clearly identified to project specified taxonomic level is returned to the laboratory for identification or confirmation.

8.5 Numerous Small Fishes

When small fish are captured and project requirements permit, these individuals are counted and released as rapidly as possible.

If lengths and weights are required, at least 25 individuals of each species representing the complete range of lengths are preserved for analyses in the laboratory.

The remainder of each species are counted and released. Both the number counted and the notation of specimens collected are recorded on the same sheet.

8.6 Threatened and Endangered Species

Special effort is made to return threatened or endangered species to the water in an uninjured condition after weighing and measuring. Unless voucher specimens are required and special permission is obtained, threatened and endangered species are not retained.

8.7 Field Preservation

All fish to be sent to the analytical laboratory for chemical analyses are preserved frozen on dry ice. Details concerning field preservation, labeling and shipment of fish samples are included in the QAPP.

8.8 Collection of Scale Samples

he currently accepted method for collecting scale samples utilized two different locations of collection, depending on whether the fish is soft-rayed or spiny-rayed. On soft-rayed fish, the scale sample is collected above the lateral line directly below the origin of the dorsal fin. On spiny-rayed fish, the scale sample is collected below the lateral line at the end of the appressed pectoral fin. In each case, 10 to 20 scales should be collected unless very large fish are encountered.

If scales are obviously regenerated (as evidenced by a pebbled center area under 10% magnification), additional scales should be collected from an adjacent area. Scales from very large fish are examined in the field under 10% magnification to eliminate regenerated scale; 3 or 4 scales are collected.

Scale samples are not collected from ganoid-scated fishes (gars) or fishes not having a regular scale pattern (e.g., sturgeons or leather and mirror carp), since these scales generally do not provide useful information.

Scales are collected by pulling them from their pockets with a front to back movement of a knife, scalpel, or similar instrument.

Insert the collected scales into a prepared scale envelope upon which the following are recorded on a label: species, location, collection number, length, weight, and fish number from the data sheet.

If scales are taken from all fish or none of the fish recorded on a data sheet, this fact should be recorded in the comment section. If scales are taken from some fish listed on the data sheet but not others, an asterisk or the letter "S" is placed in the box on the data sheet containing the fish number.

The envelopes are always checked for correct data prior to releasing the fish. As soon as possible after collection (generally the same day), scale envelopes are spread to dry.

9.0 Quality Control Checks

Quality control checks will ensure that: the appropriate taxonomic key is being used; the correct fish sampling equipment is used; data sheets and labels are correctly filled out; field equipment is working properly; preservative materials (alcohol, formalin, etc.) are fresh and not deteriorated; lengths and weights of fish are taken; and scale samples are properly taken.

10.0 Documentation

Documentation of activities will be completed by an ecologist or biologist conducting the activities. The documentation will be kept on field data forms and/or a field log book. At a minimum, the documentation will include the date, field crew names, methods used, and sample identification numbers.

The documentation will be peer reviewed, authorized (signed-off) by the Ecological Project Manager/Field Team Leader, and dated.

All documentation will be retained in the project files following completion of the project, consistent with the QAPP requirements.

A-7 Fish Field and Laboratory Processing

STANDARD OPERATING PROCEDURE
FISH FIELD AND LABORATORY PROCESSING

1.0 FISH PROCESSING IN THE FIELD

Fish will be collected by location and retained in live wells containing location- specific water until sample processing is initiated. Fish containers (e.g., live wells) will be labeled with capture location information and aerated to minimize fish mortality before fish processing. All fish retained for potential sample analysis will be enumerated and separated by species and size class. This information will subsequently be used to determine the number of samples and associated IDs.

Fish will be sacrificed by cervical separation or sharp blow to the head with a stunning rod. All fish not retained for analysis will be released unharmed after processing to their respective locations.

The following metrics will be recorded for each individual fish included in any sample.

- Total Length (cm) – The greatest of a fish from its anterior most extremity to the end of the tail fin. For fish with a forked tail, the two lobes should be pressed together, and length of the longest lobe should be recorded.
- Total Weight (g) – Fish will be placed in a pre-weighted decontaminated tray and weighed to the nearest gram.
- Sex (MIF) – When possible (i.e., bass), fish sex will be identified by external morphological characteristics or internal reproductive examination.
- Physical Exam – Gross pathological examination of all fish will be conducted and documented. Special consideration will be given to gross pathological conditions on largemouth bass.

Upon completion of collection of metrics, fish samples will be packaged for shipment to the analytical laboratory. Fish will be placed in zip-lock bags. The sample ID labels will be placed on the outside of the aluminum foil and secured with clear tape. If more than one fish is used for a sample (composite), all fish used for the sample will be placed on one piece of aluminum foil, wrapped and labeled with the appropriate sample ID. To preserve sample integrity, samples will be placed in double zip-lock freezer bags with a second ID label and placed in a cooler with dry ice for shipment.

2.0 WHOLE BODY SAMPLE PROCESSING IN THE LABORATORY

The fish will be unwrapped and inspected carefully to ensure that they have not been compromised in any way (i.e., properly preserved during shipment.) Any specimen deemed unsuitable for further processing and analysis will be discarded and identified on the sample processing record.

Fish samples for whole body analysis will be rinsed of all debris with deionized water and a wet-weight will be determined for each fish. All samples will be weighted on balances that are properly calibrated and of adequate accuracy and precision to meet program data quality objectives.

3.0 FILLET SAMPLE PROCESSING IN THE LABORATORY

Fish samples for filleting will be placed in a pre-weighed decontaminated tray and weighed to the nearest gram. The fish fillet will also be weighed and recorded.

While filleting care must be taken to avoid contaminating fillet tissues with material released from inadvertent puncture of internal organs. NOTE: If the fillet tissue is contaminated by materials released from the inadvertent puncture of the internal organs during resection, the fillet tissue must be rinsed in contarninant-free, deionized distilled water and blotted dry and a notation should be made in the sample processing record.

Filleting will be conducted by or under the supervision of experienced staff from AMEC. An initial cut should be made from the dorsal fin to the pelvic fin, just behind the opercular flap. Run the tip of the knife along the dorsal side of the fish, from the initial cut to the caudal fin. Continue making successively deeper cuts, running the knife blade as close to

the neural spines and ribs as possible. After the fillet is obtained, remove the skin. Place the skin side of the fillet down on the dissecting tray, hold on to the WI portion of the fillet, and run the knife between the skin and the muscle tissue. Remove any debris from the skinless fillet by rinsing with deionized water.
After a fillet is cleaned, place the sample in a pre-weighted decontaminated tray and record the weight to the nearest gram. For composite samples, obtain all the fillets for the composite and weigh to the nearest gram. Fillet samples will be placed in decontaminated aluminum foil (dull side toward the fish). The sample ID label will be placed on the outside of the aluminum foil and secured with clear tape. Place the samples in double zip-lock freezer bags with a second ID label and store frozen (-10°C) until analysis.

4.0 SAMPLE SIZE

Individual and composite fish samples will be collected for the aforementioned sample sites. Both sides of the fish will be filleted to obtain the minimum sample weight of 150 grams (allows for re-analysis and matrix QC). All fillet samples will have the skin removed. If composite samples of fish are made, each, composite will contain fish within 75% of the total length between the largest and smallest fish of each composite.

5.0 DOCUMENTATION

All sample documentation will follow project specific SOPs for field sample ID, data sheet, chain-of-custody, and custody seal procedures, as described in the QAPP.

6.0 DECONTAMINATION

Prior to filleting, hands will be washed with soap and rinsed thoroughly in tap water, followed by distilled water (U.S. EPA, 1991. Gloves must be wrm, and will be talc- or dust-free and of non- contaminating materials. Specimens will come into contact with non-contaminating surfaces only. Fish will be filleted on glass or PTFE cutting boards that have been cleaned properly according to EPA Laboratory standards, (EPA 1995) and will be cleaned properly between fish samples thoroughly with a detergent solution, rinsed with tap water, and then rinsed with analyte-free water.

All dissection equipment will be decontaminated following the project-specific SOP for equipment decontamination including detergent/water wash, potable water rinse, hexane rinse, isopropyl alcohol rinse, and deionized water rinse. All aluminum foil will be hexane rinsed prior to use.

7.0 SAMPLE SHIPPING

Samples will be sent by overnight delivery service (next morning delivery) to the analytical laboratory. Shippers will notify the receiving laboratory that samples are being sent for next-day delivery. Shippers should also call the receiving laboratory the day of delivery to verify the receipt of samples. If samples are collected on a Friday, the field sampler/shipper should contact the analytical laboratory to make sure they have staff to receive the samples on Saturday. If not, the field sampler will maintain the fish samples on dry ice or frozen (-1 °C) for subsequent shipment on Monday.

8.0 REFERENCES

U.S. EPA. (U.S. Environmental Protection Agency). Sept. 1995. *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Vol 1.* Fish Sampling and Analysis Second Edition. U.S. EPA, Office of Water.

U.S. EPA, (U.S. Environmental Protection Agency). 1991. *Environmental Monitoring and Assessment Program (EMAP) Near-Coastal Program Laboratory Methods for Filleting and Compositing fish for Organic and Inorganic Contaminant Analyses.* Draft. U.S. EPA, Office of Research and Development, Environmental Research Laboratory Narragansett, RI.

A-8 Horiba U-10 Operation and Calibration Manual

HORIBA

INSTRUCTION MANUAL

WATER QUALITY CHECKER U-10

CODE:I0040801000F5

HORIBA Ltd.

Miyanohigashi, Kisshoin, Minami-ku,
Kyoto, Japan
Phone: (81) 75-313-8123

WARNING

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

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

⚠CAUTION

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

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

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

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

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

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

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

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

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

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Note that the contents of this Instruction Manual are subject to change without prior notice as design changes are made on the instrument.

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Section 1

Getting Started

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

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The keypad 6
Setting up the U-10 8
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and the reference sensor 8
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Inserting the battery 9
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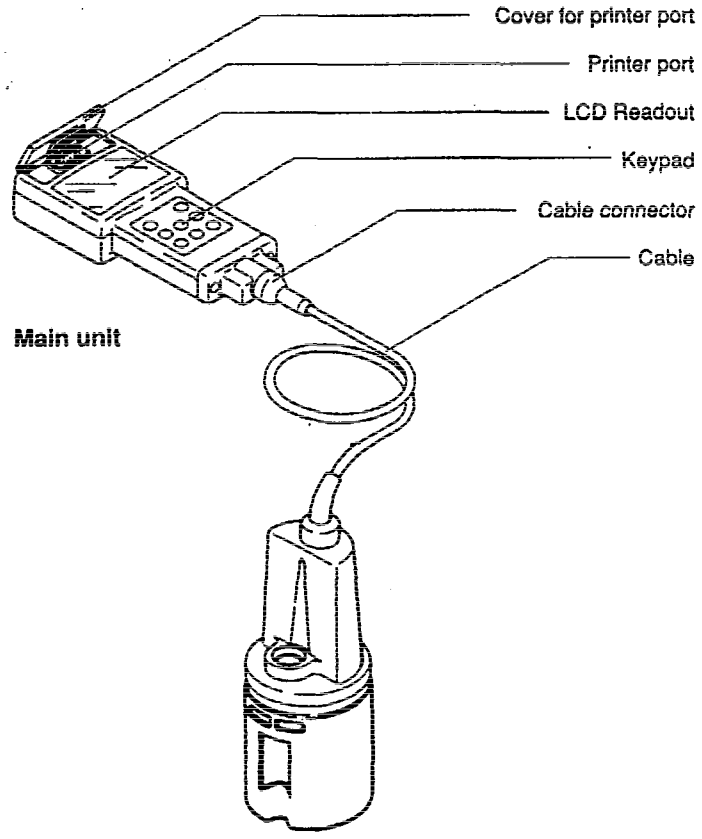
Configuration of the U-10

Main unit



Main unit

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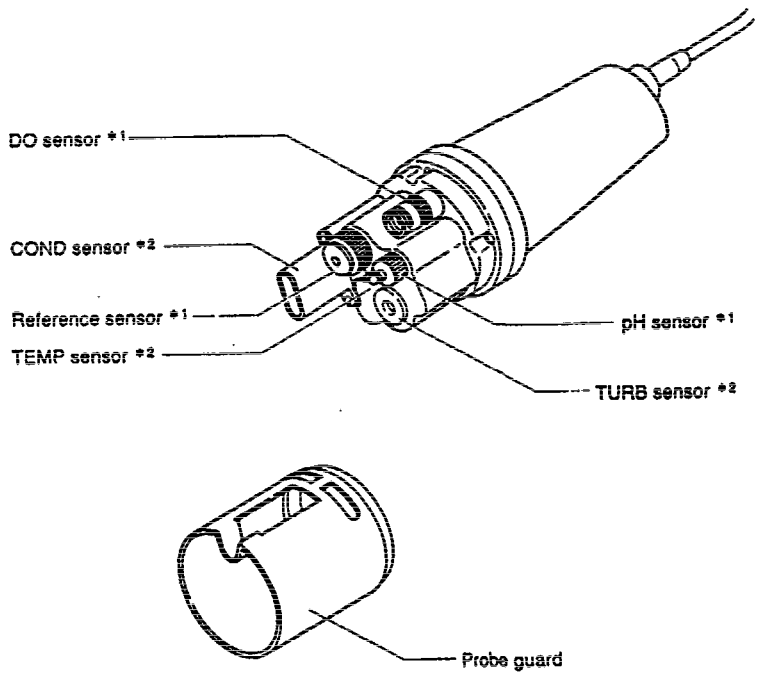
Probe

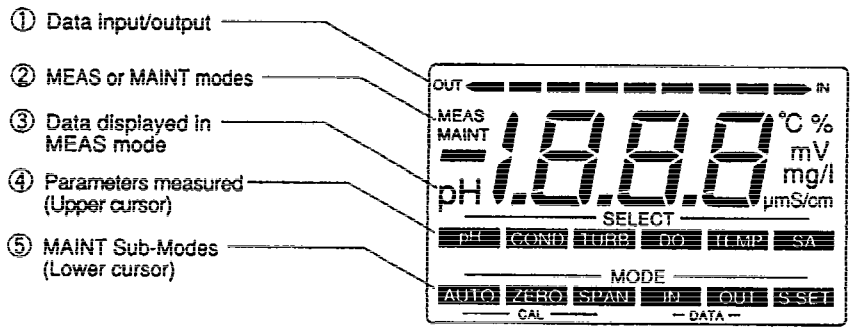


*1. Removable
  DO (Dissolved oxygen)
  Reference
  pH

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

The Readout

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



① Data input/output

OUT ═══ Data output

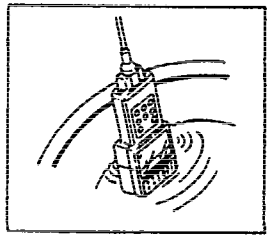
═══ IN Data input

② MEAS or MAINT modes

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

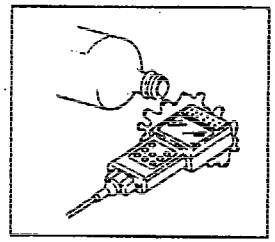
MEAS the U-10 is ready to make 6-parameter measurements

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



- Never immerse the main unit directly in water.

  The main unit is water-resistant and may be safely used in the rain; however, it is not of waterproof construction. Immersing the main unit in water or any other liquid can damage the internal electronic circuits

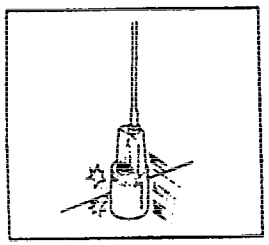


- Never allow any organic solvent to come in contact with either the probe or the main unit. This includes such organic solvents as methylethyl ketone (MEK) and acetone.

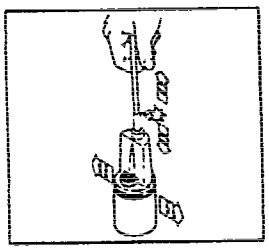
  (The probe is made of polyphenylene ether (PPE); the main unit case is acrylic resin.)

Precautions when using the U-10

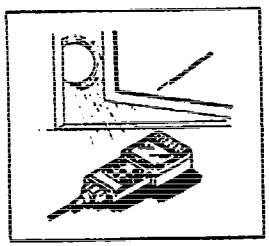
The U-10 Water Quality Checker is carefully designed for trouble-free operation. However, it is a sophisticated electronic instrument, and it can be damaged if used carelessly. Please read the following precautions and observe them when using your U-10 Water Checker.



- Do not swing or jerk the probe by its cable.
- Do not subject the cable connector to stress by pulling or stretching it.



- Do not drop either the U-10 probe or main unit. Never subject either component to sudden impact.



- Do not store the U-10 where it may be exposed to prolonged direct sunlight. Never leave the U-10 inside a vehicle with the windows closed.

③ Data displayed in MEAS mode

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

④ Parameters measured

Value displayed on readout is highlighted by upper cursor.

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

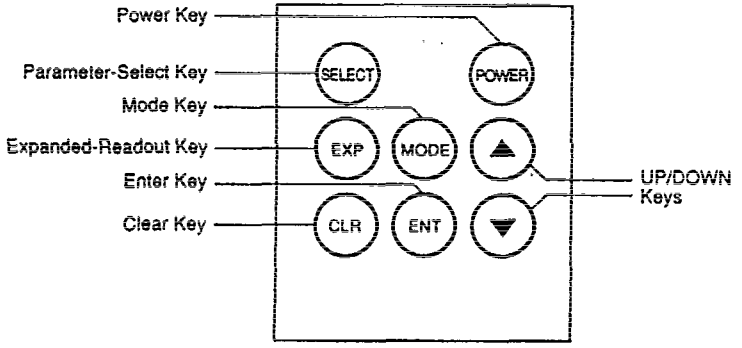
⑤ MAINT Sub-Modes

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

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

The Keypad

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



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

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

pH → COND → TURB → DO → TEMP → SAL

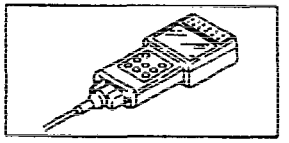
(MODE) **Mode Key (MODE)**
Toggles back and forth between MEAS and MAINT modes. When in the MAINT mode, this key toggles the lower cursor through the six maintenance Sub-Modes.

AUTO → ZERO → SPAN → IN → OUT → S.SET

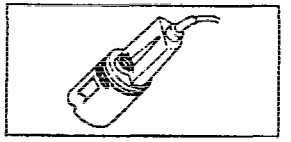
Unpacking the U-10

The following items are included with your U-10 Water Quality Checker.
When you unpack the probe and main unit, confirm that all the other accessories are included as well.

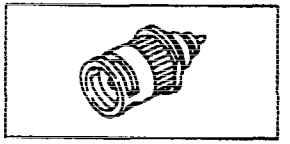
- Main unit



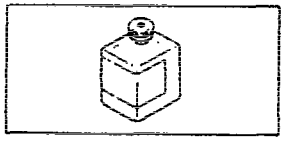
- Probe



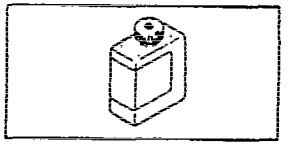
- DO sensor



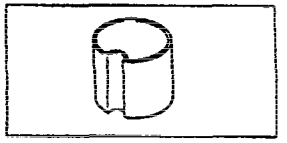
- Standard solution (pH 4 standard solution, 100-4) 500 ml bottle



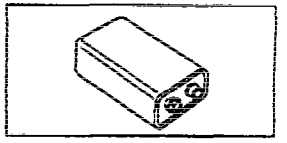
- Reference solution 250ml bottle



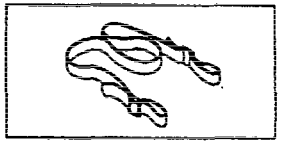
- Calibration breaker



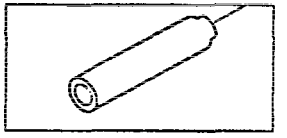
- 9V battery (6F22)



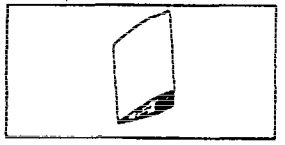
- Carrying strap for main unit



- DO sensor tool

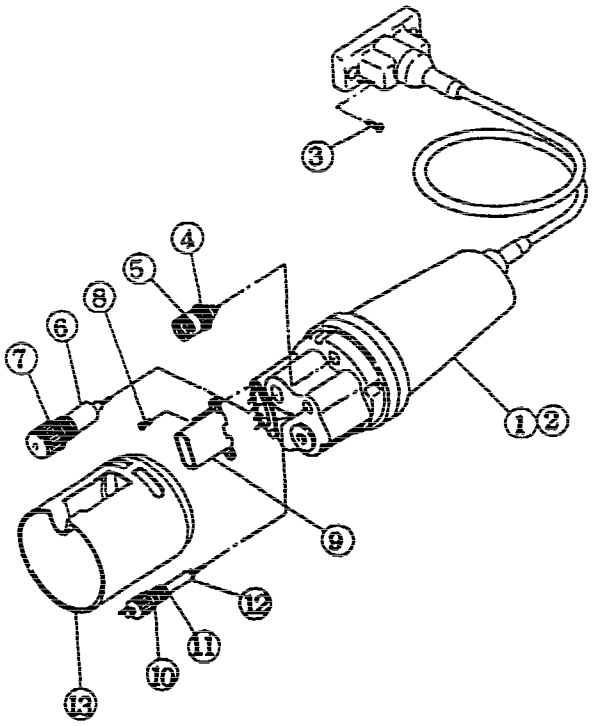


- This Instruction Manual



- Carrying Case

Exploded Views—prove



NO.	PARTS NO.	PARTS NAME	DESCRIPTION
3	F020911500	SCREW, PANHEAD	JISB1111 M3X6 (S-ZN3)
4	F020518700	O-RING	NOK S 11.2 (SI)
5	9037004900	DO SENSOR	
6	9037005000	REFERENCE	
7	F020246900	O-RING, S18	NOK S18 FPM
8	F020009500	SCREW, PANHEAD	M3-6L SUS304
9	H542141-01	COND GUARD	
10	F020058000	O-RING, P9	B2401 P9 FPM
11	9037004800	PH TIP	
12	F020058100	O-RING, P5	B2401 P5 FPM
13	H358290-01	PROTECTING TUBE	

EXP

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

ENT

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

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

CLR

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

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

▲
▼

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

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

Setting up the U-10

Preparations of the pH sensor and the reference sensor

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

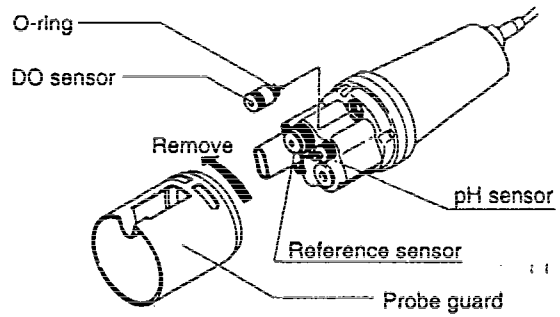
Inserting the DO sensor

WARNING

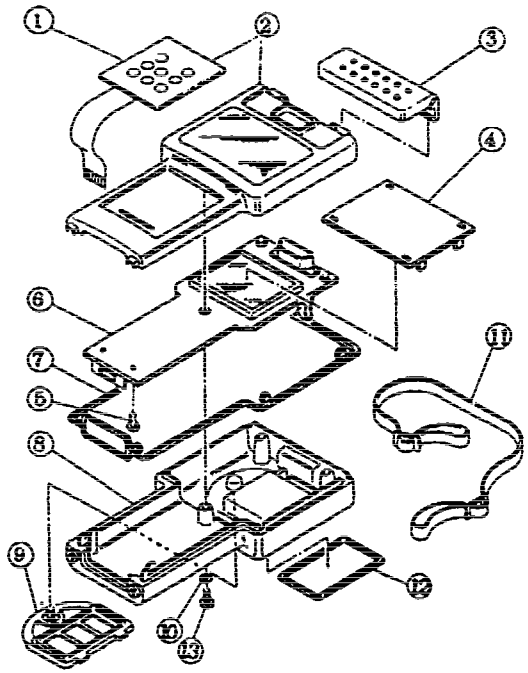
The DO sensor contains a strong alkaline solution. Should any of this solution come in contact with your clothing or skin, wash it away immediately with plenty of water. Be especially careful not to allow any of the liquid in the DO sensor to get in your eyes.

The Dissolved-Oxygen (DO) sensor has a delicate membrane that can easily be ruptured. For safety's sake, the U-10 is shipped to you with the DO sensor packed separately. You should insert the DO sensor when you unpack your U-10 unit.

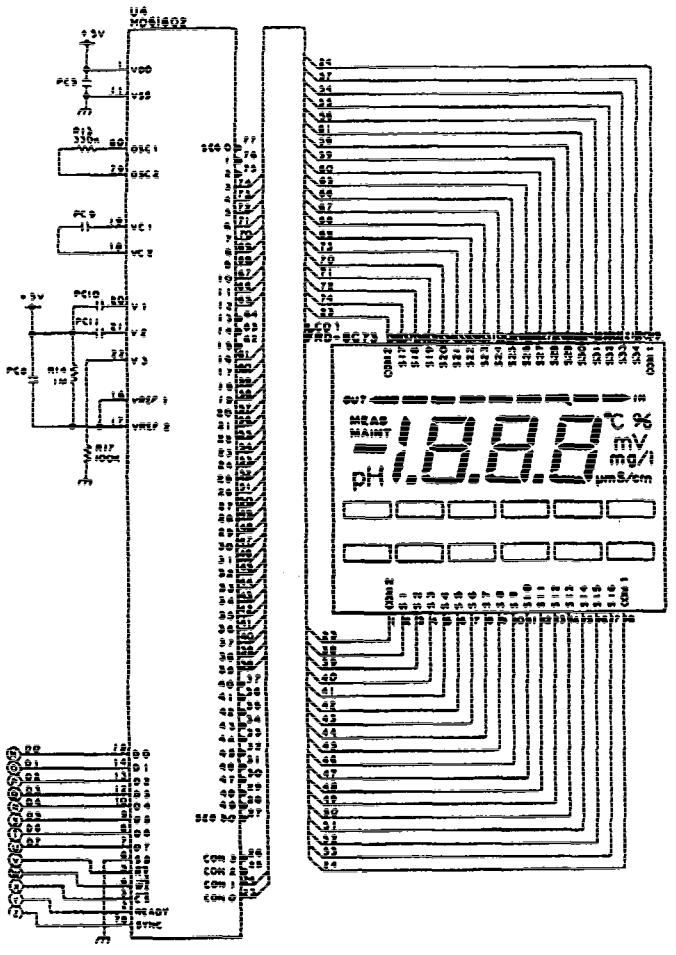
1. Make sure that the DO sensor has the correct O-ring, as shown.
2. First, fit the DO sensor lightly into its socket, and then put on the probe guard to align it correctly.
3. Then, tighten the DO sensor securely to the probe body. When doing this, be especially careful not to damage the membrane, which is located in the front of the DO sensor.



Exploded Views—main unit



NO.	PARTS NO.	PARTS NAME	DESCRIPTION
1	H357911-01	SHEET SWITCH	WATER CHECKER U-10
2	U800842300	CASE ASSY, TOP	U-10 Including ①
3	H357944-01	PRT COVER	U-10 METER
4	H542233-01	WINDOW, LCD	U-10 H357887-01
5	F020527500	TAPPING SCREWS	M3X6 (S-ZN3)
6	U800842400	PCB ASSY	U-10
7	H357945-01	CASE PACKING	U-10 METER
8	U800842500	CASE ASSY, BOT	U-10
9	U800842600	COVER ASSY, BAT	U-10
10	H543958-01	SEAR WASHER	U-10 METER
11	H544105-01	METER STRAP	U-10 20X1300 T=1.8
12	H542137-01	BATTERY PACKING	U-10 METER
13	F020911500	SCREW, PANHEAD	JISB1111 M3X6 (S-ZN3)



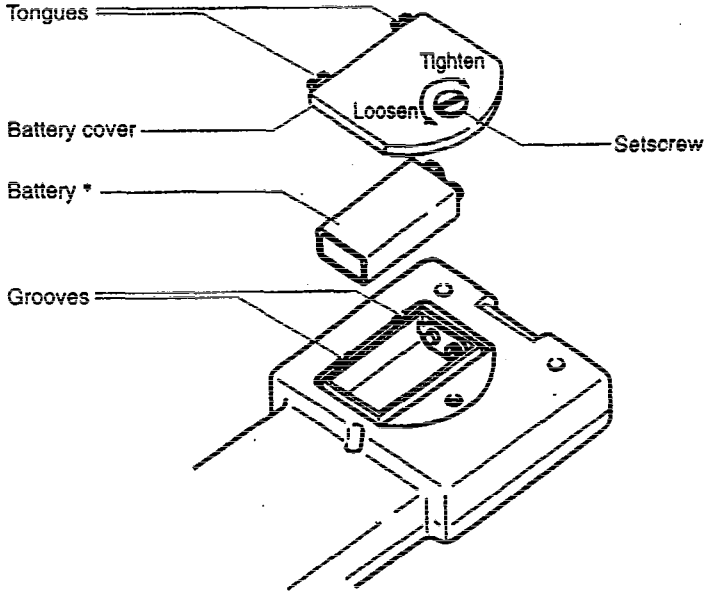
Inserting the battery

The U-10 is shipped from the factory with the battery packed separately.

The battery may be inserted by loosening the set-screw on the battery cover and pulling up the cover. Make sure that the plus and minus poles of the battery match the terminals correctly.

If the readout shows the message Er 1, it means that the battery is defective or exhausted and should be replaced.

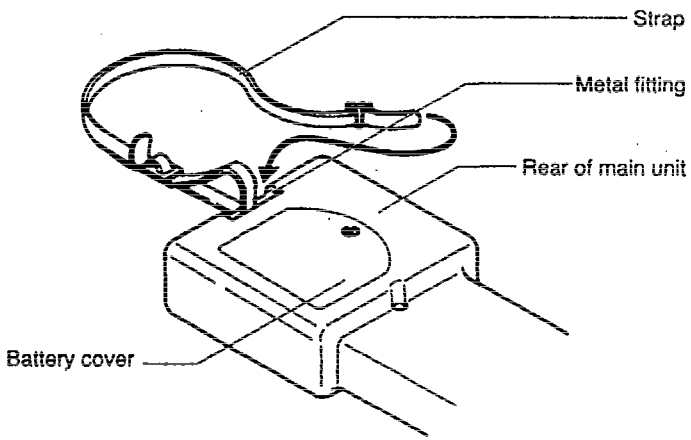
If you are replacing the battery and already have data stored in the U-10 memory that you wish to save, be sure to turn OFF the POWER Key before you remove the old battery. This will assure that data stored in memory will be maintained by the internal backup battery.

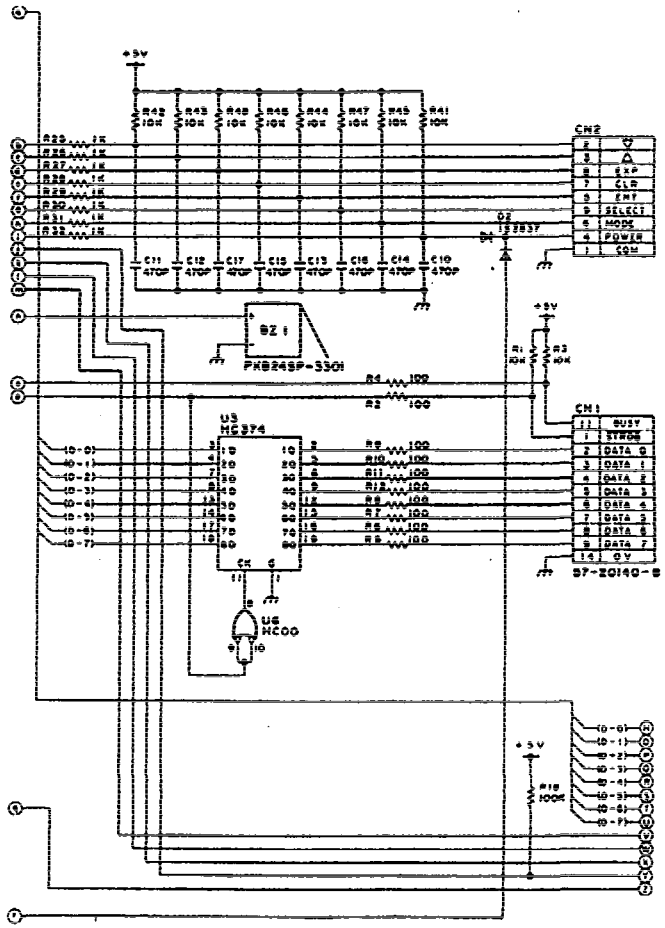


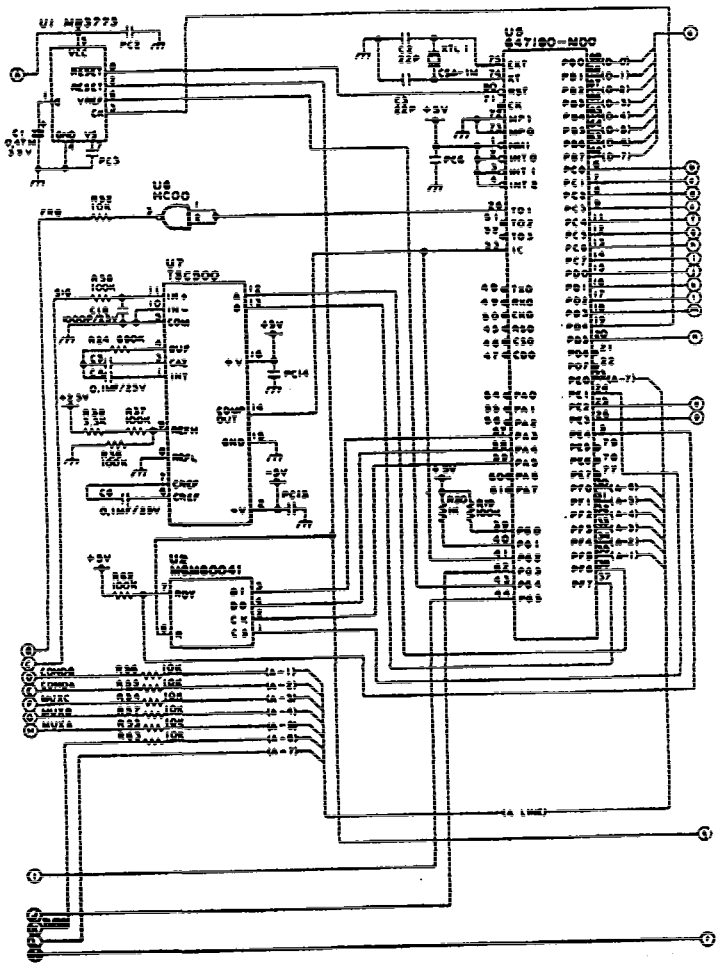
* Use a 9V-battery.

Attaching the carrying strap

Hook both ends of the strap through the metal fitting on back of the main unit, as illustrated.







Section 2

Making Measurements

Making a measurement with the U-10 Water Checker is extremely simple. Just turn on the power and place the probe in the sample of water you wish to measure.

All six parameters are measured simultaneously. These parameters may be stored in memory, printed out, or viewed one-by-one on the LCD readout. For printing and data storage, see the appropriate sections following this one. To view the parameters one-by-one on the readout, use the SELECT Key to toggle the upper cursor through them.

While the U-10 is both rugged and precise, the key to accurate measurements is cleanliness and frequent calibration. It is essential to clean the U-10 thoroughly after each measurement, and it is recommended that you re-calibrate your U-10 as frequently as possible. For best results, you should recalibrate it before each measurement session. Cleaning and calibration procedures are described below in this section and in the following one.

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Expanded readout 15
Measuring fresh water 16
Measuring salt water 17
After measurement: Cleaning and storing the U-10 18

How to make a measurement

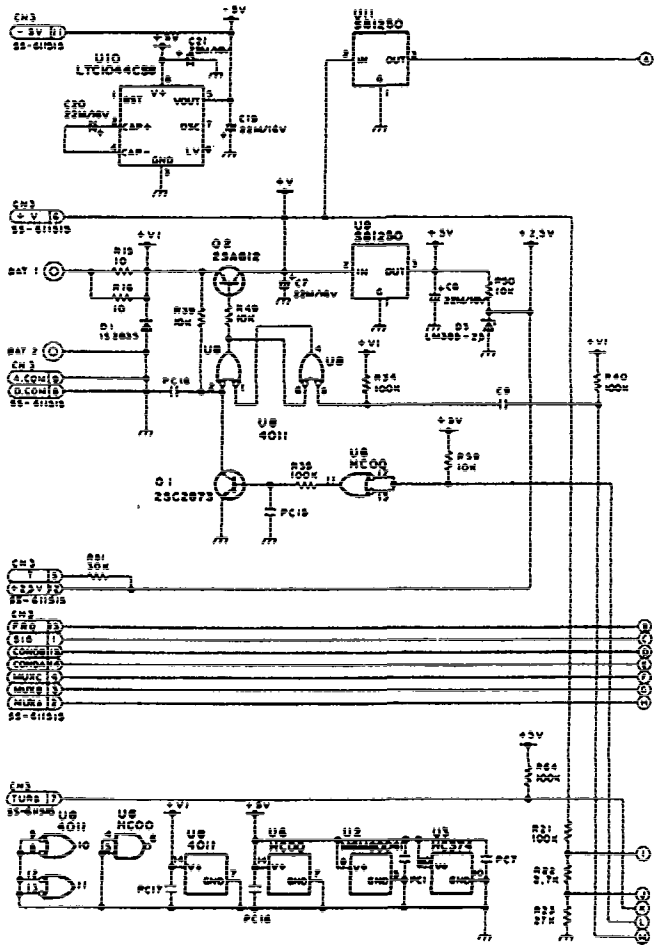
POWER **1** Turn the power on.

2 Gently place the probe into the water sample.

Basically, that's all there is to it: just turn it on and put the probe in the sample. Of course, the U-10 can do many sophisticated things with the sample data, and for best results, you should be careful about calibrating the unit and maintaining it in good condition. This is explained in detail below and in the next section.

Be careful!
Never drop or throw the probe into the water. It is a precision instrument containing five delicate sensors and five pre-amps; you can damage it beyond repair by unnecessary rough handling.

Circuit Diagram



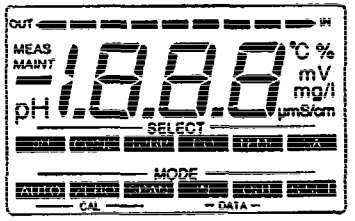
Parts List

The following expendable parts are available for the U-10 Water Checker.

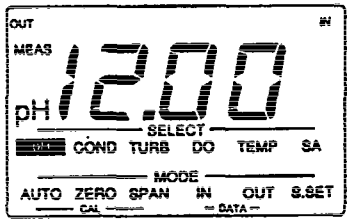
Part name	Model No.	P / N
Probe		9037-0047-00
pH sensor	#7112	9037-0048-00
DO sensor	#7542	9037-0049-00
pH reference sensor		9037-0050-00
Liquid junction (1 pair)	#7210	9037-0051-00
Reference solution	#330	9037-0052-00
pH standard solution pH2	100-2	9003-0015-00
pH standard solution pH4	100-4	9003-0016-00
pH standard solution pH7	100-7	9003-0017-00
pH standard solution pH9	100-9	9003-0018-00
Calibration beaker		9037-0053-00

Initial readout

When you first turn the power on, the U-10 will be in the MEAS mode, the readout will look like this, with all the LCD segments activated.



After about two seconds, the readout will change to show that a new measurement is being made. The readout will show the last parameter that the upper cursor was on when the previous measurement was made, i.e., pH as illustrated here.



(Expanded readout shown)

The display of the decimal point in the readout mode will also be in the same format as was selected with the EXP Key in the previous measurement, i.e., standard or expanded (as illustrated here).

Select the parameter you want shown on the readout of the measured data

(SELECT) All six parameters are automatically measured at once. Use the SELECT Key to toggle the upper cursor to the parameter you want.

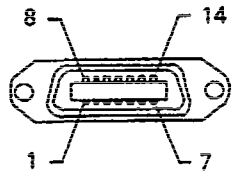
pH : pH
COND : Conductivity
TURB : Turbidity
DO : Dissolved oxygen
TEMP : Temperature
SAL : Salinity

To get a uniform reading, slowly move the probe up and down to circulate the water through it. (Move it 1 foot (30 cm) per sec.) Then wait for the readout to stabilize while doing this.

Common specification

Data storage	Max. 20 samples
Printer output	Centronics specs.
Power	Battery 9V, with auto power-off function
Operating temperature	0° - 45°C
Mass	Main unit: Approx. 400g Probe, with 2-m cable: Approx. 800g

- Output connector pin layout



Pin No.	Name	Pin No.	Name
1	STB	8	DB_8
2	DB_0	9	DB_7
3	DB_1	10	Not used
4	DB_2	11	BUSY
5	DB_3	12	Not used
6	DB_4	13	Not used
7	DB_5	14	GND

This equipment is in conformity with the following directive (s) and standard (s);

Directive (s)	the EMC Directive 89/336/EEC as amended by 91/263/EEC, 92/31/EEC and 93/68/EEC, in accordance with the Article 10 (1) of the Directive
Standard (s)	EN55011:1991 Class B Group 1 and EN50082-1:1992

Conductivity

Principle	4-electrode
Range	0-100ms/cm
Resolution	Standard: 0-1mS/cm : 0.01mS/cm 0-10mS/cm : 0.1mS/cm 10-100mS/cm : 1mS/cm Expanded: 0-1mS/cm : 0.01mS/cm 0-10mS/cm : 0.1mS/cm 10-100mS/cm : 1mS/cm
Repeatability	±1%/F.S. within each measurement range
Temperature compensation	0°-50°C
Readout	LCD
Calibration	1-point auto (Span) Manual 2-point

Turbidity

Principle	Scattered/Transmitted light
Range	0-800 NTU
Resolution	Standard : 10 NTU Expanded : 1 NTU
Repeatability	±3%/F.S.
Temperature compensation	—
Readout	LCD
Calibration	1-point auto (Zero) Manual 2-point

Salinity

Principle	Conversion based on conductivity
Range	0-4%
Resolution	Standard : 0.1% Expanded : 0.01%
Repeatability	±0.1%
Temperature compensation	0°-30°C
Readout	LCD
Calibration	—

Expanded readout

Use the EXP readout mode when you wish to see the results with one additional decimal place of accuracy. The EXP Key toggles the readout back and forth between standard to expanded display. The table below shows the result of using the EXP readout mode for each of the six parameters.

Table 1. Accuracy of expanded readout

Parameter	Range of measurement	Accuracy	
		Standard readout	Expanded readout
pH	0-14 pH	0.1 pH	0.01 pH
COND	0-1 mS/cm	0.01 mS/cm	0.001 mS/cm
	1-10 mS/cm	0.1 mS/cm	0.01 mS/cm
	10-100 mS/cm	1 mS/cm	0.1 mS/cm
TURB	0-800 NTU	10 NTU	1 NTU
DO	0-19.9 mg/l	0.1 mg/l	0.01 mg/l
TEMP	0-50°C	1°C	0.1°C
SAL	0-4%	0.1%	0.01%

Note that the salinity parameter is the only value not measured directly with its own sensor. The U-10 obtains salinity by converting the conductivity value. If large amounts of conductive ions other than salt-water components are present in the sample, an error may occur. Be cautious when interpreting the salinity results.

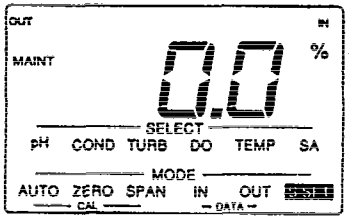
Measuring fresh water or salt water?

The U-10 can be set to the salinity for either fresh water or salt water when measuring DO. This is done by using the S.SET Sub-Mode.

Measuring fresh water

1. First, use the MODE Key to put the U-10 in the MAINT mode. Keep pressing the MODE Key to toggle the lower cursor to the S.SET Sub-Mode.

2. Once you are in the S.SET Sub-Mode, use the UP/DOWN Keys to select the salinity value. For fresh water, set the salinity to 0.0%.



3. Finally, press the ENT Key to complete the salinity setting while in the S.SET Sub-Mode.

4. When the salinity setting has been made, switch back to the MEAS mode by pressing the the MODE Key.

Specifications

pH

Principle	Glass electrode
Range	pH0-14
Resolution	Standard : 0.1pH
	Expanded : 0.01pH
Repeatability	±0.05pH
Temperature compensation	0°-50°C
Readout	LCD
Calibration	1-point auto (Zero)
	Manual 2-point

Temperature

Principle	Thermistor
Range	0°-50°C
Resolution	Standard : 1°C
	Expanded : 0.1°C
Repeatability	±0.3°C
Temperature compensation	—
Readout	LCD
Calibration	—

DO

Principle	Membrane galvanic cell
Range	0-19.9mg/l
Resolution	Standard : 0.1mg/l
	Expanded : 0.01mg/l
Repeatability	±0.1mg/l
Temperature compensation	0°-40°C
Readout	LCD
Calibration	1-point auto (Span)
	Manual 2-point

In pH meters a readout of this voltage between the two terminals is obtained by increasing it with an amplifier. In actual practice, the pH meter is first calibrated using a standard reference solution of known pH, then the pH of the sample liquid is measured.

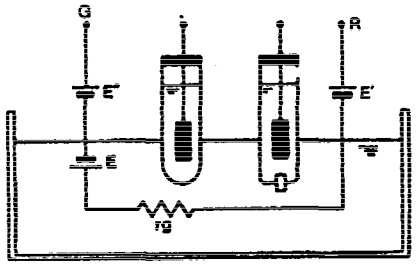
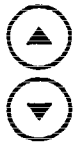


Fig. 5 Principle for Measuring pH

Measuring salt water

1. First, use the MODE Key to put the U-10 in the MAINT mode. Keep pressing the MODE Key to toggle the lower cursor to the S.SET Sub-Mode.



2. For salt water, set it to *R i.e.*, for auto-salinity.
   The *R* setting should be sufficient for measurements of normal sea water with a salinity value close to 3.3%. For sea water of an unusual salinity, however, and where the value is otherwise known, you may wish set the value manually to any salinity within the range of 0.0%-4.0%. (You may also possibly want to use a manual setting if, for example, the COND sensor is malfunctioning but it is still desirable to take readings of the other parameters.)

3. Finally, press the ENT Key to complete the salinity setting while in the S.SET Sub-Mode.

4. When the salinity setting has been made, switch back to the MEAS mode by pressing the the MODE Key.

After measurement: Cleaning and storing the U-10

POWER

1. Turn OFF the power.

2. Wash the probe thoroughly with tap water. Be sure to flush off all of sample solution from the probe.

Storing the U-10 for brief periods, *i.e.*, about 1 week or less:

Fill the calibration beaker with tap water and fit the probe over it.

For longer storage

The pH sensor must always be kept moist. Fill the small rubber cap with water and use it to cover the pH sensor.

The KCl internal solution in the reference sensor may seep out over time. Place vinyl tape around the O-ring portion to prevent this.

If you are going to store the U-10 for a prolonged period without using it, remove the battery from the main unit.

pH

Principle of measurement

The following is the basic equation for obtaining pH:

$$\mathbf{pH = -\log aH^+}$$

Where,

$\mathbf{aH^+}$: the activity of hydrogen ions

If a thin glass membrane is used to separate two liquids of differing pH values, an electric current will be generated in proportion to the difference between these two pH values. The value of this electrical current, *E(V)* , is shown by the folowing Nernst equation:

$$\mathbf{E = 0.0001983T\ (pH_1 - pH_0) + e}$$

Where,

- $\mathbf{T}$: the temperature of the liquids
- $\mathbf{pH_1}$: the pH of the internal liquid (i.e., inside the glass membrane)
- $\mathbf{pH_0}$: the pH of the sample liquid (i.e., the illiquid outside the glass membrane)
- $\mathbf{e}$: the assymetric potential

A conventional glass electrode for measuring pH contains a fluid inside the electrode with a pH of 7. If this is used to measure a sample that also has a pH value of 7, the assymetric potential will be close to 0V. Consequently, when a glass pH electrode is immersed in an acid solution, a positive electric current is generated; when it is immersed in an alkaline solution, a negative electric current is generated.

For actual use in a pH meter, a pair of reference electrodes with extremely stable characteristics is used. These are configured as shown in **Fig. 5**. As shown in **Fig. 5**, it can be seen that the electrical potentials generated in the internal electrodes, *E'* and *E"*, are canceled out by each other, so that the only electrical potential difference obtained is the current generated by the glass membrane, *E*, through the resistance of the membrane, *r*, and transmitted to terminals *G* and *R*.

DO correction for salinity

When a solution and air are in contact and in complete equilibrium (saturated), DO:C[mg/*l*] in the solution, and the oxygen partial-pressure:Ps[MPa] in air are in the following relation:

$$C = Ps/H$$

H [*MPa*/(mg/*l*)] is referred to as Henry's constant, which depends on the composition of the solution. In general, *C* becomes smaller as the salinity in the solution increases, since *H* becomes larger.

A DO sensor is intended to detect *Ps* in the above expression. Therefore, the DO measurement of an aqueous solution containing salt would be in error if the DO electrode were standardized either on air-saturated pure water or on air. To settle this problem, it is necessary to correct the DO reading based on the salinity of the sample.

Conventional DO meters make this salinity correction by inputting a known salinity value. This poses no problems if the salinity of the sample is known. In practice, however, the salinity of the sample is usually not known, unless measured by a device such as the U-10. Therefore, until now, DO meters have not been practical, even if they were provided with a salinity-correcting function.

The U-10 is capable of measuring the salinity of a sample and automatically correcting the DO reading for the amount salinity measured in the sample.

Section 3

Calibrating the U-10

The U-10 Water Checker may be calibrated either manually or automatically. The 4-parameter auto-calibration procedure is quite handy and should be sufficient for most measurement operations.

Manual calibration for each of the four parameters is more accurate but, of course, also more time-consuming. This method should be used for more precise measurement. The manual calibration procedure is explained below in detail, following the description of the auto-calibration procedure.

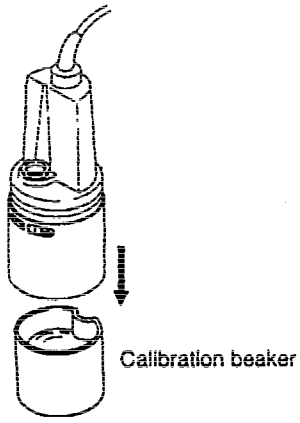
The auto-calibration procedure is extremely simple. The U-10 Water Checker uses just a single solution to do a simultaneous calibration of four parameters: *pH*, *COND*, *TURB*, and *DO*. Your U-10 comes with a bottle of standard phthalate pH solution and a calibration beaker for this purpose.

Auto-calibration procedure 20
Manual (2-point) calibration procedures 23
pH Calibration 24
1.Zero calibration 24
2.Span calibration 25
COND Calibration 26
1.Zero calibration 28
2.Span calibration 29
TURB Calibration 30
1.Zero calibration 31
2.Span calibration 31
DO Calibration 32
1.Zero calibration 33
2.Span calibration 33

Auto-calibration procedure

Fill the calibration beaker to about 2/3 with the standard solution. Note the line on the beaker.

Fit the probe over the beaker, as illustrated. Note that the beaker is specially shaped to prevent the DO sensor from being immersed in the standard solution. This is because the DO auto-calibration is done using atmospheric air.



(MODE) With the power on, press the MODE Key to put the unit into the MAINT mode. The lower cursor should be on the AUTO Sub-Mode; if it is not, use the MODE Key to move the lower cursor to AUTO.

(ENT) With the lower cursor on AUTO, press the ENT Key. The readout will show CAL. Wait a moment, and the upper cursor will gradually move across the four auto-calibration parameters one-by-one: *pH, COND, TURB,* and *DO.* When the calibration is complete, the readout will briefly show End and then will switch to the MEAS mode.

The upper cursor will blink while the auto-calibration is being made. When the auto-calibration has stabilized, the upper cursor will stop blinking.

Dissolved-Oxygen (DO)

Principle of measurement

The "DO" referred to here means the concentration of oxygen dissolved in water.

Fig. 4 shows the principle of measurement using a DO sensor.

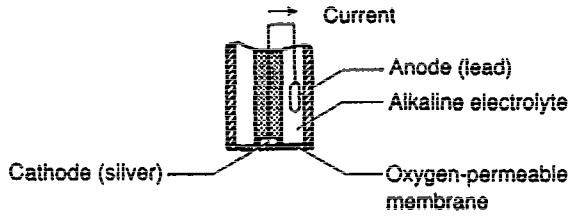


Fig. 4 Principle of DO sensor

A noble metal (silver) is fitted closely to an oxygen-permeable diaphragm to make the cathode; a base metal (lead) is used as the anode. Both are immersed in an alkaline electrolyte with the anode-to-cathode external circuit complete. Oxygen diffusing through the oxygen-permeable membrane causes a reduction reaction at the cathode; this allows flow of current in the external circuit:

$$O_2 + 2H_2O + 4e^- = 4OH^-$$

At the anode, oxidation reaction occurs as follows:

$$2Pb = 2Pb^{2+} + 4e^-$$

The current is proportional to the quantity of oxygen diffusing through the oxygen-permeable diaphragm. Accordingly, measurement of the current makes the DO in a sample known.

The DO measuring method based on this principle is called the *membrane-electrode method.* This method allows convenient measurement of DO, especially when compared with chemical-analysis methods, which need complicated pre-treatment to eliminate the effects of oxidizing or reducing substances.

Salinity (SAL)

The U-10 is designed to measure salinity as well as the other parameters.

Note that the "salinity" referred to here is the salinity of sea water. There is a constant relation between conductivity and salinity at certain temperatures.

Therefore, if data on the conductivity and temperature are available, the corresponding salinity is known. In other words, the salinity measurement of the U-10 is based on the principle of calculating the salt content, making use of the measured values of conductivity and temperature.

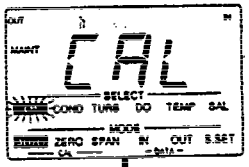
Note carefully, therefore, that measured results of all substances whose conductivity is detected are displayed as salinity. For example, the measured result is displayed as NaCl concentration, even if in fact the sample component is, for example, hydrochloric acid (HCl).

Temperature

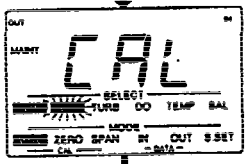
Temperature changes in water have extreme biological effects on the life cycles of fish and seaweed, as well as on that of the minute organisms that cleanse the water of organic pollutants. In general, as the temperature of water increases, the amount of oxygen dissolved in the water decreases and there is a tendency for the amount of pollutants to increase.

The U-10 uses a thermistor to measure temperature. A thermistor also measures the change in electrical resistance accompany changes in temperature; these changes in resistance are measured by the thermistor and are used to calculate the temperature.

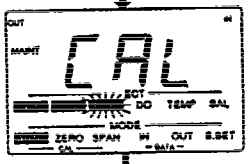
This temperature data is used by the U-10 in four different ways: (1) In pH temperature compensation, (2) in conductivity temperature conversion, (3) in the calculation of salinity, and (4) in dissolved-oxygen temperature compensation.



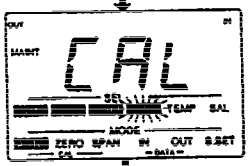
First, pH is being auto-calibrated



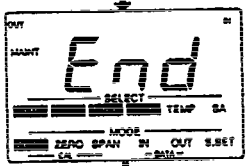
Then, COND is being auto-calibrated



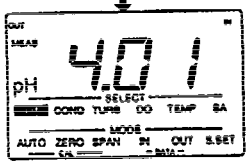
Next, TURB is being auto-calibrated



Finally, DO is being auto-calibrated



Auto-calibration now ends

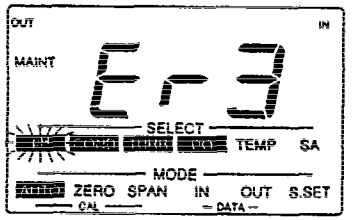


And the readout switches to the MEAS mode

Note: If you wish to abort the auto-calibration for any reason, press the CLR Key. The parameters auto-calibrated so far will be stored in memory.

Auto-calibration error

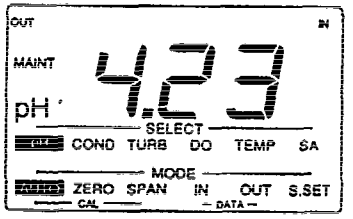
After the DO auto-calibration, if the unit does not switch to the MEAS mode as it should, and the readout shows either Er3 or Er4, an auto-calibration error has occurred. Parameters will blink where an error occurred.



pH auto-calibration error

CLR

If this happens, re-do the auto-calibration. First, press the CLR Key to cancel the error code.



ENT

Then press the ENT Key to re-start the auto-calibration. Restart the auto-calibration beginning again with pH.

NTUs (Nephelometric Turbidity Units)

For the calibration of turbidity, the U-10 uses a standard formazine solution.

Kaolin has been the conventional standard solution for many years. However, the composition of kaolin solutions often vary depending on the country of origin, and turbidity varies with the degree of purify. Furthermore, there is often individual error in preparing the solution. Kaolin is thus known for bringing about very large disparity in measurement results. As a turbidity standard solution, formazine standard solution is now increasingly being used internationally. In view of these facts, the U-10 uses the formazine standard solution for its calibration of turbidity.

In addition, the U-10 uses *NTUs* as the unit of turbidity. Other units conventionally used are formazine degrees and *FTUs*.

When the measurement of turbidity is based on the phenomenon of scattering, the use of *NTUs* is preferable, and in fact, these are being used increasingly. It should be noted that *NTUs* used as turbidity units of the formazine standard solution are equivalent to formazine degrees and to *FTUs*.

Turbidity (TURB)

Principle of measurement

From among several types of turbidity-measuring methods available, the U-10 uses the light-absorption-scattering method, shown in Fig. 3.
Irradiation of a beam of light onto a sample brings about separation of the beam into (1) the light transmitted by the solution and (2) the light scattered by turbidity components in the sample. In the light-absorption-scattering method, the intensity of both transmitted light and the scattered light are measured using separate receptors, and the turbidity is obtained based on the ratio of the two.

With the U-10, the light source is a pulse-lighting infrared-emission diode. The scattered light is measured at a point 30° offset from the light source. This light-absorption-scattering method has several advantages, including the fact that (1) the actual color of the sample fluid has little effect on the measurement of turbidity, (2) fluctuations in light quantity from the light source are easily compensated for, and (3) it allows the U-10 to be operated with relatively low-power consumption.

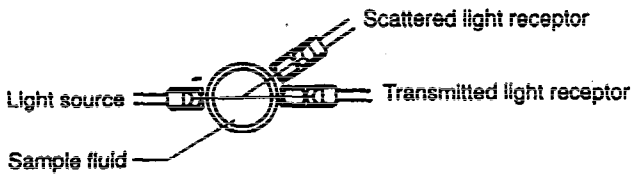


Fig. 3 Principle of the light-absorption-scattering method

Manual (2-point) calibration procedures

For normal measurements, the 4-parameter auto-calibration described above is sufficiently accurate. However, you may wish to do a parameter-by-parameter, 2-point manual calibration of one or more of the four parameters. This is recommended either for high-accuracy measurements, especially when using the expanded readout mode. It is necessary if a new probe is being used for the *first time*.

Parameters to be calibrated manually.

pH	• Zero	(see page 24.)
	• Span	(see page 25.)
COND	• Zero	(see page 28.)
	• Span	(see page 29.)
TURB	• Zero	(see page 31.)
	• Span	(see page 31.)
DO	• Zero	(see page 32.)
	• Span	(see page 33.)

Parameters not to be calibrated.

Sample temperature
Salinity

pH calibration

pH calibration on the U-10 is done using two commercially-available standard solutions of different pH values, one for the zero calibration, the other for the span calibration. Note that the temperature characteristics of the various standard solutions that are available may differ; therefore, before using these two solutions to make the pH calibration, carefully measure the temperature and determine the temperature characteristics of each.

Preparation

Wash the probe 2-3 times, using de-ionized or distilled water. Place it in a beaker of each standard solution.

1. Zero calibration

Use a pH7 standard solution for the zero calibration.

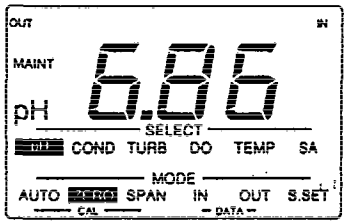
Operation

1. With the power on, press the MODE Key to put the unit into the MAINT mode.

2. Press the MODE Key again to move the lower cursor to ZERO.

3. Use the SELECT Key to move the upper cursor to pH.

4. When the readout has stabilized, use the UP/DOWN Keys to select the value of the pH 7 standard solution at the temperature of the sample. Refer to Table 2 for pH values of standard solutions at various temperatures.



5. Press the ENT Key to complete the zero calibration for pH.

Temperature compensation

In general, the conductivity of a solution varies largely with its temperature. The conductivity of a solution depends on ionic conductivity, described earlier. As the temperature rises, conductivity becomes higher, since ions begin to move more actively.

The temperature coefficient shows the change in % of conductivity per °C, with a certain temperature taken as the reference temperature. This is expressed in units of %/°C. The temperature coefficient assumes the premise that the conductivity of a sample changes linearly according to temperature. Strictly speaking, with actual samples, however, conductivity changes along a curve.

Furthermore, these curves form different shapes depending on the type of sample. In the ranges of smaller temperature changes, however, samples are said to have the temperature coefficient of 2%/°C; this holds for most samples, except in certain special cases. The U-10 Water Checker uses an automatic temperature conversion function to calculate conductivity at 25°C at a temperature coefficient of 2%/°C, based on the measured value of the temperature. Results are displayed on the readout. The U-10's temperature conversion function is based on the following formula.

$$L_{25} = L_t / \{1+0.02(t-25)\}$$

Where,

- L_{25}: Conductivity of solution converted to 25°C (value displayed on U-10)
- t: Temperature of solution at time of measurement (°C)
- L_t: Conductivity of solution at t (°C)

Let us assume that the current, *I(A)*, flows in a sample of conductivity *L*—under automatic control of the voltage-applying electrodes—so that the voltage at the voltage detecting-electrodes, *E(V)*, remains constant at all times. Then, the resistance of the sample, *R* (Ω), across the voltage-detecting electrodes is *R=E/I*. The resistance, *R*, of the sample is inversely proportional to its conductivity, *L*. That is, the conductivity, *L*, is proportional to the current, *I*. Accordingly, calibration of a standard solution of known conductivity, *Ls*, enables calculation of conductivity of a sample according to the formula *L=Ls(I/Is)* from the relation of *L:Ls=I:Is*.

Even in the 4-electrode method, polarization occurs, since *AC* current flows in the voltage-applying electrodes. The voltage-detecting electrodes are, however, free from the effects of polarization, since they are separated from the voltage-applying electrodes, and furthermore, current flow is negligible. Therefore, the 4-electrode method is an excellent method to enable measurement of conductivity covering a very high range.

2. Span calibration

Use either a pH4 or a pH9(10) standard solution for the span calibration.

Operation

1. Use the MODE Key to move the lower cursor to SPAN.

2. As in Step 4. above in zero calibration, when the readout has stabilized, use the UP/DOWN Keys to select the value of the standard solution (i.e., either pH4 or pH9) at the temperature of the sample. Again, refer to Table 2 for pH values of standard solutions at various temperatures.

3. Press the ENT Key to complete the span calibration for pH.

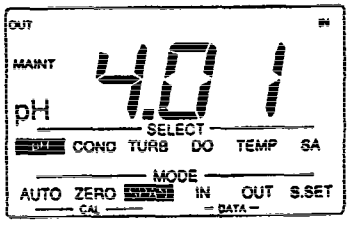


Table 2 pH values of standard solutions at various temperatures*

Temperature °C / °F	pH2[a]	pH4[b]	pH7[c]	pH9[d]	pH10[e]	pH12[f]
0 / 32	1.67	4.01	6.98	9.46	10.32	13.43
5 / 41	1.67	4.01	6.95	9.39	10.25	13.21
10 / 50	1.67	4.00	6.92	9.33	10.18	13.00
15 / 59	1.67	4.00	6.90	9.27	10.12	12.81
20 / 68	1.68	4.00	6.88	9.22	10.06	12.63
25 / 77	1.68	4.01	6.86	9.18	10.01	12.45
30 / 86	1.69	4.01	6.85	9.14	9.97	12.30
35 / 95	1.69	4.02	6.84	9.10	9.93	12.14
40 / 104	1.70	4.03	6.84	9.07	9.89	11.99
45 / 113	1.70	4.04	6.83	9.04	9.86	11.84
	1.71	4.06	6.83	9.01	9.83	11.70

a : oxalate, b : phthalate, c : neutral phosphate, d : borax,
e : carbonate, f : Sat.calcium hydroxide solution

* These pH valves are for Japanese standard solutions. Should you prefer to use different standard solutions, be sure to make the proper adjustments in calibration.

COND calibration

The U-10 can measure conductivity in the range of 0-100 mS/cm. Depending on the sample concentration, however, the U-10 automatically selects the proper range out of its three possible ranges of 0-1 mS/cm, 1-10 mS/cm, and 10-100 mS/cm.

Therefore, if you are doing a manual calibration for COND, this must be done for each of the three ranges. However, since the zero point is common for all three ranges, only the three one-point span calibrations need be done separately.

If direct current is sent between them, it will cause electroplating or decomposition, i.e., polarization; this results in inaccurate measurement of conductivity.

Even a flow of alternating current will also cause a certain amount of polarization. Measures must be taken to minimize the effect of this polarization, such as the application of platinum black plating to the electrode surfaces. In spite of such measures, however, the effect of polarization cannot be neglected in conductivity measurements of a high-conductivity solution. This makes accurate measurement difficult. Furthermore, depositions or stains on the electrode surfaces can cause a large apparent resistance, also making accurate conductivity measurement difficult.

The U-10 Water Checker has adopted the 4-electrode method to overcome these disadvantages of the the 2-electrode method. As shown in **Fig. 2**, the U-10 Water Checker uses two voltage-detecting electrodes and two voltage-applying electrodes, for a total of total four electrodes.

The voltage-detecting electrodes are for detecting AC voltage, and the voltage-applying electrodes are for applying AC voltage.

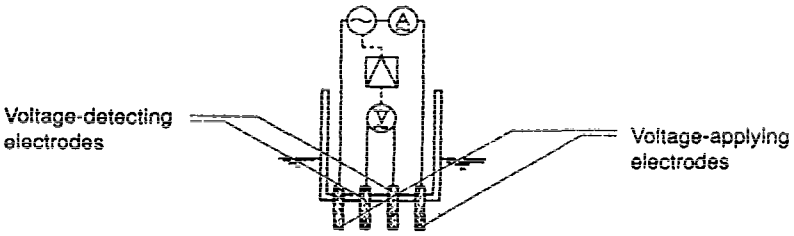


Fig. 2 Principle of the 4-electrode method

Conductivity (COND)

Principle of measurement

Conductivity is an index of the flow of electrical current in a substance.

Salts dissolved in water are separated into cations and anions. Such a solution is called an electrolytic solution. An electrolytic solution has the property of allowing the flow of current according to Ohm's law. This property is referred to *ionic conductivity*, since current flow is due to ion movement in an electrolytic solution. Metals, on the other hand, allow the flow of current by means of electrons. This property is called *electronic conductivity*, which is distinguished from ionic conductivity.

A cube 1 cm on each side, as each shown in Fig. 1, is used to demonstrate an electrolytic solution. Two electrode plates are placed on opposite sides, and the cube is filled with a solution. If the resistance between these two electrode plates represented by r (Ω), the conductivity of the solution L (S.cm^{-1}) is $L=1/r$. S stands for *Siemens*, a unit of measurement of conductance.

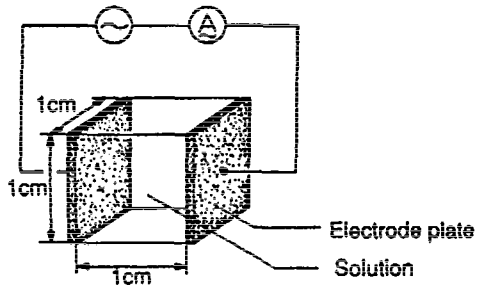


Fig. 1 Definition of conductivity

The most general method for measuring conductivity is based on the above principle, and is called the 2-electrode method. In this method, to take a measurement, it is necessary to allow flow of alternating current between the two electrode plates.

Preparing the standard solution for COND span calibration

This solution uses a potassium chloride as a reagent. For greater accuracy, the solution should be freshly prepared each time. If it is unavoidable to use a stored solution, be sure to keep it tightly capped in a polyethylene or hard glass bottle. The shelf life of this solution is six months. Date-stamp the bottle for reference. Never use a KCl standard solution that has been stored for more than six months: the calibration accuracy may be adversely affected.

Use potassium chloride powder of the best quality commercially available. Dry the powder for two hours at 105°C, and cool it down, in a desiccator. Weigh out an appropriate amount of dried and cooled potassium chloride powder according to the table below. Make the potassium chloride standard solution as shown.

Table 3 Making the potassium chloride standard solution

KCl standard solution	KCl weight g	Conductivity* mS/cm	Range to be calibrated mS/cm
0.005N	0.373	0.718	0-1
0.05N	3.73	6.67	1-10
0.5N	37.28	58.7	10-100

* Value at the temperature, 25°C

To prepare the standard solution, use a 1-liter volumetric flask. First, dissolve the KCl in a small amount of de-ionized or distilled water. Then fill the flask with de-ionized or distilled water up to the 1-liter line. Finally, shake the solution to mix it thoroughly.

1. Zero calibration

This calibration is carried out in atmospheric air; no solution is needed.

Preparation

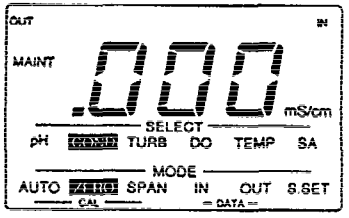
Wash the probe 2-3 times, using de-ionized or distilled water. Shake the probe to remove any water droplets from the COND sensor. Then allow it to dry by exposing it to fresh air.

Operation

1. Use the MODE Key to move the lower cursor to ZERO.

2. Use the SELECT Key to move the upper cursor to COND.

3. Use the UP/DOWN Keys to set the readout to zero.



4. Press the ENT Key. This completes the zero calibration for COND.

Section **6**

Reference Materials

The following descriptive information is provided for a better understanding of the U-10 Water Checker and its functions.

Conductivity (COND) 54
Turbidity (TURB) 58
Salinity 60
Temperature 60
Dissolved-Oxygen (DO) 61
pH 63
Specifications 65
Parts List 68

2. Span calibration

This procedure uses a standard solution of potassium chloride. For best results, a fresh batch of the solution should be prepared each time. See page 27 for details.

Preparation

Wash the probe 2-3 times using de-ionized or distilled water. Following this, wash it 2-3 times in the KCl standard solution you have prepared. Then place the probe in a beaker of the KCl solution maintained at a temperature of 25±5°C.

Operation

1. Use the MODE Key to move the lower cursor to SPAN.

2. After the readout stabilizes, as you did for the pH calibration, use the UP/DOWN Keys to select set the value of the KCl standard solution, referring to the KCl table.

3. Press the ENT Key to complete the span calibration for this COND range.

4. Repeat this procedure for the three ranges, using each of three values of KCl standard solutions.

TURB calibration

Use good-quality de-ionized water, which may be considered as having a turbidity of zero. If that is not readily available, distilled water may be used instead. When doing the turbidity zero calibration, it is particularly crucial that you clean the probe thoroughly. Never use a dirty probe; otherwise the calibration will be unreliable.

Preparing the standard solution for TURB span calibration

1. Weigh out 5.0 g of hydrazine sulfate.
2. Dissolve this in 400 m*l* of de-ionized or distilled water.
3. Then weigh out 50 g of hexamethylenetetramine, and dissolve it in 400 m*l* of de-ionized or distilled water.
4. Mix these two solutions, add enough de-ionized or distilled water to make 1,000 m*l*, and stir the mixed solution thoroughly.
5. Allow this solution to stand for 24 hours at a temperature of 25±3°C.

   The turbidity of this solution is equivalent to 4000 NTUs. The shelf-life of this solution is six months; i.e., this 4,000-NTU value will remain accurate for a maximum of six months.

   Each time you carry out this calibration, it is necessary to dilute the 4,000-NTU standard solution to prepare an 800-NTU standard solution for calibration. To do this, measure out 50 m*l* of the 4,000-NTU solution into a 250-m*l* measuring flask.

   It is recommended that you use a rubber pipette aspirator for this. Then add de-ionized or distilled water up to the 250-m*l* line.

   The standard solution used here for the turbidity calibration will precipitate easily. Therefore, be sure to stir the solution thoroughly before use.

Connect the new probe

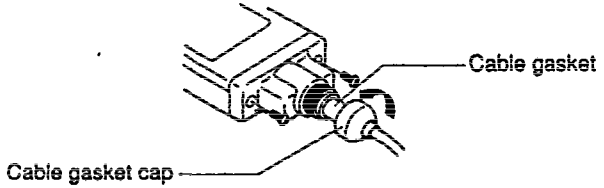
1. Insert the connector until it clicks.
2. Re-attach the cable-connector cover to the main unit.
3. Slide the cable gasket toward the cable-connector cover, and screw on the cable gasket cap.

   Before you use a new probe for the first time, it is necessary to calibrate it manually for all four parameters. Refer to Section 3, "Calibrating the U-10," for instructions on manual calibration.

Replacing a faulty probe

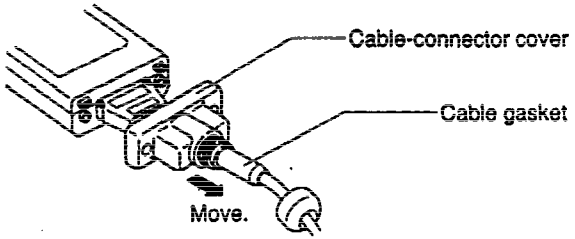
Disconnect the cable from the main unit

1. Loosen the cable gasket cap, and remove cap from gasket.



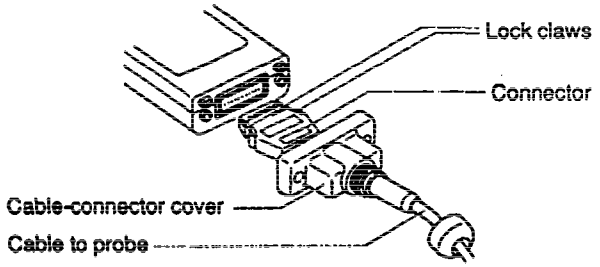
2. Slide back the gasket.

3. Back off the two screws on the cable-connector cover.



4. Slide off the cable-connector cover to expose the connector lock claws.

5. Press lock claws on both sides with your fingers to release the connector. Pull out the connector from the main unit.



1. Zero calibration

Preparation

Wash the probe thoroughly 2-3 times using de-ionized or distilled water. Shake off excess water droplets, and then place it in a beaker of de-ionized or distilled water.

Operation

1. Use the MODE Key to move the lower cursor to ZERO.

2. Use the SELECT Key to move the upper cursor to TURB.

3. After the readout has stabilized, set it to 0.0, using the UP/DOWN Keys.

4. Press the ENT Key to complete the zero calibration for TURB.

2. Span calibration

Preparation

Wash the probe thoroughly, using de-ionized or distilled water. Shake off excess water droplets. Then place it in a beaker of the 800-NTU solution you have prepared for this purpose.

Operation

1. Stir this 800-NTU span standard solution thoroughly.

2. Use the MODE Key to move the lower cursor to SPAN.

3. After readout has stabilized, i.e., about 60 to 90 seconds, set the readout to "800" NTU, which is the value for this standard solution.

4. Press the ENT Key to complete the span calibration for TURB.

DO calibration

Unlike the other calibration procedures, the solution for the DO calibration cannot be stored for use; because the amount of dissolved oxygen in the solution is crucial, a fresh batch must be prepared each time, just before it is used in the DO calibration.

1. Zero calibration

Use a solution of sodium sulfite dissolved in either de-ionized water or tap water.

Preparation

1. Add about 50g of sodium sulfite to 1,000 ml of water (either de-ionized water or tap water will do). Stir this mixtuer to dissolve.
2. Wash the probe 2-3 times in tap water, and place it in the zero standard solution.

Operation

1. Use the MODE Key to move the lower cursor to ZERO.

2. Use the SELECT Key to move the upper cursor to DO.

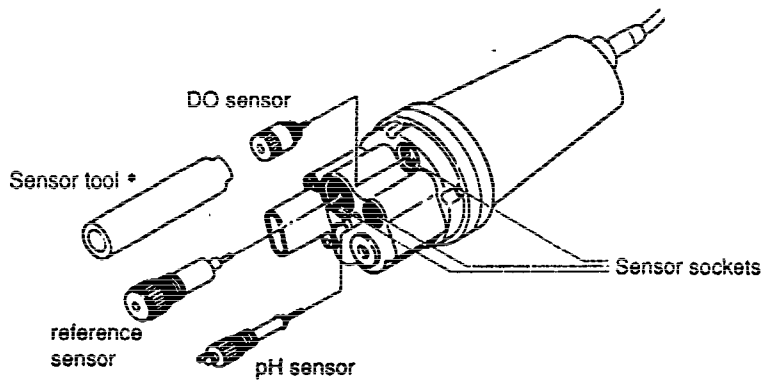
3. After the readout has stabilized, set it to 0.0, using the UP/DOWN Keys.

4. Press the ENT Key. This completes the zero calibration for DO.

Replacing faulty sensors

Three of the U-10's sensors are replaceable: the *pH sensor*, the *reference sensor*, and the *DO sensor*.
These may be replaced as follows.

1. Wipe off any water droplets from the probe.
2. Remove faulty sensor.
3. Insert the new sensor carefully with your fingers.
4. Be careful not to let the sensor sockets get wet.

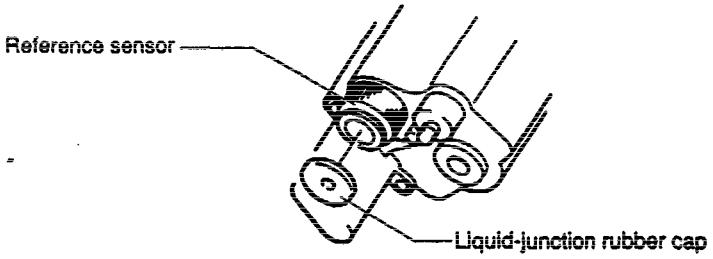


* When replacing the DO sensor, use the sensor tool provided as an accessory.

Recharging the reference sensor with reference solution

Recharge the reference sensor with reference solution about once every two months, as follows.

1. Remove the liquid-junction rubber cap from the reference sensor, and pour out the old solution.
2. Fill the reference sensor completely with new reference solution. Make sure there are no air bubbles.
3. Replace the liquid-junction rubber cap.
4. Carefully wash off all excess reference solution from the probe.



2. Span calibration

Use either de-ionized water or tap water that has been saturated with oxygen in air.

Preparation

1. Put 1 or 2 liters of water in a container (either de-ionized water or tap water will do). Use an air pump to bubble air through the solution until it is oxygen-saturated.
2. Wash the probe 2-3 times in tap water, and put it in the span calibration solution.

Operation

1. First, be sure the U-10 is set for fresh water readings. To do this, set the S.SET Sub-Mode to 0.0%.

MODE

2. Then, use the MODE Key to move the lower cursor to SPAN.
3. After the readout has stabilized, while slowly moving the probe up and down in the solution, set the readout value to the appropriate DO value for the temperature of this solution. For DO values at various temperatures, refer to Table 4.

ENT

4. Press the ENT Key to complete the span calibration for DO.

Table 4 Amounts of saturated dissolved oxygen in water at various temperatures, salinity = 0.0%

Temperature	DO	Temperature	DO
0 °C	14.16 mg/l	21 °C	8.68 mg/l
1	13.77	22	8.53
2	13.40	23	8.39
3	13.04	24	8.25
4	12.70	25	8.11
5	12.37	26	7.99
6	12.06	27	7.87
7	11.75	28	7.75
8	11.47	29	7.64
9	11.19	30	7.53
10	10.92	31	7.42
11	10.67	32	7.32
12	10.43	33	7.22
13	10.20	34	7.13
14	9.97	35	7.04
15	9.76	36	6.94
16	9.56	37	6.86
17	9.37	38	6.76
18	9.18	39	6.68
19	9.01	40	6.59
20	8.84		

Normal probe maintenance

Washing the turbidity sensor

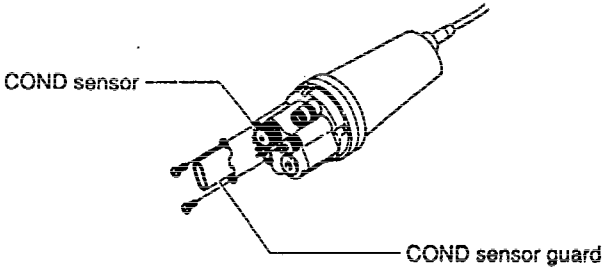
The sensor is a glass tube. Wash out the tube and remove stains carefully, using tap water and a test tube brush.

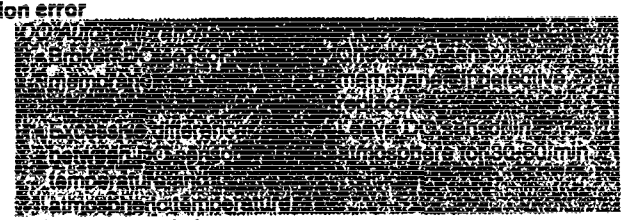
Be careful not to scratch the inside of the glass tube. Never use abrasives or cleansers.

Cleaning the conductivity sensor

Remove COND sensor guard, and carefully use a soft brush to clean off any dust from the sensor unit.

Be sure to replace the COND sensor guard before taking measurements.



Error Code	Cause	Action
Span-calibration error Er4		
	DO aqueous solution calibration	
	• Broken DO sensor membrane.	• Check DO sensor membrane. If defective, replace.
	• Contaminated electrode.	• Clean the electrode using a soft brush, taking care not to scratch membrane.
	• Insufficient agitation of solution.	• Agitate solution thoroughly.
Memory full Er5	• Data-sets for 20 samples are already in memory.	• To delete all data from memory, put the U-10 in the IN Sub-Mode mode and press the CLR Key.
Printer error Er6	• Jammed printer paper.	• Eliminate jamming of printer paper.
	• Poor cable connection .	• Replace the cable.
	• Wrong printer.	• Use proper parallel Centronics printer.
	• Defective printer.	• Replace the printer as necessary.

Section 4

Data Storage and Printout

The U-10 can store up to 20 sets of data, 120 data points, of the values measured for each of the six parameters: pH, COND, TURB, DO, TEMP, and SALINITY. Values stored in memory can be recalled to the readout as desired.

If a printer is connected to the U-10 printer port, whenever a Data-Set is either stored in memory or recalled to the readout, it can also be simultaneously output to the printer.

Storing data 36
Recalling data 38
Deleting data 40
Printing out data 41

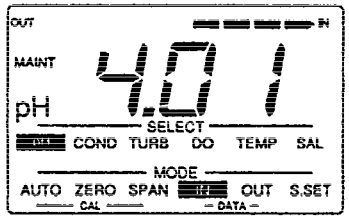
Storing data

MODE 1. Press the MODE Key to put the U-10 in the MAINT mode.

MODE 2. Continue to press the MODE Key to move the lower cursor to IN, the *Input* Sub-Mode.

SELECT 3. Use the SELECT Key to move the upper cursor to the parameter you wish to see on the readout.

ENT 4. When the readout stabilizes on a value, press the ENT Key. This will automatically input the set of six parameters for this measurement into memory.



The readout will first show the Data-Set No. for about two seconds. At the top right-hand corner, a dashed arrow points to IN, showing that data is being input. Then each parameter is automatically read into memory, one-by-one from pH to salinity. The upper cursor skips along to show this. If a printer is connected, these six values will also be printed out at the same time.

The upper cursor then returns to pH, with the U-10 still in the IN Sub-Mode.

ENT 5. You may now continue and input another set of data: simply press the ENT Key again.

The Data-Set No. will automatically advance one digit, and the next set of six parameters will be read into memory in the same manner. This procedure can be repeated for up to a total of 20 Data-Sets.

Error Code	Cause	Action
	for TURB • Contaminated or defective LED sensor	• Clean out the tube containing the LED turbidity sensor, using test tube brush and neutral detergent. Never use an abrasives or cleansers for this.
	for DO • Broken DO sensor membrane.	• Check the LED turbidity sensor. If it defective, the entire probe must be replaced. Check DO sensor. If defective, replace.

Span-calibration error

Er4	*for all parameters* • Poor connection in probe to maintunit cable • Water in one of the sensor sockets • Temperature of sample exceeds maximum scale of U-10	• Connect the cable securely. • Dry out the sensor sockets. • Replace the probe.
	for pH • Contaminated pH sensor. • Improper concentration of reference solution in reference sensor	• Clean the pH sensor. • Replace the reference solution.
	for COND • Contaminated COND sensor	• Clean the sensor using tooth brush and neutral detergent.
	for TURB • Contaminated or defective LED sensor	• Clean out the tube containing the LED turbidity sensor, using test tube brush and neutral detergent. Never use an abrasives or cleansers for this. • Check the LED turbidity sensor. If it defective, the entire probe must be replaced.

Error Codes

The U-10 has an easy-to-understand error message function so you can spot trouble readily. Error codes are displayed on the readout and the unit will beep if an error occurs.
(Note that if you press an incorrect sequence of keys, the unit will beep three times to indicate you have pushed the wrong key.)

Error Code	Cause	Action
Bad battery Er 1	• Defective or low battery	• Replace battery
Failure in main unit Er 2	• Malfunction of memory backup IC	• Push POWER Key to turn the U-10 ON again. If this error code is still displayed, contact your Horiba dealer for repair or replacement.
Zero-calibration error Er 3	*for all parameters*	
	• Poor connection in probe-to-main unit cable	• Connect the cable securely.
	• Water in one of the sensor sockets	• Dry out the sensor sockets.
	• Temperature of sample exceeds maximum scale of U-10	• Replace the probe.
	for COND	
	• Contaminated COND sensor	• Clean the sensor, using tooth brush and neutral detergent.

If 20 Data-Sets have been read into memory, the storage capacity is full and no more data may be input. The U-10 will beep three times to indicate the memory is full.

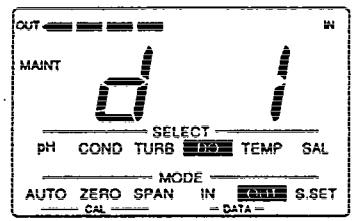
6. To return the readout to the previous setting in the MEAS mode, press the MODE Key again.

Recalling data

(MODE) 1. Press the MODE Key to put the U-10 in the MAINT mode.

(MODE) 2. Continue to press the MODE Key to move the lower cursor to OUT, the *Output* Sub-Mode. The readout will show d.1, meaning Data-Set No. 1.

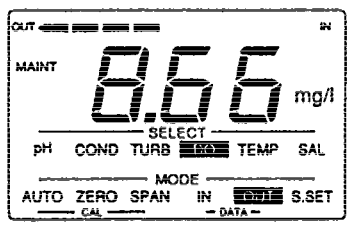
At the top left-hand corner, a dashed arrow points to OUT, showing that data can be output now to the readout.



(▲) (▼) 3. Use the UP/DOWN Keys to display the Data-Set No. of the values you wish to recall.

(SELECT) 4. Use the SELECT Key to move the upper cursor to the parameter you wish to view.

(ENT) 5. Press the ENT Key to display the data on the readout.



If a printer is connected, all six parameters in this Data-Set will also be printed out at the same time.

Section **5**

Daily Maintenance and Troubleshooting

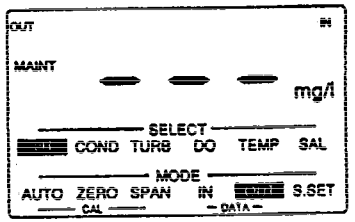
For accurate measurements and prevention of malfunction, routine careful maintenance of the U-10 is important. In particular, failure to maintain the sensors properly can lead to serious trouble or incorrect measurements. The U-10 is provided with error-code functions for the ready detection of potential problems.

Error codes 44
Normal probe maintenance 47
Replacing faulty sensors 49
Replacing a faulty probe 50

ENT

6. When the ENT Key is pressed again, the next Data-Set No. is displayed in order, i.e., d2, if two data sets are in memory. At this point, you can either press the ENT Key again to view the contents of this Data-Set, or you can use the UP/DOWN Keys to go up or down to another Data-Set No.

   If a particular Data-Set is empty, three dashes appear on the readout.



MODE

7. To return the readout to the previous setting in the MEAS mode, press the MODE Key again.

Deleting data

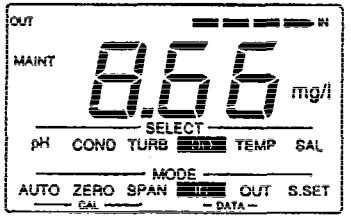
Set the U-10 as if you were going to input data:

MODE

1. Press the MODE Key to put the U-10 in the MAINT mode.

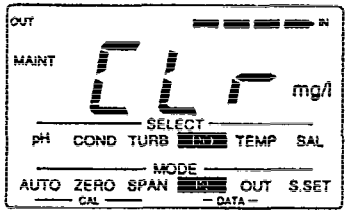
MODE

2. Continue to press the MODE Key to move the lower cursor to IN, the Input Sub-Mode.



CLR

3. Then, to erase all the data from all the Data-Sets in memory, press the CLR Key. The readout will show the message CLr for about two seconds.

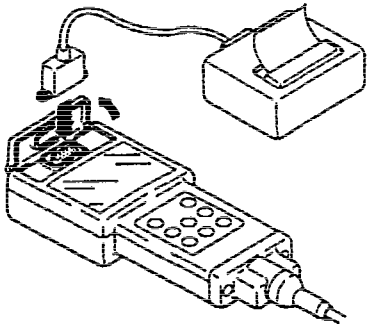


Be careful!
You cannot delete individual Data-Sets. The CLR Key always erases all data from memory.

Printing out data

If a printer is connected to the U-10 printer port, whenever a Data-Set is either stored in memory or recalled to the readout, it is also simultaneously output to the printer.

The U-10 printer port is a standard Centronics parallel port. To connect a parallel printer to the U-10: Open the rubber printer-port cover, located directly over the readout on the main unit, and connect the printer cable.



Note:
When a printer is not being used, disconnect the cable from the U-10 printer port, and close the cover tightly.

- **Sample printout**

```
NO. 1 DATE  /  /
 pH      5.0
 COND    1.5    mS/cm
 TURB  390      NTU
 DO      0.5    mg/l
 TEMP   23      °C
 SAL     3.8    %
NO. 2 DATE  /  /
 pH      3.1
 COND    1.3    mS/cm
 TURB  270      NTU
 DO      0.7    mg/l
 TEMP   25      °C
 SAL     0.1    %
NO. 3 DATE  /  /
 pH      3.1
```

Appendix B

Laboratory Standard Operating Procedures

Severn Trent Laboratories

B-1 Laboratory Method Detection Limits

B-2 Volatile Compounds by GC/MS (EPA 8260B)

B-3 Semi-Volatile Compounds by GC/MS (EPA 8270C)

B-4 Organochlorine Pesticides and PCBs by GC

B-5 Polychlorinated Biphenyls (PCBs) by GC/MS (Method 8082)

B-6 Chlorinated Herbicides

B-7 Continuous Liquid-Liquid Extraction

B-8 GEL Permeation Chromotography

B-9 Preparation of Organics in Biological Tissues

B-10 Total Organic Carbon-Shimadzu TOC Analyzer

B-11 Total metals by EPA by ICP/AES (Method 6010B)

Triangle Laboratories, Inc.

B-12 Dioxin / Dibenzofuran

B-1 Laboratory Method Detection Limits

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (ug/L)	RLA (ug/L)
Aluminum (ICP)	200.7(Drinking Water)	86	70-130	≤20	27	200
	200.7(NPDES)	3	75-125	≤20	27	200
	6010(3005/3010)	2	75-125	≤20	27	200
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	8.2	50
Antimony (ICP)	200.7(NPDES)	3	75-125	≤20	5.0	20
	6010(3005/3010)	2	75-125	≤20	5.0	20
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	1.5	5.0
Antimony (GFAA)	200.9	86	80-120	≤20	1.2	6.0
	204.2	3	80-120	≤20	1.2	10
	3113B	4	80-120	≤20	1.2	6.0
	7041(3020)	2	80-120	≤20	1.2	10
Arsenic (ICP)	200.7 (Drinking Water)	86	70-130	≤20	3.2	10
	200.7(NPDES)	3	75-125	≤20	3.2	10
	6010(3005/3010)	2	75-125	≤20	3.2	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	1.1	2.5
Arsenic (GFAA)	200.9	86	80-120	≤20	1.4	4.0
	206.2	3	80-120	≤20	2.0	10
	3113B	4	80-120	≤20	2.0	4.0
	3114B	4	80-120	≤20	0.40	2.0
	7060(3020)	2	80-120	≤20	1.5	10
	7062(3010)	2	80-120	≤20	0.40	2.0
Barium (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.2	10
	200.7(NPDES)	3	75-125	≤20	1.2	10
	6010(3005/3010)	2/3	75-125	≤20	1.2	10
	200.7/6010(3005/3010)-4X	2/3	75-125	≤20	0.094	2.5
Beryllium (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.54	4.0
	200.7(NPDES)	3	75-125	≤20	0.54	4.0
	6010(3005/3010)	2	75-125	≤20	0.54	4.0
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.014	1.0
Boron (ICP)	200.7(NPDES)	3	75-125	≤20	9.5	50
	6010(3005/3010)	2	75-125	≤20	9.5	50
Cadmium (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.71	5.0
	200.7(NPDES)	3	75-125	≤20	0.71	5.0
	6010(3005/3010)	2	75-125	≤20	0.71	5.0
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.091	1.2
Cadmium (GFAA)	200.9	86	80-120	≤20	0.16	1.0
	213.2	3	80-120	≤20	0.16	1.0
	3113B(3030E)	4	80-120	≤20	0.16	1.0
	7131(3020)	2	80-120	≤20	0.16	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug/L)	RLA (ug/L)
Calcium (ICP)	200.7(Drinking Water)	86	70-130	≤20	44	500
	200.7(NPDES)	3	75-125	≤20	44	500
	6010(3005/3010)	2	75-125	≤20	44	500
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	13	120
Chromium (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.7	10
	200.7(NPDES)	3	75-125	≤20	1.7	10
	6010(3005/3010)	2	75-125	≤20	1.7	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.25	2.5
Chromium (GFAA)	200.9	86	80-120	≤20	0.81	10
	218.2	3	80-120	≤20	0.81	10
	3113B(3030E)	4	80-120	≤20	0.81	10
	7191(3020)	2	80-120	≤20	0.81	10
Chromium, hexavalent	200.7(218.4)	3	70-130	≤20	5.0	5.0
Chromium, hexavalent	7195	2	85-115	≤20	1.4	10
(colorimetric)	7196/3500-Cr-D	2/4	85-115	≤20	1.4	10
Cobalt (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.4	10
	200.7(NPDES)	3	75-125	≤20	1.4	10
	6010(3005/3010)	2	75-125	≤20	1.4	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.12	2.5
Copper (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.90	20
	200.7(NPDES)	3	75-125	≤20	0.90	20
	6010(3005/3010)	2	75-125	≤20	0.90	20
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.53	5.0
Copper (GFAA)	220.2	3	80-120	≤20	0.67	10
	7211(3020)	2	80-120	≤20	0.67	10
	Chelation/Extraction	5	60-140	≤20	0.74	1.0
Iron (ICP)	200.7(Drinking Water)	86	70-130	≤20	18	50
	200.7(NPDES)	3	75-125	≤20	18	50
	6010(3005/3010)	2	75-125	≤20	18	50
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	3.2	12
Iron (ferrous)	3500-Fe-D (colorimetric)	4	80-120	≤20	9.4	100
Lead (ICP)	200.7(NPDES)	3	75-125	≤20	1.5	5.0
	6010(3005/3010	2	75-125	≤20	1.5	5.0
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.25	1.2
Lead (GFAA)	200.9	86	80-120	≤20	0.54	2.0
	239.2	3	80-120	≤20	0.54	5.0
	3113B(3030E)	4	80-120	≤20	0.54	2.0
	7421(3020)	2	80-120	≤20	0.54	5.0
	Chelation/Extraction	5	60-140	≤40	0.50	0.50

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug/L)	RLA (ug/L)
Lithium (ICP)	200.7(NPDES)	3	75-125	≤20	100	100
	6010(3005/3010)	2	75-125	≤20	100	100
Lithium (FLAA)	7430(3010)	2	80-120	≤20	42	100
Lithium (FLES)	SL SOP	100	80-120	≤20	0.26	2.0
Magnesium (ICP)	200.7(Drinking Water)	86	70-130	≤20	110	500
	200.7(NPDES)	3	75-125	≤20	110	500
	6010(3005/3010)	2	75-125	≤20	110	500
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	2.4	120
Manganese (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.4	10
	200.7(NPDES)	2/3	75-125	≤20	1.4	10
	6010(3005/3010)	2/3	75-125	≤20	1.4	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.082	2.5
Mercury (CVAA)	245.1	3	85-115	≤20	0.072	0.20
	7470	2	80-120	≤20	0.072	0.20
	3112B	4	80-120	≤20	0.072	0.20
Molybdenum (ICP)	200.7(Drinking Water)	86	70-130	≤20	2.6	10
	200.7(NPDES)	3	75-125	≤20	2.6	10
	6010(3005/3010)	2	75-125	≤20	2.6	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.44	2.5
Nickel (ICP)	200.7(Drinking Water)	86	70-130	≤20	4.7	40
	200.7(NPDES)	3	75-125	≤20	4.7	40
	6010(3005/3010)	2	75-125	≤20	4.7	40
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.56	10
Nickel (GFAA)	Chelation/Extraction	5	60-140	≤40	0.17	2.0
Phosphorus (ICP)	200.7(Drinking Water)	86	70-130	≤20	50	50
	6010(3010)	2	75-125	≤20	50	50
Potassium (ICP)	200.7(Drinking Water)	86	70-130	≤20	190	1000
	200.7(NPDES)	2/3	75-125	≤20	190	1000
	6010(3005/3010)	2/3	75-125	≤20	190	1000
	6010(3005/3010)-4X	86/3/2	75-125	≤20	5.7	250
Potassium (FLAA)	7610(3010)	2	80-120	≤20	100	100
	258.1	3	80-120	≤20	100	100

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug/L)	RLA (ug/L)
Selenium (ICP)	200.7(NPDES)	3	75-125	≤20	4.2	10
	6010(3005/3010)	2	75-125	≤20	4.2	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.94	2.5
Selenium (GFAA)	200.9	86	80-120	≤20	3.3	10
	270.2	3	80-120	≤20	3.3	10
	3113B(3030E)	4	80-120	≤20	3.3	10
	7740(3020)	2	80-120	≤20	3.3	10
	3114B	4	80-120	≤20	1.2	2.0
	7742(3020)	2	80-120	≤20	1.2	2.0
Silica, dissolved (ICP)	200.7(Drinking Water)	86	70-130	≤20	22	500
	200.7	3	75-125	≤20	22	500
	6010	2	75-125	≤20	22	500
Silver (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.9	10
	200.7(NPDES)	3	75-125	≤20	1.9	10
	6010(3005/3010)	2	75-125	≤20	1.9	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.27	2.5
Silver(FLAA)	272.1	3	80-120	≤20	10	10
Silver(GFAA)	272.2	3	80-120	≤20	0.17	1.0
	7761(3020)	2	80-120	≤20	0.17	1.0
	Chelation/Extraction	5	60-140	≤40	0.013	0.050
Sodium (ICP)	200.7(Drinking Water)	86	70-130	≤20	310	500
	200.7(NPDES)	3	75-125	≤20	310	500
	6010(3005/3010)	2	75-125	≤20	310	500
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	10	120
Sodium (FLAA)	273.1/7770(3010)	3/2	75-125	≤20	5000	5000
Strontium (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.46	10
	200.7(NPDES)	3	75-125	≤20	0.46	10
	6010(3005/3010)	2	75-125	≤20	0.46	10
Thallium (ICP)	200.7(NPDES)	3	75-125	≤20	4.9	10
	6010(3005/3010)	2	75-125	≤20	4.9	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	1.1	2.5
Thallium (GFAA)	200.9	86	80-120	≤20	1.2	2.0
	279.2	3	80-120	≤20	1.2	10
	7841(3020)	2	80-120	≤20	1.2	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug/L)	RLA (ug/L)
Tin (ICP)	200.7(Drinking Water)	86	70-130	≤20	6.4	50
	200.7(NPDES)	3	75-125	≤20	6.4	50
	6010(3005/3010)	2	75-125	≤20	6.4	50
	200.7/6010(3005/3010)-4X	2	75-125	≤20	1.2	12
Titanium (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.96	10
	200.7(NPDES)	3	75-125	≤20	0.96	10
	6010(3005/3010)	2	75-125	≤20	0.96	10
Tributyl tin (GFAA)	Atomic absorption	40	60-140	≤40	0.0010	0.0010
Vanadium (ICP)	200.7(Drinking Water)	86	70-130	≤20	2.2	10
	200.7(NPDES)	3	75-125	≤20	2.2	10
	6010(3005/3010)	2	75-125	≤20	2.2	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.15	2.5
Zinc (ICP)	200.7(Drinking Water)	86	70-130	≤20	5.9	20
	200.7(NPDES)	3	75-125	≤20	5.9	20
	6010(3005/3010)	2	75-125	≤20	5.9	20
	2000.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	2.6	5.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
FLAA = flame atomic adsorption spectrophotometer
FLES = flame emission spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Acetate	300.0	82	75-125	≤30	0.095	1.0
Acidity	305.1/2310B	3/4	80-120	≤30	0.40	10
	305.2	3	80-120	≤30	0.40	10
Alkalinity, total, as $CaCO_3$	310.1/2320B	3/4	80-120	≤30	0.40	1.0
Ammonia (as N)	350.1/4500-NH3-H	82/4	85-115	≤30	0.015	0.030
	350.3	82	85-115	≤30	0.011	0.050
Ammonia, un-ionized	FL-DER	60	NA	NA	NA	Calculated based on sample pH and temperature
Bicarbonate, as $CaCO_3$	4500-CO_2 D	4	NA	NA	NA	1.0
BOD-5	405.1/5210B	3/4	85-115	≤30	NA	2.0
Bromide	300.0/9056	82/2	90-110	≤30	0.068	1.0
	4110B	4	80-120	≤30	0.068	1.0
	320.1	3	75-125	≤30	0.57	2.0
Carbon, total organic	415.1/9060/5310-B	3/2/4	80-120	≤25	0.53	1.0
	5310C	4	80-120	≤25	0.54	1.0
Carbonate, as $CaCO_3$	4500-CO_2 D	4	NA	NA	NA	1.0
CBOD(1)	5210-B	4	81-119	≤30	NA	2.0
Chloride	300.0/9056	82/2	90-110	≤30	0.034	1.0
	4110B	4	80-120	≤30	0.034	1.0
	325.2/9251/4500-Cl-E	3/2/4	85-115	≤30	0.24	1.0
	325.3/9252A	3/2	75-125	≤30	0.40	1.0
	4500-Cl⁻ C	4	75-125	≤30	0.40	1.0
	4500-Cl⁻ D	4	80-120	≤30	0.40	1.0
Chlorine, residual	330.2	3	NA	≤30	NA	1.0
	330.3	3	NA	≤30	NA	1.0
	330.4	3	NA	≤30	NA	1.0
	330.5	3	NA	≤30	NA	1.0
	4500-Cl⁻ G	4	NA	≤40	NA	0.05
	4500-Cl⁻ B	4	NA	≤30	NA	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/L)	RLA (ug/L)
Alachlor	8081(3510/3520)	2	62-128	≤30	0.41	2.0
Aldrin (MS)	8081(3510/3520)	2	38-129	≤25	0.0099	0.050
Benfluralin	8081(3520)	2	40-140	≤40	0.0025	0.020
alpha BHC	8081(3510/3520)	2	46-131	≤30	0.0079	0.050
beta BHC	8081(3510/3520)	2	36-158	≤35	0.0074	0.050
delta BHC	8081(3510/3520)	2	53-137	≤41	0.012	0.050
gamma BHC (Lindane) (MS)	8081(3510/3520)	2	40-139	≤25	0.0074	0.050
Captafol	8081(3510/3520)	2	20-130	≤50	0.025	0.10
alpha Chlordane	8081(3510/3520)	2	55-125	≤17	0.0076	0.050
gamma Chlordane	8081(3510/3520)	2	54-128	≤18	0.0074	0.050
technical Chlordane	8081(3510/3520)	2	54-140	≤30	0.043	0.50
Chlorobenzilate	8081(3510/3520)	2	50-150	≤40	0.078	0.50
Chloroneb	8081(3510/3520)	2	49-125	≤30	0.091	0.40
Chloropropylate	8081(3510/3520)	2	51-125	≤30	0.17	0.50
Chlorothalonil	8081(3510/3520)	2	55-125	≤30	0.050	0.20
Dacthal (DCPA)	8081(3510/3520)	2	50-150	≤25	0.014	0.020
4,4'-DDD	8081(3510/3520)	2	32-155	≤39	0.018	0.10
4,4'-DDE	8081(3510/3520)	2	48-145	≤18	0.014	0.10
4,4'-DDT (MS)	8081(3510/3520)	2	50-147	≤27	0.017	0.10
Dicofol (Kelthane)	8081(3510/3520)	2	55-115	≤40	0.0067	0.050
Dieldrin (MS)	8081(3510/3520)	2	34-150	≤42	0.012	0.10
Endosulfan I	8081(3510/3520)	2	34-161	≤24	0.0094	0.050
Endosulfan II	8081(3510/3520)	2	40-162	≤22	0.018	0.10
Endosulfan sulfate	8081(3510/3520)	2	28-156	≤28	0.020	0.10
Endrin (MS)	8081(3510/3520)	2	41-158	≤25	0.014	0.10
Endrin aldehyde	8081(3510/3520)	2	20-146	≤34	0.021	0.10
Endrin ketone	8081(3510/3520)	2	42-122	≤25	0.020	0.10
Etradiazole	8081(3510/3520)	2	60-125	≤30	0.0018	0.020
Heptachlor (MS)	8081(3510/3520)	2	37-148	≤26	0.0062	0.050
Heptachlor epoxide	8081(3510/3520)	2	43-141	≤31	0.0069	0.050

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/L)	RLA (ug/L)
Hexachlorobenzene	8081(3510/3520)	2	25-112	≤33	0.0070	0.050
Isodrin	8081(3510/3520)	2	55-110	≤40	0.019	0.050
Kepone	8081(3510/3520)	2	10-150	≤50	0.038	1.0
Methoxychlor	8081(3510/3520)	2/26	60-155	≤43	0.038	0.50
Mirex	8081(3510/3520)	2	52-112	≤37	0.046	0.50
PCNB	8081(3510/3520)	2	60-125	≤30	0.0033	0.60
Pendimethalin	8081(3510/3520)	2	50-146	≤25	0.0021	0.050
Permethrin	8081(3510/3520)	2	50-130	≤40	0.12	1.0
Propachlor	8081(3510/3520)	2	51-125	≤30	0.084	0.50
Toxaphene	8081(3510/3520)	2	12-130	≤30	1.0	5.0
Trifluralin	8081(3510/3520)	2	54-124	≤40	0.0026	0.020
PCB 1016(MS)	8082(3510/3520)	2	45-134	≤34	0.21	1.0
PCB 1221	8082(3510/3520)	2	20-173	≤110	0.36	2.0
PCB 1232	8082(3510/3520)	2	10-228	≤86	0.095	1.0
PCB 1242	8082(3510/3520)	2	37-160	≤74	0.20	1.0
PCB 1248	8082(3510/3520)	2	50-113	≤30	0.13	1.0
PCB 1254	8082(3510/3520)	2	50-122	≤39	0.22	1.0
PCB 1260(MS)	8082(3510/3520)	2	41-144	≤34	0.11	1.0
PCB 1268	8082(3510/3520)	2	40-140	≤40	0.11	1.0
Surrogate – Dibutylchlorendate (DBC)	8081/8082(3510/3520)	2	30-150	NA	NA	NA
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081/8082 (3510/3520)	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	8081/8082 (3510/3520)	2	30-150	NA	NA	NA

NOTE: If only PCBs are requested, PCB1016 and PCB1260 are the matrix spiking compounds.

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Acetone	8260(5030)	2	32-174	≤52	9.9	50
	8260(5030)(low level)	2	32-174	≤52	12	25
Acetonitrile	8260(5030)	2	70-130	≤30	75	200
	8260(5030)(low level)	2	70-130	≤30	14	40
Acrolein	8260(5030)	2	54-145	≤74	43	100
	8260(5030) (low level)	2	54-145	≤74	9.2	20
Acrylonitrile	8260(5030)	2	10-183	≤41	9.7	100
	8260(5030) (low level)	2	10-183	≤1	5.0	20
Benzene (MS)	8260(5030)	2	62-135	≤37	0.27	5.0
	8260(5030)(low level)	2	62-135	≤37	0.10	1.0
Benzyl chloride	8260(5030)	2	70-130	≤30	25	100
	8260(5030)(low level)	2	70-130	≤30	12	50
Bromobenzene	8260(5030)	2	62-124	≤35	0.58	5.0
	8260(5030)(low level)	2	62-124	≤35	0.11	1.0
Bromochloromethane	8260(5030)	2	45-131	≤33	0.58	5.0
	8260(5030)(low level)	2	45-131	≤33	0.21	1.0
Bromodichloromethane	8260(5030)	2	65-125	≤28	0.35	5.0
	8260(5030)(low level)	2	65-125	≤28	0.14	1.0
Bromoform	8260(5030)	2	52-148	≤31	0.58	5.0
	8260(5030)(low level)	2	52-148	≤31	0.24	1.0
Bromomethane	8260(5030)	2	40-141	≤33	2.5	10
	8260(5030)(low level)	2	40-141	≤32	0.44	1.0
1,3-Butadiene	8260(5030)	2	70-130	≤30	1.0	10
2-Butanone (MEK)	8260(5030)	2	42-167	≤31	11	25
	8260(5030)(low level)	2	42-167	≤31	5.5	10
n-Butylbenzene	8260(5030)	2	51-117	≤33	0.67	5.0
	8260(5030)(low level)	2	51-117	≤33	0.14	1.0
sec-Butylbenzene	8260(5030)	2	41-150	≤34	0.63	5.0
	8260(5030)(low level)	2	41-150	≤34	0.11	1.0
tert-Butylbenzene	8260(5030)	2	42-141	≤34	0.84	5.0
	8260(5030)(low level)	2	42-141	≤34	0.070	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Carbon disulfide	8260(5030)	2	28-152	≤23	1.5	5.0
	8260(5030)(low level)	2	28-152	≤23	0.35	1.0
Carbon tetrachloride	8260(5030)	2	57-128	≤38	0.42	5.0
	8260(5030)(low level)	2	57-128	≤38	0.090	1.0
Chlorobenzene (MS)	8260(5030)	2	72-127	≤22	0.63	5.0
	8260(5030)(low level)	2	72-127	≤22	0.090	1.0
2-Chloro-1,3-butadiene	8260(5030)	2	70-130	≤30	0.89	5.0
(Chloroprene)	8260(5030)(low level)	2	70-130	≤30	0.17	1.0
Chloroethane	8260(5030)	2	47-148	≤34	1.6	10
	8260(5030)(low level)	2	47-148	≤34	0.22	1.0
2-Chloroethyl vinyl ether	8260(5030)	2	10-177	≤142	11	50
	8260(5030)(low level)	2	10-177	≤142	0.97	10
Chloroform	8260(5030)	2	62-130	≤20	0.90	5.0
	8260(5030)(low level)	2	62-130	≤20	0.14	1.0
Chloromethane	8260(5030)	2	34-145	≤44	2.1	10
	8260(5030)(low level)	2	34-145	≤44	0.30	1.0
3-Chloropropene (Allyl chloride)	8260(5030)	2	70-130	≤30	1.1	5.0
	8260(5030)(low level)	2	70-130	≤30	0.12	1.0
2-Chlorotoluene	8260(5030)	2	47-142	≤39	0.65	5.0
	8260(5030)(low level)	2	47-142	≤39	0.11	1.0
4-Chlorotoluene	8260(5030)	2	49-140	≤25	0.52	5.0
	8260(5030)(low level)	2	49-140	≤25	0.14	1.0
Cyclohexanone	8260(5030)(low level)	2	70-130	≤30	59	500
Dibromochloromethane	8260(5030)	2	68-126	≤31	0.51	5.0
	8260(5030)(low level)	2	68-126	≤31	0.17	1.0
1,2-Dibromo-3-chloropropane	8260(5030)	2	70-130	≤30	0.74	5.0
(DBCP)	8260(5030)(low level)	2	70-130	≤30	0.38	1.0
1,2-Dibromoethane	8260(5030)	2	45-137	≤40	0.50	5.0
	8260(5030)(low level)	2	45-137	≤40	0.13	1.0
Dibromomethane	8260(5030)	2	62-130	≤40	0.41	5.0
	8260(5030)(low level)	2	62-130	≤40	0.070	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
1,2-Dichlorobenzene	8260(5030)	2	57-129	≤39	0.44	5.0
	8260(5030)(low level)	2	57-129	≤39	0.080	1.0
1,3-Dichlorobenzene	8260(5030)	2	52-140	≤29	0.64	5.0
	8260(5030)(low level)	2	52-140	≤29	0.11	1.0
1,4-Dichlorobenzene	8260(5030)	2	51-138	≤31	0.52	5.0
	8260(5030)(low level)	2	51-138	≤31	0.14	1.0
trans-1,4-Dichloro-2-butene	8240(5030)/8260(5030)	2	70-130	≤30	2.5	10
	8260(5030)(low level)	2	70-130	≤30	1.2	2.0
Dichlorodifluoromethane	8260(5030)	2	30-126	≤34	1.2	10
	8260(5030)(low level)	2	30-126	≤34	0.20	1.0
1,1-Dichloroethane	8260(5030)	2	51-140	≤47	0.52	5.0
	8260(5030)(low level)	2	51-140	≤47	0.38	1.0
1,2-Dichloroethane	8260(5030)	2	65-131	≤23	0.57	5.0
	8260(5030)(low level)	2	65-131	≤23	0.46	1.0
Dichloroethenes (Total)	8260(5030)	2	43-136	≤22	0.44	5.0
(sum of cis- and trans- isomers)	8260(5030)(low level)	2	43-136	≤22	0.19	1.0
cis-1,2-Dichloroethene	8240(5030)/8260	2	50-128	≤28	0.65	5.0
	8240(5030)/8260(5030)(low level)	2	50-128	≤28	0.51	1.0
trans-1,2-Dichloroethene	8260	2	43-136	≤22	0.44	5.0
	8260(5030)(low level)	2	43-136	≤22	0.19	1.0
1,1-Dichloroethene	8260(5030)	2	46-147	≤30	0.45	5.0
	8260(5030)(low level)	2	46-147	≤30	0.19	1.0
1,2-Dichloropropane	8260(5030)	2	67-128	≤24	0.52	5.0
	8260(5030)(low level)	2	67-128	≤24	0.090	1.0
1,3-Dichloropropane	8260(5030)	2	49-139	≤40	0.39	5.0
	8260(5030)(low level)	2	49-139	≤40	0.14	1.0
2,2-Dichloropropane	8260(5030)	2	46-119	≤21	1.1	5.0
	8260(5030)(low level)	2	46-119	≤21	0.40	1.0
1,1-Dichloropropene	8260(5030)	2	54-114	0-37	0.31	5.0
	8260(5030)(low level)	2	54-114	0-37	0.12	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
cis-1,3-Dichloropropene	8260(5030)	2	66-125	≤21	0.47	5.0
	8260(5030)(low level)	2	66-125	≤21	0.17	1.0
trans-1,3-Dichloropropene	8260(5030)	2	49-136	≤24	0.38	5.0
	8260(5030)(low level)	2	49-136	≤24	0.49	1.0
Diethyl ether	8260(5030)	2	70-130	≤30	2.5	10
	8260(5030)(low level)	2	70-130	≤30	0.50	2.0
Ethanol	8260(5030)(low level)	2	70-130	≤30	250	1000
Ethyl acetate	8260(5030)	2	70-130	≤30	2.5	10
Ethylbenzene	8260(5030)	2	74-122	≤18	0.83	5.0
	8260(5030)(low level)	2	74-122	≤18	0.11	1.0
Ethyl methacrylate	8260(5030)	2	70-130	≤30	0.53	5.0
	8260(5030)(low level)	2	70-130	≤30	0.42	1.0
Ethylene oxide	8240(5030)/8260(5030)(low level)	2	70-130	≤30	99	120
Hexachlorobutadiene	8260(5030)	2	55-127	≤29	2.3	5.0
	8260(5030)(low level)	2	55-127	≤29	0.14	1.0
Hexane	8260(5030)	2	70-130	≤30	1.2	5.0
2-Hexanone	8260(5030)	2	48-155	≤36	4.4	25
	8260(5030)(low level)	2	48-155	≤36	1.5	10
Iodomethane	8260(5030)	2	70-130	≤30	0.67	5.0
	8260(5030)(low level)	2	70-130	≤30	0.37	1.0
Isobutyl alcohol	8260(5030)	2	70-130	≤30	31	200
	8260(5030)(low level)	2	70-130	≤30	11	40
Isopropylbenzene	8260(5030)	2	45-136	≤36	0.95	5.0
	8260(5030)(low level)	2	45-136	≤36	0.10	1.0
p-Isopropyltoluene	8260(5030)	2	52-132	≤34	0.69	5.0
	8260(5030)(low level)	2	52-132	≤34	0.13	1.0
Methacrylonitrile	8260(5030)	2	70-130	≤30	8.4	100
	8260(5030)(low level)	2	70-130	≤30	8.1	20
Methylene chloride	8260(5030)	2	47-140	≤50	0.31	5.0
(Dichloromethane)	8260(5030)(low level)	2	47-140	≤50	0.25	5.0
Methylmethacrylate	8240(5030)	2	70-130	≤30	0.66	5.0
	8240(5030)(low level)	2	70-130	≤30	0.55	1.0
4-Methyl-2-pentanone (MIBK)	8260(5030)	2	50-150	≤42	8.6	25
	8260(5030)(low level)	2	50-150	≤42	0.59	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Methyl t-butyl ether (MTBE)	8260(5030)	2	56-124	≤38	1.5	10
	8260(5030)(low level)	2	56-124	≤38	0.85	10
Naphthalene	8260(5030)	2	40-139	≤50	2.3	5.0
	8260(5030)(low level)	2	40-139	≤50	0.21	1.0
n-Octane	8260	2	70-130	≤30	1.2	5.0
Pentachloroethane	8260(5030)	2	70-130	≤30	11	25
	8260(5030)(low level)	2	70-130	≤30	4.2	5.0
Propionitrile (ethylcyanide)	8260(5030)	2	70-130	≤30	7.2	100
	8260(5030)(low level)	2	70-130	≤30	13	20
n-Propylbenzene	8260(5030)	2	56-122	≤34	0.59	5.0
	8260(5030)(low level)	2	56-122	≤34	0.19	1.0
Styrene	8260(5030)	2	66-130	≤28	0.98	5.0
	8260(5030)(low level)	2	66-130	≤28	0.16	1.0
1,1,1,2-Tetrachloroethane	8260(5030)	2	59-137	≤20	0.63	5.0
	8260(5030)(low level)	2	59-137	≤20	0.11	1.0
1,1,2,2-Tetrachloroethane	8260(5030)	2	67-133	≤22	0.75	5.0
	8260(5030)(low level)	2	67-133	≤22	0.13	1.0
Tetrachloroethene	8260(5030)	2	60-148	≤24	1.6	5.0
	8260(5030)(low level)	2	60-148	≤24	0.38	1.0
Toluene (MS)	8260(5030)	2	68-131	≤33	0.51	5.0
	8260(5030)(low level)	2	68-131	≤33	0.28	1.0
1,2,3-Trichlorobenzene	8260(5030)	2	30-140	≤65	0.77	5.0
	8260(5030)(low level)	2	30-140	≤65	0.17	1.0
1,2,4-Trichlorobenzene	8260(5030)	2	40-147	≤57	0.58	5.0
	8260(5030)(low level)	2	40-147	≤57	0.16	1.0
1,1,1-Trichloroethane	8260(5030)	2	69-120	≤27	0.46	5.0
	8260(5030)(low level)	2	69-120	≤27	0.14	1.0
1,1,2-Trichloroethane	8260(5030)	2	63-133	≤21	0.47	5.0
	8260(5030)(low level)	2	63-133	≤21	0.15	1.0
Trichloroethene(MS)	8260(5030)	2	56-143	≤35	0.28	5.0
	8260(5030)(low level)	2	56-143	≤35	0.17	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL A (ug/L)
Trichlorofluoromethane	8260(5030)	2	53-136	≤21	0.98	5.0
	8260(5030)(low level)	2	53-136	≤21	0.49	1.0
1,2,3-Trichloropropane	8260(5030)	2	67-122	≤26	1.3	5.0
	8260(5030)(low level)	2	67-122	≤26	0.49	1.0
1,1,2-Trichloro-1,2,2-Trifluoroethane	8260(5030)	2	70-130	≤30	0.26	5.0
	8260(5030)(low level)	2	70-130	≤30	0.57	1.0
1,2,4-Trimethylbenzene	8260(5030)	2	61-133	≤20	0.86	5.0
	8260(5030)(low level)	2	61-133	≤20	0.090	1.0
1,3,5-Trimethylbenzene	8260(5030)	2	47-138	≤48	1.1	5.0
	8260(5030)(low level)	2	47-138	≤48	0.15	1.0
Vinyl acetate	8260(5030)	2	10-166	≤22	1.5	10
	8260(5030)(low level)	2	10-166	≤22	0.15	2.0
Vinyl chloride	8260(5030)	2	43-142	≤21	0.50	10
	8260(5030)(low level)	2	43-142	≤21	0.28	2.0
Xylenes (total)	8260(5030)	2	73-135	≤26	1.9	10
	8260(5030) (low level)	2	73-135	≤26	0.31	2.0
o-Xylene	8260(5030)	2	72-129	≤26	0.78	5.0
	8260(5030)(low level)	2	72-129	≤26	0.12	1.0
m+p-Xylene	8260(5030)	2	73-135	≤26	1.9	5.0
	8260(5030)(low level)	2	73-135	≤26	0.31	1.0
Toluene-d8 (Surrogate)	8260(5030)	2	77-122	NA	NA	NA
	8260(5030)(low level)	2	77-122	NA	NA	NA
p-Bromofluorobenzene (Surrogate)	8260(5030)	2	74-126	NA	NA	NA
	8260(5030)(low level)	2	74-126	NA	NA	NA
Dibromofluoromethane (Surrogate)	8260(5030)	2	70-130	NA	NA	NA
	8260(5030)(low level)	2	70-130	NA	NA	NA
1,2-Dichloroethane-d4 (Surrogate)	8240(5030)	2	70-130	NA	NA	NA
	8240(5030)(low level)	2	70-130	NA	NA	NA
1,2-Dichlorobenzene-d4 (Surrogate)	8260(5030)	2	70-130	NA	NA	NA
	8260(5030)(low level)	2	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Acenaphthene (MS)	8270(3510/3520)	2	36-121	≤35	0.25	10
	8270(3510/3520)-low level	2	36-121	≤35	0.088	0.20
Acenaphthylene	8270(3510/3520)	2	41-121	≤28	0.33	10
	8270(3510/3520)-low level	2	41-121	≤28	0.074	0.20
Acetophenone	8270(3510/3520)	2	10-150	≤50	0.52	10
2-Acetylaminofluorene	8270(3510/3520)	2	25-150	≤50	0.50	10
Aldrin	8270(3510/3520)	2	62-119	≤21	0.34	10
4-Aminobiphenyl	8270(3510/3520)	2	10-150	≤50	4.8	10
Aniline	8270(3510/3520)	2	10-180	≤32	4.2	20
Anthracene	8270(3510/3520)	2	45-126	≤21	0.33	10
	8270(3510/3520)-low level	2	45-126	≤21	0.076	0.20
Aramite	8270(3510/3520)	2	40-150	≤50	1.1	10
Benzidine	8270(3510/3520)	2	10-133	≤121	5.6	80
Benzoic acid	8270(3510/3520)	2	10-150	≤66	9.9	50
Benzo(a)anthracene	8270(3510/3520)	2	33-136	≤34	0.30	10
	8270(3510/3520)-low level	2	33-136	≤34	0.060	0.20
	SIM	73	64-100	≤25	0.042	0.10
Benzo(b)fluoranthene	8270(3510/3520)	2	33-132	≤32	0.28	10
	8270(3510/3520)-low level	2	33-132	≤32	0.057	0.20
	SIM	73	60-103	≤25	0.036	0.10
Benzo(k)fluoranthene	8270(3510/3520)	2	33-150	≤34	0.72	10
	8270(3510/3520)-low level	2	33-150	≤34	0.057	0.20
	SIM	73	63-101	≤25	0.038	0.10
p-Benzoquinone	8270(3510/3520)	2	40-140	≤40	3.7	10
Benzo(g,h,i)perylene	8270(3510/3520)	2	28-146	≤39	0.68	10
	8270(3510/3520)-low level	2	28-146	≤39	0.056	0.20
Benzo(a)pyrene	8270(3510/3520)	2	45-120	≤24	0.41	10
	8270(3510/3520)-low level	2	45-120	≤24	0.057	0.20
	SIM	73	48-100	≤25	0.036	0.10
Benzyl alcohol	8270(3510/3520)	2	13-134	≤32	0.49	10
Benzyl chloride	8270(3510/3520)	2	10-150	≤50	2.5	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Alpha-BHC	8270(3510/3520)	2	44-131	≤30	0.37	10
Beta-BHC	8270(3510/3520)	2	29-127	≤33	0.80	10
Delta-BHC	8270(3510/3520)	2	10-161	≤44	1.7	10
Gamma-BHC	8270(3510/3520)	2	43-120	≤27	0.30	10
Biphenyl (Diphenyl)	8270(3510/3520)	2	50-130	≤25	2.2	10
Bis(2-chloroethoxy) methane	8270(3510/3520)	2	47-110	≤20	0.26	10
Bis(2-chloroethyl) ether	8270(3510/3520)	2	34-114	≤58	0.44	10
Bis(2-chloroisopropyl) ether (2,2-Oxybis(1-chloropropane)	8270(3510/3520)	2	15-144	≤23	0.23	10
Bis(2-ethylhexyl) phthalate	8270(3510/3520)	2	34-160	≤26	0.48	10
4-Bromophenyl phenyl ether	8270(3510/3520)	2	36-124	≤26	0.35	10
Butyl benzyl phthalate	8270(3510/3520)	2	38-141	≤41	0.41	10
Carbazole	8270(3510/3520)	2	48-127	≤23	0.54	10
4-Chloroaniline	8270(3510/3520)	2	10-130	≤67	0.98	20
4-Chloro-3-methyl-phenol (p-Chloro-m-cresol)(MS)	8270(3510/3520)	2	34-126	≤31	0.33	10
1-Chloronaphthalene	8270(3510/3520)	2	45-107	≤22	2.5	10
2-Chloronaphthalene	8270(3510/3520)	2	45-107	≤22	0.39	10
2-Chlorophenol (MS)	8270(3510/3520)	2	38-115	≤34	0.24	10
4-Chlorophenylphenyl ether	8270(3510/3520)	2	22-140	≤26	0.66	10
Chrysene	8270(3510/3520)	2	44-128	≤31	0.44	10
	8270(3510/3520)-low level	2	44-128	≤31	0.069	0.20
	SIM	73	72-109	≤25	0.044	0.10
1,8-Cineole	8270(3510/3520)	2	50-101	≤25	0.80	10
p-Cymene	8270(3510/3520)	2	52-110	≤25	1.6	10
o-Cresol (2-Methyl phenol)	8270(3510/3520)	2	31-119	≤33	0.29	10
m- Cresol (3 -Methyl phenol)	8270(3510/3520)	2	24-136	≤27	0.71	10
p-Cresol (4-Methyl phenol)	8270(3510/3520)	2	24-136	≤27	0.71	10
m- and p-Cresols(3 and 4-Methyl phenol)	8270(3510/3520)	2	24-136	≤27	0.71	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
4,4'-DDD	8270(3510/3520)	2	16-124	≤50	0.35	10
4,4'-DDE	8270(3510/3520)	2	30-125	≤35	0.30	10
4,4'-DDT	8270(3510/3520)	2	22-119	≤26	0.40	10
Diallate	8270(3510/3520)	2	10-150	≤50	1.2	10
Dibenz(a,b)anthracene	8270(3510/3520)	2	33-143	≤35	0.80	10
	8270(3510/3520)-low level	2	33-143	≤35	0.054	0.20
	SIM	73	47-117	≤25	0.042	0.10
Dibenzofuran	8270(3510/3520)	2	50-119	≤20	0.29	10
Dibenzo(a,e)pyrene	8270(3510/3520)	2	29-177	≤60	2.5	10
Di-n-butyl phthalate	8270(3510/3520)	2	43-145	≤29	0.26	10
1,2-Dichlorobenzene	8270(3510/3520)	2	34-130	≤30	0.31	10
1,3-Dichlorobenzene	8270(3510/3520)	2	28-130	≤26	0.32	10
1,4-Dichlorobenzene(MS)	8270(3510/3520)	2	27-130	≤31	0.29	10
3,3'-Dichlorobenzidine	8270(3510/3520)	2	10-144	≤72	4.4	20
2,4-Dichlorophenol	8270(3510/3520)	2	25-134	≤30	0.66	10
2,6-Dichlorophenol	8270(3510/3520)	2	10-150	≤50	1.0	10
Dieldrin	8270(3510/3520)	2	29-133	≤20	0.41	10
Di(2-ethylhexyl)adipate	8270(3520)	2	35-130	≤50	0.36	10
Diethyl phthalate	8270(3510/3520)	2	46-134	≤49	0.47	10
Dimethoate	8270(3510/3520)	2	10-150	≤50	2.5	10
p-(Dimethylamino)azobenzene	8270(3510/3520)	2	10-150	≤50	1.3	10
7,12-Dimethylbenz(a)anthracene	8270(3510/3520)	2	10-150	≤50	2.7	10
3,3'-Dimethylbenzidine	8270(3510/3520)	2	10-200	≤100	13	20
a,a-Dimethylphenethylamine	8270(3510/3520)	2	10-200	≤50	9.7	2000
2,4-Dimethylphenol	8270(3510/3520)	2	28-130	≤43	0.39	10
Dimethylphthalate	8270(3510/3520)	2	22-141	≤31	0.39	10
m-Dinitrobenzene	8270(3510/3520)	2	10-150	≤50	0.91	10
p-Dinitrobenzene	8270(3510/3520)	2	44-125	≤58	8.0	50
4,6-Dinitro-2-methylphenol	8270(3510/3520)	2	10-173	≤33	5.0	50

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
2,4-Dinitrophenol	8270(3510/3520)	2	10-209	≤63	0.80	50
2,4-Dinitrotoluene (MS)	8270(3510/3520)	2	37-129	≤32	0.41	10
2,6-Dinitrotoluene	8270(3510/3520)	2	24-139	≤24	0.34	10
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270(3510/3520)	2	10-150	≤50	1.1	10
Di-n-octyl phthalate	8270(3510/3520)	2	24-152	≤33	0.35	10
Diphenylamine	8270(3510/3520)	2	15-135	≤50	5.4	10
1,4-Dioxane	8270(3510/3520)	2	10-150	≤50	1.0	10
1,2-Diphenyl hydrazine	8270(3510/3520)	2	25-130	≤32	0.81	10
Disulfoton	8270(3510/3520)	2	10-150	≤50	2.5	10
Endosulfan I	8270(3510/3520)	2	15-135	≤68	1.8	20
Endosulfan II	8270(3510/3520)	2	22-118	≤60	1.3	20
Endosulfan sulfate	8270(3510/3520)	2	37-118	≤34	0.75	20
Endrin	8270(3510/3520)	2	21-125	≤50	2.3	20
Endrin aldehyde	8270(3510/3520)	2	10-234	≤59	18	50
Endrin ketone	8270(3510/3520)	2	10-150	≤50	12	50
Ethyl carbamate	8270(3510/3520)	2	52-100	≤25	2.5	10
Ethyl methanesulfonate	8270(3510/3520)	2	10-150	≤50	0.70	10
Ethyl parathion	8270(3510/3520)	2	10-150	≤50	2.5	10
Famphur	8270(3510/3520)	2	10-150	≤50	2.5	10
Fluoranthene	8270(3510/3520)	2	41-129	≤24	0.33	10
	8270(3510/3520)-low level	2	41-129	≤24	0.054	0.20
Fluorene	8270(3510/3520)	2	50-124	≤23	0.38	10
	8270(3510/3520)-low level	2	50-124	≤23	0.087	0.20
Heptachlor	8270(3510/3520)	2	17-130	≤27	0.42	20
Heptachlor epoxide	8270(3510/3520)	2	34-130	≤29	0.58	20
Hexachlorobenzene	8270(3510/3520)	2	49-121	≤31	0.19	10
Hexachlorobutadiene	8270(3510/3520)	2	27-130	≤30	0.35	10
Hexachlorocyclopentadiene	8270(3510/3520)	2	D-130	≤67	2.4	10
Hexachloroethane	8270(3510/3520)	2	26-130	≤35	0.32	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Hexachlorophene(1)	8270(3510/3520)	2	10-200	≤80	29	5000
Hexachloropropene	8270(3510/3520)	2	10-150	≤50	0.53	10
Indeno(1,2,3-cd)pyrene	8270(3510/3520)	2	37-140	≤38	0.56	10
	8270(3510/3520)-low level	2	37-140	≤38	0.064	0.20
	SIM	73	38-100	≤35	0.043	0.10
Isophorone	8270(3510/3520)	2	29-130	≤33	0.37	10
Isosafrole	8270(3510/3520)	2	10-150	≤50	0.64	10
Kepone	8270(3510/3520)	2	10-150	≤50	5.3	10
d-Limonene	8270(3510/3520)	2	36-74	≤25	1.4	10
Methapyrilene	8270(3510/3520)	2	10-150	≤50	1.6	2000
4,4-Methylbis(2-chloroaniline)	8270(3510/3520)	2	39-121	≤73	92	500
3-Methylcholanthrene	8270(3510/3520)	2	10-150	≤50	0.76	10
Methylmethanesulfonate	8270(3510/3520)	2	10-150	≤50	0.72	10
2-Methylnaphthalene	8270(3510/3520)	2	43-130	≤30	0.33	10
	8270(3510/3520)-low level	2	43-130	≤30	0.054	0.20
1-Methylnaphthalene	8270(3510/3520)	2	35-130	≤30	0.68	10
	8270(3510/3520)-low level	2	35-130	≤30	0.073	0.20
Methyl parathion	8270(3510/3520)	2	10-150	≤50	2.5	10
2,4(8)-p-Menthadiene	8270(3510/3520)	2	22-85	≤42	1.6	10
Naphthalene	8270(3510/3520)	2	41-130	≤33	0.36	10
	8270(3510/3520)-low level	2	41-130	≤33	0.071	0.20
1,4-Naphthoquinone	8270(3510/3520)	2	10-150	≤50	0.78	10
1-Naphthylamine	8270(3510/3520)	2	10-150	≤50	1.1	10
2-Naphthylamine	8270(3510/3520)	2	10-150	≤50	0.98	10
Nicotine	8270(3510/3520)	2	10-150	≤50	25	100
2-Nitroaniline	8270(3510/3520)	2	26-130	≤49	5.3	50
3-Nitroaniline	8270(3510/3520)	2	10-130	≤57	5.0	50
4-Nitroaniline	8270(3510/3520)	2	10-140	≤39	7.7	50
Nitrobenzene	8270(3510/3520)	2	50-111	≤21	0.31	10
2-Nitrophenol	8270(3510/3520)	2	35-125	≤24	0.36	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
4-Nitrophenol (MS)	8270(3510/3520)	2	12-143	≤44	4.9	50
4-Nitroquinoline-1-oxide	8270(3510/3520)	2	10-150	≤50	2.5	20
N-Nitrosodi-n-butylamine	8270(3510/3520)	2	10-150	≤50	0.62	10
N-Nitrosodiethylamine	8270(3510/3520)	2	10-150	≤50	0.46	10
N-Nitrosodimethylamine	8270(3510/3520)	2	10-133	≤31	0.49	10
N-Nitrosodi-n-propylamine(MS)	8270(3510/3520)	2	31-138	≤30	0.29	10
N-nitrosodiphenylamine/ Diphenylamine	8270(3510/3520)	2	24-146	≤25	0.34	10
N-Nitrosodiphenylamine	8270(3510/3520)	2	24-146	≤25	0.34	10
N-Nitrosomethylethylamine	8270(3510/3520)	2	10-150	≤50	8.6	10
N-Nitrosomorpholine	8270(3510/3520)	2	10-150	≤50	1.1	10
N-Nitrosopiperidine	8270(3510/3520)	2	10-150	≤50	1.6	10
N-Nitrosopyrrolidine	8270(3510/3520)	2	10-150	≤50	0.86	10
5-Nitro-o-toluidine	8270(3510/3520)	2	10-150	≤50	1.3	10
Pentachlorobenzene	8270(3510/3520)	2	10-150	≤50	0.68	10
Pentachloronitrobenzene	8270(3520)◊	2	10-150	≤50	1.3	10
Pentachlorophenol (MS)	8270(3510/3520)	2	19-148	≤33	4.0	50
	SIM	73	10-102	≤50	0.60	1.0
beta-Phellandrene	8270(3510/3520)	2	33-65	≤25	1.9	10
Phenacetin	8270(3510/3520)	2	10-150	≤50	1.2	10
Phenanthrene	8270(3510/3520)	2	50-121	≤20	0.33	10
	8270(3510/3520)-low level	2	50-121	≤20	0.089	0.20
Phenol (MS)	8270(3510/3520)	2	33-122	≤36	0.28	10
Phenyl ether (Diphenyl oxide)	8270(3510/3520)	2	74-116	≤25	2.0	10
p-Phenylenediamine	8270(3510/3520)	2	10-200	≤50	500	2000
Phorate	8270(3510/3520)	2	10-150	≤50	2.5	10
2-Picoline	8270(3510/3520)	2	10-150	≤50	1.1	10
cis-Pinane	8270(3510/3520)	2	50-103	≤29	2.9	10
alpha-Pinene	8270(3510/3520)	2	12-105	≤25	1.3	100
beta-Pinene	8270(3510/3520)	2	DL-155	≤56	2.0	100

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Pronamide	8270(3510/3520)	2	10-150	≤50	0.56	10
Pyrene(MS)	8270(3510/3520)	2	31-139	≤42	0.53	10
	8270(3510/3520)-low level	2	31-139	≤42	0.089	0.20
Pyridine	8270(3510/3520)	2	D-134	≤50	0.93	50
Resorcinol	8270 (3510/3520)	2	6-110	≤54	2.2	200
Safrole	8270(3510/3520)	2	10-150	≤50	0.95	10
Strychnine	8270(3510/3520)	2	10-150	≤50	25	100
Sulfotepp	8270(3510/3520)	2	10-150	≤50	2.5	10
alpha-Terpinene	8270(3510/3520)	2	DL-94	≤96	1.2	10
Terpineol	8270(3510/3520)	2	50-150	≤50	0.82	10
Terpinolene	8270(3510/3520)	2	20-150	≤45	1.7	10
1,2,4,5-Tetrachlorobenzene	8270(3510/3520)	2	10-150	≤50	0.75	10
2,3,4,5-Tetrachlorophenol	8270(3510/3520)	2	10-150	≤50	0.61	10
2,3,4,6-Tetrachlorophenol	8270(3510/3520)	2	34-130	≤31	0.57	10
Tetrachlorophenols (2,3,4,5 and 2,3,4,6)	8270(3510/3520)	2	10-150	≤50	0.61	10
Thionazin	8270(3510/3520)	2	10-150	≤50	2.5	10
o-Toluidine	8270(3510/3520)	2	10-150	≤50	0.53	10
1,2,4-Trichlorobenzene (MS)	8270(3510/3520)	2	28-110	≤28	0.36	10
2,4,5-Trichlorophenol	8270(3510/3520)	2	38-127	≤28	0.74	10
2,4,6-Trichlorophenol	8270(3510/3520)	2	36-126	≤22	0.35	10
Trichlorophenols (2,4,5 and 2,4,6)	8270(3510/3520)	2	38-127	≤28	0.74	10
o,o,o-Triethyl-phosphorothioate	8270(3510/3520)	2	10-150	≤50	1.6	10
1,3,5-Trinitrobenzene	8270(3510/3520)	2	10-150	≤50	1.5	10
Nitrobenzene-d5 (Surrogate)	8270(3510/3520)	2	34-130	NA	NA	NA
2-Fluorobiphenyl (Surrogate)	8270(3510/3520)	2	36-124	NA	NA	NA
Terphenyl-d14 (Surrogate)	8270(3510/3520)	2	14-148	NA	NA	NA
Phenol-d5 (Surrogate)	8270(3510/3520)	2	25-128	NA	NA	NA
2-Fluorophenol (Surrogate)	8270(3510/3520)	2	29-121	NA	NA	NA
2,4,6-Tribromophenol (Surrogate)	8270(3510/3520)	2	29-143	NA	NA	NA
Orhto Terphenyl (Surrogate)	8270(3520)-low level	2	30-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (ug/L)	RLA (ug/L)
Polychlorinated Dibenzo-p-dioxin 2,3,7,8- substituted Congeners						
2,3,7,8-TCDD	8280	2	47-155	≤40	0.00070	0.0050
Polychlorinated Dibenzo-p-dioxin and Dibenzofuran classes						
tetra-CDD (MS)	8280	2	47-155	≤40	0.00070	0.0050
tetra-CDF (MS)	8280	2	67-154	≤40	0.00060	0.0050
penta-CDD (MS)	8280	2	50-168	≤40	0.0012	0.0050
Penta-CDF (MS)	8280	2	71-158	≤40	0.0011	0.0050
hexa-CDD (MS)	8280	2	72-164	≤40	0.0012	0.0050
hexa-CDF (MS)	8280	2	72-175	≤40	0.0013	0.0050
hepta-CDD (MS)	8280	2	20-170	≤50	0.0016	0.010
hepta-CDF (MS)	8280	2	20-170	≤50	0.00060	0.010
octa-CDD (MS)	8280	2	20-170	≤50	0.0057	0.010
octa-CDF (MS)	8280	2	20-170	≤50	0.0020	0.010
Internal Standards						
2,3,7,8-tetra-CDD-13C12	8280	2	25-150	NA	NA	NA
octa-CDD-13C12	8280	2	25-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Dissolved Gases In Water (GC/FID)						
Methane	SL SOP	95	75-125	≤30	0.040	0.19
Ethane	SL SOP	95	75-125	≤30	0.050	0.35
Propane	SL SOP	95	75-125	≤30	0.13	0.51
Butane	SL SOP	95	75-125	≤30	0.17	0.68
Pentane	SL SOP	95	75-125	≤30	0.21	0.84
Hexane	SL SOP	95	75-125	≤30	0.25	1.0
Ethene	SL SOP	95	75-125	≤30	0.051	0.33
1-Propene	SL SOP	95	75-125	≤30	0.25	0.49
1-Butene	SL SOP	95	75-125	≤30	0.25	0.65
1-Pentene	SL SOP	95	75-125	≤30	0.25	0.82
1-Hexene	SL SOP	95	75-125	≤30	0.25	0.98
Dissolved Gases In Water (GC/TCD)						
Carbon dioxide	SL SOP	95	75-125	≤30	32	2600
Carbon monoxide	SL SOP	95	75-125	≤30	21	82
Ethane	SL SOP	95	75-125	≤30	12	88
Ethene	SL SOP	95	75-125	≤30	5.4	82
Methane	SL SOP	95	75-125	≤30	3.0	47
Nitrogen	SL SOP	95	75-125	≤30	200	200
Oxygen	SL SOP	95	75-125	≤30	200	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Aluminum (ICP)	6010(3050)	2	75-125	≤20	5.8	20
Antimony (ICP)	6010(3050)	2	75-125	≤20	0.50	2.0
Antimony (GFAA)	7041(3050)	2	80-120	≤20	0.11	1.0
Arsenic (ICP)	6010(3050)	2	75-125	≤20	0.45	1.0
Arsenic (GFAA)	7060(3050)	2	80-120	≤20	0.35	1.0
	7062(3050)	2	80-120	≤20	0.25	1.0
Barium (ICP)	6010(3050)	2	75-125	≤20	0.16	1.0
Beryllium (ICP)	6010(3050)	2	75-125	≤20	0.057	0.40
Boron(ICP)	6010(3050)	2	75-125	≤20	0.59	5.0
Cadmium (ICP)	6010(3050)	2	75-125	≤20	0.087	0.50
Cadmium (GFAA)	7131 (3050)	2	80-120	≤20	0.087	0.10
Calcium (ICP)	6010(3050)	2	75-125	≤20	20	50
Chromium (ICP)	6010(3050)	2	75-125	≤20	0.17	1.0
Chromium (GFAA)	7191(3050)	2	80-120	≤20	0.079	1.0
Chromium, hexavalent	7196 (3060)	2	80-120	≤30	0.30	0.40
Cobalt (ICP)	6010 (3050)	2	75-125	≤20	0.19	1.0
Copper (ICP)	6010 (3050)	2	75-125	≤20	0.72	2.0
Copper (GFAA)	7211(3050)	2	80-120	≤20	0.042	1.0
Iron (ICP)	6010 (3050)	2	75-125	≤20	4.5	5.0
Lead (ICP)	6010 (3050)	2	75-125	≤20	0.42	0.50
Lead (GFAA)	7421(3050)	2	80-120	≤20	0.36	0.50
Lithium (GFAA)	7430(3050)	2	70-130	≤20	0.19	1.0
Lithium (FLES)	SL SOP	100	70-130	≤20	0.026	0.20
Magnesium (ICP)	6010 (3050)	2	75-125	≤20	6.8	50
Manganese (ICP)	6010(3050)	2	75-125	≤20	0.21	1.0
Mercury (CVAA)	7471	2	80-120	≤20	0.0028	0.020
	3112B	4	70-130	≤20	0.0028	0.020
Molybdenum (ICP)	6010(3050)	2	75-125	≤20	0.14	1.0
Nickel (ICP)	6010(3050)	2	75-125	≤20	0.43	4.0
Potassium (ICP)	6010(3050)	2	75-125	≤20	16	100
Selenium (ICP)	6010(3050)	2	75-125	≤20	0.43	1.0
Selenium (GFAA)	7740(3050)	2	80-120	≤20	0.66	1.0
	7742(3050)	2	80-120	≤20	0.38	1.0
Silica (water soluble)	6010(ASTM 3987-85)	2	70-130	≤20	0.67	10

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Silver (ICP)	6010(3050)	2	75-125	≤20	0.19	1.0
Silver (GFAA)	7761 (3050)	2	80-120	≤20	0.0040	0.10
Sodium (ICP)	6010(3050)	2	75-125	≤20	49	50
Thallium (ICP)	6010(3050)	2	75-125	≤20	0.57	1.0
Thallium (GFAA)	7841 (3050)	2	80-120	≤20	0.20	1.0
Tin (ICP)	6010(3050)	2	75-125	≤20	4.0	5.0
Titanium(ICP)	6010(3050)	2	70-130	≤20	0.40	1.0
Vanadium (ICP)	6010 (3050)	2	75-125	≤20	0.11	1.0
Zinc (ICP)	6010 (3050)	2	75-125	≤20	0.98	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
FLAA = flame atomic adsorption spectrophotometer
FLES = flame emission spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Ammonia (as N)	350.1(EPA-CE:3-140)	3/46	75-125	≤30	0.075	0.50
	350.3(EPA-CE)	3/46	75-125	≤30	0.055	0.50
BOD	EPA-CE:3-380	46	60-140	≤40	NA	200
BTU	D240-87	38	70-130	≤30	NA	200 BTU/lb
Carbon, total organic	EPA-CE [Walkley-Black]	46/43	60-140	≤40	64	500
	9060	2	60-140	≤40	150	500
Cation exchange capacity	9080/EPA-CE:3-20	2/46	70-130	≤40	NA	0.0033 meq/100 g
	9081	2	70-130	≤40	NA	0.0033 meq/100 g
Chloride (extractable)(1)	9251	2	75-125	≤30	4.8	20
	9252	2	75-125	≤30	17	20
	4500-Cl⁻C	4	75-125	≤30	5.0	20
	300.0/9056	82/2	75-125	≤25	0.68	20
Chloride, total	9251(5050)	2	70-130	≤40	NA	200
	9056(5050)	2	70-130	≤40	NA	200
COD	EPA-CE:3-373	46	60-140	≤40	NA	100
Coliform, fecal	9221C(EPA:62)	4/67	NA	≤200	NA	3 MPN/g
Coliform, total	9221B(EPA:62)	4/67	NA	≤200	NA	3 MPN/g
Cyanide, amenable to chlorination	9012(9013)	2	NA	≤50	NA	1.0
	9010(9013)	2	NA	≤40	NA	1.0
Cyanide, reactive	7.3.3.2/9014	2	NA	≤50	NA	100mg HCN/ Kg Waste
Cyanide, total	9012(9013)	2	75-125	≤30	0.50	1.0
	9012(9010)	2	75-125	≤30	0.50	1.0
Fluoride (extractable)(1)	340.2/4500F⁻C	3/4	75-125	≤25	0.38	4.0
	300.0/9056	82/2	75-125	≤25	0.20	2.0
Halogens, total	9056(5050)	2	70-130	≤40	NA	200
	9251(5050)	2	70-130	≤40	NA	200
Halogens, total organic (EOX)	EPA-600/4-84-008	2/44	60-140	≤50	8.6	10
Hydrogen ion (pH)(2)	9045	2	63-158	≤40	NA	NA
Ignitability	1010	2	NA	NA	NA	NA
Nitrate (as N)(extractable)	4500 NO_3^-F (EPA-CE:3-183)	4/46	75-125	≤30	1.0	5.0
	300.0/9056	82/2	75-125	≤25	0.18	5.0

[1]Extraction procedures for solids and semisolids are contained in Sections 8.2 and 8.3

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Pendimethaiin	8081(3550)	2	35-125	≤50	17	67
Permethrin (total)	8081(3550)	2	40-140	≤50	8.2	33
Propachlor	8081(3550)	2	51-125	≤30	2.5	16
Toxaphene	8081(3550)	2	41-126	≤50	33	170
Trifluralin	8081(3550)	2	40-140	≤40	0.20	0.66
PCB-1016	8082(3550)	2	34-138	≤44	5.7	33
PCB 1221	8082(3550)	2	15-178	≤30	7.6	67
PCB 1232	8082(3550)	2	10-215	≤30	3.5	33
PCB-1242	8082(3550)	2	39-150	≤30	6.5	33
PCB-1248	8082(3550)	2	38-158	≤30	5.9	33
PCB-1254	8082(3550)	2	40-122	≤30	8.2	33
PCB-1260	8082(3550)	2	39-138	≤30	8.0	33
PCB-1268	8082(3550)	2	40-140	≤30	4.0	33
Surrogate - Dibutylchlorendate (DBC)	8081(3550)	2	30-150	NA	NA	NA
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081/8082(3550)	2	30-150	NA	NA	NA
Surrogate- Decachlorobiphenyl (DCB)	8081/8082(3550)	2	30-150	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Individual Congeners (*)						
2,4'-Dichlorobiphenyl (8)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',5-Trichlorobiphenyl (18)	8082(3550)	2	50-150	≤50	0.25	1.0
2,4,4'-Trichlorobiphenyl (28)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,5'-Tetrachlorobiphenyl (44)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',5,5'-Tetrachlorobiphenyl (52)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3',4,4'-Tetrachlorobiphenyl (66)	8082(3550)	2	50-150	≤50	0.25	1.0
3,3',4,4'-Tetrachlorobiphenyl (77)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',4,5,5'-Pentachlorobiphenyl (101)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3,3',4,4'-Pentachlorobiphenyl (105)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',4,5,5'-Pentachlorobiphenyl (101)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3',4,4',5-Pentachlorobiphenyl (118)	8082(3550)	2	50-150	≤50	0.25	1.0
3,3',4,4',5-Pentachlorobiphenyl (126)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,3',4,4'-Hexachlorobiphenyl (128)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,4,4',5'-Hexachlorobiphenyl (138)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',4,4',5,5'-Hexachlorobiphenyl (153)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3,3',4,4',5-Hexachlorobiphenyl (156)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3,3',4,4',6-Hexachlorobiphenyl (158)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3',4,4',5,5'-Hexachlorobiphenyl (167)	8082(3550)	2	50-150	≤50	0.25	1.0
3,3',4,4',5,5'-Hexachlorobiphenyl (169)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,3',4,4',5-Heptachlorobiphenyl (170)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,4,4',5,5'-Heptachlorobiphenyl (180)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,4',5,5',6-Heptachlorobiphenyl (187)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,3',4,4',5,6-Octachlorobiphenyl (195)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (206)	8082(3550)	2	50-150	≤50	0.25	1.0
Decachlorobiphenyl (209)	8082(3550)	2	50-150	≤50	0.25	1.0
Surrogate - 2,4,5,6-Tetrachloro-m- xylene (TCMX)	8082(3550)	2	30-150	NA	NA	NA
Surrogate- Decachlorobiphenyl (DCB)	8082(3550)	2	30-150	NA	NA	NA
Surrogate- Octachloronaphthalene	8082(3550)	2	30-150	NA	NA	NA

*These congeners are representitive of the 209 individual PCBs that may be determined using Method 8082. The number in parenthesis after the compound name is the PCB congener number. PCBs as Aroclors are listed in the previous section.

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL.A (ug/kg)
Acetone	8260(5035)	2	43-154	≤28	3.6	50
	8260(5035ext)	2	43-154	≤28	140	2000
Acetonitrile	8260(5035)	2	78-151	≤40	62	200
	8260(5035ext)	2	78-151	≤40	6200	8000
Acrolein	8260(5035)	2	22-164	≤65	38	100
	8260(5035ext)	2	22-164	≤65	1500	4000
Acrylonitrile	8260(5035)	2	41-140	≤40	11	100
	8260(5035ext)	2	41-140	≤40	440	4000
Benzene (MS)	8260(5035)	2	49-142	≤42	1.7	5.0
	8260(5035ext)	2	49-142	≤42	68	200
Benzyl Chloride	8260(5035)	2	50-150	≤40	25	100
	8260(5035ext)	2	50-150	≤40	1000	4000
Bromobenzene	8260(5035)	2	34-145	≤33	1.2	5.0
	8260(5035ext)	2	34-145	≤33	48	200
Bromochloromethane	8260(5035)	2	41-146	≤30	1.2	5.0
	8260(5035ext)	2	41-146	≤40	48	200
Bromodichloromethane	8260(5035)	2	32-149	≤33	0.88	5.0
	8260(5035ext)	2	32-149	≤33	35	200
Bromoform	8260(5035)	2	41-138	≤24	0.82	5.0
	8260(5035ext)	2	41-138	≤24	33	200
Bromomethane	8260(5035)	2	23-173	≤79	3.6	10
	8260(5035ext)	2	23-173	≤79	140	400
2-Butanone (MEK)	8260(5035)	2	45-154	≤39	3.3	25
	8260(5035ext)	2	45-154	≤39	130	1000
n-Butylbenzene	8260(5035)	2	29-142	≤72	2.1	5.0
	8260(5035ext)	2	29-142	≤72	84	200
sec-Butylbenzene	8260(5035)	2	35-142	≤63	2.4	5.0
	8260(5035ext)	2	35-142	≤63	96	200
tert-Butylbenzene	8260(5035)	2	54-130	≤40	2.0	5.0
	8260(5035ext)	2	54-130	≤40	80	200
Carbon disulfide	8260(5035)	2	40-135	≤68	2.2	5.0
	8260(5035ext)	2	40-135	≤68	88	200
Carbon tetrachloride	8260(5035)	2	40-135	≤59	1.9	5.0
	8260(5035ext)	2	40-135	≤59	76	200
Chlorobenzene (MS)	8260(5035)	2	66-135	≤34	1.3	5.0
	8260(5035ext)	2	66-135	≤34	52	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2-Chloro-1,3-butadiene (Chloroprene)	8260(5035)	2	28-256	≤65	1.7	5.0
	8260(5035ext)	2	28-256	≤65	68	200
Chloroethane	8260(5035)	2	30-135	≤51	2.2	10
	8260(5035ext)	2	30-135	≤51	88	400
2-Chloroethyl vinyl ether	8260(5035)	2	D-208	≤28	3.2	50
	8260(5035ext)	2	D-208	≤28	130	2000
Chloroform	8260(5035)	2	50-133	≤38	1.7	5.0
	8260(5035ext)	2	50-133	≤38	68	200
Chloromethane	8260(5035)	2	32-142	≤53	1.3	10
	8260(5035ext)	2	32-142	≤53	52	400
3-Chloropropene (Allyl chloride)	8260(5035)	2	50-150	≤40	0.26	5.0
	8260(5035ext)	2	50-150	≤40	10	200
2-Chlorotoluene	8260(5035)	2	34-150	≤33	1.0	5.0
	8260(5035ext)	2	34-150	≤33	40	200
4-Chlorotoluene	8260(5035)	2	34-139	≤37	1.2	5.0
	8260(5035ext)	2	34-139	≤37	48	200
Cyclohexanone	8260 (5035)	2	38-111	≤52	240	750
	8260**(5035ext)	2	38-111	≤52	9600	30000
Dibromochloromethane	8260(5035)	2	47-135	≤22	0.84	5.0
	8260(5035ext)	2	47-135	≤22	34	200
1,2-Dibromo-3-chloropropane (DBCP)	8260(5035)	2	28-148	≤36	0.61	10
	8260(5035ext)	2	28-148	≤36	24	400
1,2-Dibromoethane (EDB)	8260(5035)	2	29-161	≤40	0.86	5.0
	8260(5035ext)	2	29-161	≤40	34	200
Dibromomethane	8260(5035)	2	35-155	≤24	0.93	5.0
	8260(5035ext)	2	35-155	≤24	37	200
1,2-Dichlorobenzene	8260(5035)	2	37-139	≤28	1.1	5.0
	8260(5035ext)	2	37-139	≤28	44	200
1,3-Dichlorobenzene	8260(5035)	2	35-143	≤34	1.3	5.0
	8260(5035ext)	2	35-143	≤34	52	200
1,4-Dichlorobenzene	8260(5035)	2	34-138	≤36	1.4	5.0
	8260(5035)	2	34-138	≤36	56	200
trans-1,4-Dichloro-2-butene	8260(5035)	2	79-178	≤40	0.94	10
	8260(5035ext)	2	79-178	≤40	38	400
Dichlorodifluoromethane	8260(5035)	2	53-126	≤41	2.4	5.0
	8260(5035ext)	2	53-126	≤41	96	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL A (ug/kg)
1,1-Dichloroethane	8260(5035)	2	51-129	≤38	1.6	5.0
	8260(5035ext)	2	51-129	≤38	64	200
1,2-Dichloroethane	8260(5035)	2	49-136	≤25	1.2	5.0
	8260(5035ext)	2	49-136	≤25	48	200
cis-1,2-Dichloroethene	8260(5035)	2	44-124	≤37	1.7	5.0
	8260(5035ext)	2	44-124	≤37	68	200
trans-1,2-Dichloroethene	8260(5035)	2	37-142	≤56	1.9	5.0
	8260(5035ext)	2	37-142	≤56	76	200
1,1-Dichloroethene (MS)	8260(5035)	2	40-164	≤46	2.2	5.0
	8260(5035ext)	2	40-146	≤46	88	200
1,2-Dichloropropane	8260(5035)	2	52-124	≤27	1.4	5.0
	8260(5035ext)	2	52-124	≤27	56	200
1,3-Dichloropropane	8260(5035)	2	44-136	≤24	1.6	5.0
	8260(5035ext)	2	44-136	≤24	64	200
2,2-Dichloropropane	8260(5035)	2	43-129	≤53	2.0	5.0
	8260(5035ext)	2	43-129	≤53	80	200
1,1-Dichloropropene	8260(5035)	2	34-134	≤58	2.0	5.0
	8260(5035ext)	2	34-134	≤58	80	200
cis-1,3-Dichloropropene	8260(5035)	2	40-133	≤34	0.99	5.0
	8260(5035ext)	2	40-133	≤34	40	200
trans-1,3-Dichloropropene	8260(5035)	2	45-131	≤50	2.1	5.0
	8260(5035ext)	2	45-131	≤50	84	200
Diethyl ether	8260(5035)	2	50-150	≤40	2.5	10
	8260(5035ext)	2	50-150	≤40	100	400
Ethanol	8240(5030)8260 (5035)	2	40-160	≤40	250	1000
	8260(5035ext)	2	40-160	≤40	10000	40000
Ethyl Acetate	8260(5035)	2	73-137	≤21	1.7	5.0
	8260(5035ext)	2	73-137	≤21	68	200
Ethylbenzene	8260(5035)	2	51-135	≤44	2.0	5.0
	8260(5035ext)	2	51-135	≤44	80	200
Ethyl methacrylate	8260(5035)	2	50-150	≤40	1.6	5.0
	8260(5035ext)	2	50-150	≤40	64	200
Ethylene oxide	8260(5035)	2	D-411	≤83	900	3500
	8260**(5035ext)	2	D-411	≤83	36000	140000
Hexachlorobutadiene	8260(5035)	2	30-140	≤40	1.4	5.0
	8260**(5035ext)	2	30-140	≤40	56	200
2-Hexanone	8260(5035)	2	45-127	≤32	4.8	25
	8260(5035ext)	2	45-127	≤32	190	1000

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REP	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Iodomethane	8260(5035)	2	50-150	≤40	1.9	5.0
	8260(5035ext)	2	50-150	≤40	76	200
Isobutyl alcohol	8260(5035)	2	63-173	≤40	100	200
	8260(5035ext)	2	63-173	≤40	4000	8000
Isopropylbenzene	8260(5035)	2	38-148	≤39	3.8	5.0
	8260(5035ext)	2	38-148	≤39	150	200
p-Isopropyltoluene	8260(5035)	2	70-130	≤30	1.4	5.0
	8260(5035ext)	2	70-130	≤30	56	200
Methacrylonitrile	8260(5035)	2	69-145	≤60	32	100
	8260(5035ext)	2	69-145	≤60	1300	4000
Methylene chloride	8260(5035)	2	44-142	≤32	1.4	5.0
	8260(5035ext)	2	44-142	≤32	56	200
Methyl methacrylate	8260(5035)	2	50-150	≤45	2.2	5.0
	8260(5035ext)	2	50-150	≤45	88	200
4-Methyl-2-pentanone (MIBK)	8260(5035)	2	34-159	≤37	4.9	25
	8260(5035ext)	2	34-159	≤37	200	1000
Methyl t-butyl ether (MTBE)	8260(5035)	2	67-128	≤20	1.2	50
	8260(5035ext)	2	67-128	≤20	48	2000
Naphthalene	8260(5035)	2	37-131	≤29	0.57	5.0
	8260(5035ext)	2	37-131	≤29	23	200
2-Nitropropane	8260(5035)	2	35-128	≤47	2.4	10
	8260(5035ext)	2	35-128	≤47	96	400
Pentachloroethane	8260(5035)	2	41-165	≤50	10	25
	8260(5035ext)	2	41-165	≤50	400	1000
Propionitrile (ethylcyanide)	8260(5035)	2	73-227	≤65	37	100
	8260(5035ext)	2	73-227	≤65	1500	4000
n-Propylbenzene	8260(5035)	2	31-148	≤32	1.7	5.0
	8260(5035ext)	2	31-148	≤32	68	200
Styrene	8260(5035)	2	43-140	≤45	1.8	5.0
	8260(5035ext)	2	43-140	≤45	72	200
1,1,1,2-Tetrachloroethane	8260(5035)	2	38-132	≤32	1.3	5.0
	8260(5035ext)	2	38-132	≤32	52	200
1,1,2,2-Tetrachloroethane	8260(5035)	2	49-144	≤28	1.0	5.0
	8260(5035ext)	2	49-144	≤28	40	200
Tetrachloroethene	8260(5035)	2	71-146	≤44	3.0	5.0
	8260(5035ext)	2	71-146	≤44	120	200
Toluene (MS)	8260(5035)	2	38-158	≤32	1.8	5.0
	8260(5035ext)	2	38-158	≤32	72	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
1,2,3-Trichlorobenzene	8260(5035)	2	24-138	≤26	0.96	5.0
	8260(5035ext)	2	24-138	≤26	38	200
1,2,4-Trichlorobenzene	8260(5035)	2	22-138	≤35	1.0	5.0
	8260(5035ext)	2	22-138	≤35	40	200
1,1,1-Trichloroethane	8260(5035)	2	41-134	≤54	2.1	5.0
	8260(5035ext)	2	41-134	≤54	84	200
1,1,2-Trichloroethane	8260(5035)	2	34-148	≤27	0.96	5.0
	8260(5035ext)	2	34-148	≤27	38	200
Trichloroethene (MS)	8260(5035)	2	51-146	≤34	2.1	5.0
	8260(5035ext)	2	51-146	≤34	84	200
Trichlorofluoromethane	8260(5035)	2	32-138	≤30	2.8	5.0
	8260(5035ext)	2	32-138	≤30	110	200
1,2,3-Trichloropropane	8260(5035)	2	30-167	≤25	1.0	5.0
	8260(5035ext)	2	30-167	≤25	40	200
1,1,2-Trichloro-1,2,2-trifluoroethane	8260(5035)	2	17-181	≤65	2.8	5.0
	8260(5035ext)	2	17-181	≤65	110	200
1,2,4-Trimethylbenzene	8260(5035)	2	41-146	≤23	1.0	5.0
	8260(5035ext)	2	41-146	≤23	40	200
1,3,5-Trimethylbenzene	8260(5035)	2	38-144	≤28	1.2	5.0
	8260(5035ext)	2	38-144	≤28	48	200
Vinyl acetate	8260(5035)	2	10-174	≤133	1.0	10
	8260(5035ext)	2	10-174	≤133	40	400
Vinyl chloride	8260(5035)	2	33-142	≤65	1.5	10
	8260(5035ext)	2	33-142	≤65	60	400
Xylenes (total)	8260(5035)	2	37-133	≤43	3.0	10
	8260(5035ext)	2	37-133	≤43	120	400
o-Xylene	8260(5035)	2	41-148	≤32	1.6	5.0
	8260(5035ext)	2	41-148	≤32	64	200
m+p-Xylene	8260(5035)	2	37-133	≤43	3.0	5.0
	8260(5035ext)	2	37-133	≤43	120	200
Surrogate - Toluene-d8	8260(5035)	2	64-136	NA	NA	NA
	8260(5035ext)	2	64-136	NA	NA	NA
Surrogate - p-Bromofluorobenzene	8260(5035)	2	63-135	NA	NA	NA
	8260(5035ext)	2	63-135	NA	NA	NA
Surrogate --Dibromofluoromethane	8260(5035)	2	58-142	NA	NA	NA
	8260(5035ext)	2	58-142	NA	NA	NA
Surrogate - 1,2-Dichloroethane-d4	8260(5035)	2	70-130	NA	NA	NA
	8260(5035ext)	2	70-130	NA	NA	NA
Surrogate -1,2-Dichlorobenzene-d4	8260(5035)	2	70-130	NA	NA	NA
	8260(5035ext)	2	70-130	NA	NA	NA

(ext) = methanol extraction; 1mL methanol per gram of sample

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acenaphthene (MS)	8270(3550)	2	18-123	≤49	29	330
	8270(3550)-Low Level	2	18-123	≤49	1.5	6.7
Acenaphthylene	8270(3550)	2	42-119	≤48	21	330
	8270(3550)-Low Level	2	42-119	≤48	1.2	6.7
Acetophenone	8270(3550)	2	10-150	≤50	41	330
2-Acetylaminofluorene	8270(3550)	2	25-150	≤50	49	330
Aldrin	8270(3550)	2	10-166	≤40	76	330
4-Aminobiphenyl	8270(3550)	2	10-150	≤50	59	330
Aniline	8270(3550)	2	10-150	≤50	23	330
Anthracene	8270(3550)	2	40-148	≤27	21	330
	8270(3550)-Low Level	2	40-148	≤27	1.2	6.7
Aramite	8270(3550)	2	40-150	≤50	36	330
Benzidine	8270(3550)	2	10-200	≤100	36	2700
Benzo(a)anthracene	8270(3550)	2	54-137	≤43	20	330
	8270(3550)-Low Level	2	54-137	≤43	1.5	6.7
Benzoic acid	8270(3550)	2	10-150	≤50	61	1700
Benzo(b)fluoranthene	8270(3550)	2	43-134	≤51	23	330
	8270(3550)-Low Level	2	43-134	≤51	1.5	6.7
Benzo(k)fluoranthene	8270(3550)	2	25-182	≤48	26	330
	8270(3550)-Low Level	2	25-182	≤48	1.5	6.7
Benzo(g,h,i)perylene	8270(3550)	2	10-148	≤50	26	330
	8270(3550)-Low Level	2	10-148	≤50	1.3	6.7
Benzo(a)pyrene	8270(3550)	2	41-142	≤55	29	330
	8270(3550)-Low Level	2	41-142	≤55	1.2	6.7
Benzyl alcohol	8270(3550)	2/6	10-150	≤50	30	330
Benzyl chloride	8270(3550)	2	10-150	≤50	82	330
alpha-BHC	8270(3550)	2	10-150	≤50	56	330
beta-BHC	8270(3550)	2	24-149	≤40	55	330
delta-BHC	8270(3550)	2	10-110	≤40	55	330
gamma-BHC	8270(3550)	2	10-150	≤50	62	330
Biphenyl (Diphenyl)	8270(3550)	2	31-150	≤57	197	660
Bis(2-chloroethoxy) methane	8270(3550)	2	34-108	≤52	29	330
Bis(2-chloroethyl) ether	8270(3550)	2	18-122	≤50	26	330
Bis(2-chloroisopropyl) ether	8270(3550)	2	10-135	≤28	33	330
Bis(2-ethylhexyl) phthalate	8270(3550)	2	47-143	≤22	45	330
4-Bromophenyl phenyl ether	8270(3550)	2	31-157	≤19	24	330
Butyl benzyl phthalate	8270(3550)	2	58-122	≤27	42	330
Carbazole	8270(3550)	2	10-158	≤50	22	330

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
4-Chloroaniline	8270(3550)	2	10-130	≤85	37	660
4-Chloro-3-methylphenol (MS)	8270(3550)	2	24-114	≤32	49	330
1-Chloronaphthalene	8270(3550)	2	10-150	≤50	82	330
2-Chloronaphthalene	8270(3550)	2	39-107	≤47	24	330
2-Chlorophenol (MS)	8270(3550)	2	15-111	≤38	32	330
4-Chlorophenylphenyl ether	8270(3550)	2	36-149	≤62	28	330
Chrysene	8270(3550)	2	56-133	≤41	21	330
	8270(3550)-Low Level	2	56-133	≤41	2.1	6.7
2-Methyl phenol (o-Cresol)	8270(3550)	2	33-108	≤53	32	330
3- Methyl phenol (m- Cresol)	8270(3550)	2	24-114	≤42	42	330
4-Methyl phenol (p-Cresol)	8270(3550)	2	24-114	≤42	42	330
3- and 4-Methyl phenol	8270(3550)	2	24-114	≤42	42	330
4,4'-DDD	8270(3550)	2	10-145	≤40	36	330
4,4'-DDE	8270(3550)	2	10-136	≤40	57	330
4,4'-DDT	8270(3550)	2	10-203	≤62	56	330
Diallate	8270(3550)	2	10-150	≤50	39	330
Dibenz(a,h)anthracene	8270(3550)	2	31-129	≤24	52	330
	8270(3550)-Low Level	2	31-129	≤24	1.4	6.7
Dibenzofuran	8270(3550)	2	36-132	≤42	30	330
Dibenzo(a,e)pyrene	8270(3550)	2	35-109	≤42	110	330
Di-n-butylphthalate	8270(3550)	2	42-161	≤59	28	330
1,2-Dichlorobenzene	8270(3550)	2	25-115	≤24	24	330
1,3-Dichlorobenzene	8270(3550)	2	26-108	≤28	31	330
1,4-Dichlorobenzene (MS)	8270(3550)	2	10-105	≤31	27	330
3,3'-Dichlorobenzidine	8270(3550)	2	10-115	≤39	150	660
2,4-Dichlorophenol	8270(3550)	2	32-130	≤60	31	330
2,6-Dichlorophenol	8270(3550)	2	10-150	≤50	37	330
Dieldrin	8270(3550)	2	29-136	≤40	46	330
Diethylphthalate	8270(3550)	2	31-130	≤40	32	330
Dimethoate	8270(3550)	2	10-150	≤50	100	330
p-(Dimethylamino)azobenzene	8270(3550)	2	10-150	≤50	20	330
7,12-Dimethylbenz(a)anthracene	8270(3550)	2	10-150	≤50	17	330
3,3'-Dimethylbenzidine	8270(3550)	2	10-200	≤100	91	1700
a,a-Dimethylphenethylamine	8270(3550)	2	10-150	≤50	17000	67000
2,4-Dimethylphenol	8270(3550)	2	33-84	≤54	28	330
Dimethylphthalate	8270(3550)	2	49-130	≤45	25	330
m-Dinitrobenzene	8270(3550)	2	10-150	≤50	27	330
-Dinitrobenzene	8270(3550)	2	49-105	≤40	400	1700
6-Dinitro-2-methylphenol	8270(3550)	2	10-117	≤57	200	1700

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,4-Dinitrophenol	8270(3550)	2	10-125	≤84	150	1700
2,4-Dinitrotoluene (MS)	8270(3550)	2	11-120	≤37	40	330
2,6-Dinitrotoluene	8270(3550)	2	10-112	≤45	29	330
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270(3550)	2	10-150	≤50	30	330
Di-n-octylphthalate	8270(3550)	2	22-181	≤43	54	330
1,4-Dioxane	8270(3550)	2	10-150	≤50	15	330
Diphenylamine/ N-nitrosodiphenylamine	8270(3550)	2	51-132	≤44	25	330
Diphenylamine	8270(3550)	2	51-132	≤44	25	330
1,2-Diphenyl hydrazine	8270(3550)	2	10-150	≤50	27	330
Disulfoton	8270(3550)	2	10-150	≤50	100	330
Endosulfan I	8270(3550)	2	10-150	≤50	80	660
Endosulfan II	8270(3550)	2	10-150	≤50	50	660
Endosulfan sulfate	8270(3550)	2	10-107	≤50	46	660
Endrin	8270(3550)	2	10-150	≤50	70	660
Endrin aldehyde	8270(3550)	2	10-209	≤50	72	1700
Endrin ketone	8270(3550)	2	10-150	≤50	420	1700
Ethyl carbamate	8270(3550)	2	48-100	≤20	34	330
Ethyl methanesulfonate	8270(3550)	2	10-150	≤50	55	330
Ethyl parathion	8270(3550)	2	10-150	≤50	100	330
Famphur	8270(3550)	2	10-150	≤50	100	330
Fluoranthene	8270(3550)	2	39-157	≤50	31	330
	8270(3550)-Low Level	2	39-157	≤50	1.5	6.7
Fluorene	8270(3550)	2	27-151	≤50	31	330
	8270(3550)-Low Level	2	27-151	≤50	1.1	6.7
Heptachlor	8270(3550)	2	10-192	≤40	45	660
Heptachlor epoxide	8270(3550)	2	26-155	≤55	52	660
Hexachlorobenzene	8270(3550)	2	19-155	≤33	45	330
Hexachlorobutadiene	8270(3550)	2	33-114	≤55	23	330
Hexachlorocyclopentadiene	8270(3550)	2	D-132	≤50	51	330
Hexachloroethane	8270(3550)	2	10-109	≤30	24	330
Hexachlorophene*	8270(3550)	2	10-200	≤80	1400	170000
Hexachloropropene	8270(3550)	2	10-150	≤50	41	330
Indeno(1,2,3-cd)pyrene	8270(3550)	2	24-136	≤28	67	330
	8270(3550)-Low Level	2	24-136	≤28	1.0	6.7
Isophorone	8270(3550)	2	15-115	≤50	26	330
Isosafrole	8270(3550)	2	10-150	≤50	47	330
Kepone	8270(3550)	2	10-150	≤50	80	330
Methapyrilene	8270(3550)	2	10-150	≤50	960	67000
3-Methylcholanthrene	8270(3550)	2	10-150	≤50	47	330

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
4,4-Methylenebis(2-chloroaniline)	8270(3550)	2	40-122	≤57	4500	17000
Methyl methanesulfonate	8270(3550)	2	10-150	≤50	38	330
1-Methylnaphthalene	8270(3550)	2	10-150	≤50	30	330
	8270(3550)-Low Level	2	10-150	≤50	1.2	6.7
2-Methylnaphthalene	8270(3550)	2	30-133	≤63	32	330
	8270(3550)-Low Level	2	30-133	≤63	0.74	6.7
Methyl parathion	8270(3550)	2	10-150	≤50	100	330
Naphthalene	8270(3550)	2	25-131	≤34	25	330
	8270(3550)-Low Level	2	25-131	≤34	1.0	6.7
1,4-naphthoquinone	8270(3550)	2	10-150	≤50	27	330
1-Naphthylamine	8270(3550)	2	10-150	≤50	78	330
2-Naphthylamine	8270(3550)	2	10-150	≤50	77	330
Nicotine	8270(3550)	2	10-150	≤50	820	3300
2-Nitroaniline	8270(3550)	2	17-130	≤48	42	1700
3-Nitroanline	8270(3550)	2	14-130	≤28	32	1700
4-Nitroaniline	8270(3550)	2	10-130	≤55	42	1700
Nitrobenzene	8270(3550)	2	19-120	≤30	27	330
2-Nitrophenol	8270(3550)	2	30-130	≤50	19	330
4-Nitrophenol(MS)	8270(3550)	2	15-118	≤57	160	1700
4-Nitroquinoline-1-oxide	8270(3550)	2	10-150	≤50	190	3300
N-Nitroso-di-N-butylamine	8270(3550)	2	10-150	≤50	40	330
N-Nitrosodiethylamine	8270(3550)	2	10-150	≤50	18	330
N-Nitrosodimethylamine	8270(3550)	2	10-150	≤50	49	330
N-Nitrosodiphenylamine/ Diphenylamine	8270(3550)	2	51-132	≤44	25	330
N-Nitrosodiphenylamine	8270(3550)	2	51-132	≤44	25	330
N-Nitrosos-di-N-propylamine (MS)	8270(3550)	2	11-122	≤37	32	330
N-Nitrosomethylethylamine	8270(3550)	2	10-150	≤50	130	330
N-Nitrosomorpholine	8270(3550)	2	10-150	≤50	36	330
N-Nitrosopiperidine	8270(3550)	2	10-150	≤50	42	330
N-Nitrosopyrrolidine	8270(3550)	2	10-150	≤50	44	330
5-Nitro-o-toluidine	8270(3550)	2	10-150	≤50	21	330
Pentachlorobenzene	8270(3550)	2	10-150	≤50	49	330
Pentachloronitrobenzene	8270(3550)	2	10-150	≤50	26	330
Pentachlorophenol (MS)	8270(3550)	2	10-140	≤55	180	1700
Phenacetin	8270(3550)	2	10-150	≤50	55	330
Phenanthrene	8270(3550)	2	39-152	≤30	17	330
	8270(3550)-Low Level	2	39-152	≤30	1.2	6.7
Phenol (MS)	8270(3550)	2	13-115	≤39	42	330
Phenyl ether (Diphenyl oxide)	8270(3550)	2	29-157	≤61	212	660

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL-A (ug/kg)
p-Phenylenediamine	8270(3550)	2	10-150	≤50	140	1700
Phorate	8270(3550)	2	10-150	≤50	100	330
2-Picoline	8270(3550)	2	10-150	≤50	33	330
Pronamide	8270(3550)	2	10-150	≤50	38	330
Pyrene (MS)	8270(3550)	2	10-133	≤42	63	330
	8270(3550)-Low Level	2	10-133	≤42	1.7	6.7
Pyridine	8270(3550)	2	10-150	≤50	30	330
Safrole	8270(3550)	2	10-150	≤50	50	330
Strychnine	8270(3550)	2	10-150	≤50	820	3300
Sulfotepp	8270(3550)	2	10-150	≤50	100	330
1,2,4,5-Tetrachlorobenzene	8270(3550)	2	10-150	≤50	43	330
2,3,4,5-Tetrachlorophenol	8270(3550)	2	10-150	≤50	61	330
2,3,4,6-Tetrachlorophenol	8270(3550)	2	36-121	≤31	25	330
Tetrachlorophenols (2,3,4,5 + 2,3,4,6)	8270(3550)	2	NA	NA	NA	330
Thionazin	8270(3550)	2	10-150	≤50	100	330
o-Toluidine	8270(3550)	2	10-150	≤50	31	330
1,2,4-Trichlorobenzene (MS)	8270(3550)	2	10-112	≤22	22	330
2,4,5-Trichlorophenol	8270(3550)	2	25-130	≤36	29	330
2,4,6-Trichlorophenol	8270(3550)	2	41-130	≤30	20	330
Trichlorophenols (2,4,5 + 2,4,6)	8270(3550)	2	NA	NA	NA	330
o,o,o-Triethylphosphorothioate	8270(3550)	2	10-150	≤50	51	330
1,3,5-Trinitrobenzene	8270(3550)	2	10-150	≤50	21	330
Surrogate – Nitrobenzene-d5	8270(3550)	2	20-120	NA	NA	NA
Surrogate – 2-Fluorobiphenyl	8270(3550)	2	30-120	NA	NA	NA
Surrogate – p-Terphenyl-d14	8270(3550)	2	30-131	NA	NA	NA
Surrogate – Phenol-d5	8270(3550)	2	19-114	NA	NA	NA
Surrogate – 2-Fluorophenol	8270(3550)	2	16-113	NA	NA	NA
Surrogate – 2,4,6- Tribromophenol	8270(3550)	2	23-129	NA	NA	NA
Surrogate –Ortho Terphenyl	8270(3550)-low level	2	30-130	NA	NA	NA

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/)	RLA (ug/)
Selenium(ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.46	1.0
Selenium (GFAA)	OSHA ID-121	99	80-120	≤20	0.13	1.0
Silver (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.20	1.0
Silver (GFAA)	OSHA ID-121	99	80-120	≤20	0.016	0.10
Sodium (ICP)	NIOSH 7300	98	75-125	≤20	8.3	50
Thallium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.46	1.0
Thallium (GFAA)	OSHA ID-121	99	80-120	≤20	0.14	1.0
Tin (ICP)	OSHA ID-125G	99	75-125	≤20	1.3	5.0
Titanium(ICP)	NIOSH 7300	98	70-130	≤20	0.25	1.0
Vanadium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.23	1.0
Zinc (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.45	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Chloride	NIOSH 7903	98	75-125	≤25	2.5	10
Fluoride	NIOSH 7903	98	75-125	≤25	0.25	1.0
Nitrate	NIOSH 7903	98	75-125	≤25	0.25	1.0
Sulfate	NIOSH 7903	98	75-125	≤25	0.13	50

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Aluminum (ICP)	6010(3050)	2	75-125	≤20	3.2	20
Antimony (ICP)	6010(3050)	2	75-125	≤20	0.42	5.0
Antimony (GFAA)	7041(3050)	2	80-120	≤20	0.11	1.0
Arsenic (ICP)	6010(3050)	2	75-125	≤20	0.39	1.0
Arsenic (GFAA)	7060(3050)	2	80-120	≤20	0.27	1.0
Barium (ICP)	6010(3050)	2	75-125	≤20	0.42	1.0
Beryllium (ICP)	6010(3050)	2	75-125	≤20	0.11	0.40
Boron(ICP)	6010(3050)	2	75-125	≤20	0.59	5.0
Cadmium (ICP)	6010(3050)	2	75-125	≤20	0.056	0.50
Cadmium (GFAA)	7131 (3050)	2	80-120	≤20	0.0090	0.10
Calcium (ICP)	6010(3050)	2	75-125	≤20	2.4	50
Chromium (ICP)	6010(3050)	2	75-125	≤20	0.18	1.0
Chromium (GFAA)	7191(3050)	2	80-120	≤20	0.050	1.0
Cobalt (ICP)	6010 (3050)	2	75-125	≤20	0.13	1.0
Copper (ICP)	6010 (3050)	2	75-125	≤20	0.22	2.0
Iron (ICP)	6010 (3050)	2	75-125	≤20	3.9	5.0
Lead (ICP)	6010 (3050)	2	75-125	≤20	0.20	0.50
Lead (GFAA)	7421(3050)	2	80-120	≤20	0.14	0.50
Magnesium (ICP)	6010 (3050)	2	75-125	≤20	1.8	50
Manganese (ICP)	6010(3050)	2	75-125	≤20	0.18	1.0
Mercury (CVAA)	7471	2	80-120	≤20	0.0020	0.020
Molybdenum (ICP)	6010(3050)	2	75-125	≤20	0.098	1.0
Nickel (ICP)	6010(3050)	2	75-125	≤20	1.1	4.0
Potassium (ICP)	6010(3050)	2	75-125	≤20	35	100
Selenium (ICP)	6010 (3050)	2	75-125	≤20	0.46	1.0
Selenium (GFAA)	7740(3050)	2	80-120	≤20	0.13	1.0

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Silver (ICP)	6010 (3050)	2	75-125	≤20	0.14	1.0
Silver (GFAA)	7761 (3050)	2	80-120	≤20	0.016	0.10
Sodium (ICP)	6010(3050)	2	75-125	≤20	8.3	50
Strontium(ICP)	6010(3050)	2	70-130	≤30	0.84	1.0
Thallium (ICP)	6010 (3050)	2	75-125	≤20	0.46	1.0
Thallium (GFAA)	7841 (3050)	2	80-120	≤20	0.14	1.0
Tin (ICP)	6010(3050)	2	75-125	≤20	1.3	5.0
Titanium(ICP)	6010(3050)	2	70-130	≤30	0.25	1.0
Vanadium (ICP)	6010 (3050)	2	75-125	≤20	0.23	1.0
Zinc (ICP)	6010 (3050)	2	75-125	≤20	0.45	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer
-T = trace ICP

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Cyanide, total	9012(9013)	2	75-125	≤30	0.59	1.0
	9012(9010)	2	75-125	≤30	0.14	1.0

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL*** (ug/kg)	RLA (ug/kg)
Aldrin (MS)	8081(3550)	97/2	10-144	≤38	0.021	1.7
Alpha-BHC	8081(3550)	97/2	22-101	≤40	0.088	1.7
Beta-BHC	8081(3550)	97/2	12-120	≤40	0.16	1.7
Gamma-BHC (Lindane) (MS)	8081(3550)	97/2	12-138	≤37	0.066	1.7
Delta-BHC	8081(3550)	97/2	10-142	≤47	0.18	1.7
Technical Chlordane	8081(3550)	97/2	45-119	≤40	1.7	17
Alpha Chlordane	8081(3550)	97/2	45-140	≤40	0.12	1.7
Gamma Chlordane	8081(3550)	97/2	11-141	≤40	0.078	1.7
4,4'-DDD	8081(3550)	97/2	28-134	≤50	0.22	3.3
4,4'-DDE	8081(3550)	97/2	34-121	≤23	0.13	3.3
4,4'-DDT (MS)	8081(3550)	97/2	29-134	≤26	0.49	3.3
Dieldrin (MS)	8081(3550)	97/2	28-137	≤30	0.19	3.3
Endosulfan I	8081(3550)	97/2	10-141	≤40	0.13	1.7
Endosulfan II	8081(3550)	97/2	10-159	≤65	0.22	3.3
Endosulfan sulfate	8081(3550)	97/2	26-144	≤50	0.91	3.3
Endrin (MS)	8081(3550)	97/2	33-149	≤52	0.10	3.3
Endrin aldehyde	8081(3550)	97/2	10-130	≤86	0.22	3.3
Endrin ketone	8081(3550)	97/2	29-112	≤31	0.42	3.3
Heptachlor (MS)	8081(3550)	97/2	17-138	≤38	0.12	1.7
Heptachlor epoxide	8081(3550)	97/2	15-142	≤40	0.099	1.7
Methoxychlor	8081(3550)	97/2	24-152	≤40	1.3	17
Toxaphene	8081(3550)	97/2	41-126	≤50	18	170
PCB-1016	8082(3550)	97/2	34-138	≤44	4.8	33
PCB 1221	8082(3550)	97/2	15-178	≤30	23	67
PCB 1232	8082(3550)	97/2	10-215	≤30	4.0	33
PCB-1242	8082(3550)	97/2	39-150	≤30	6.0	33
PCB-1248	8082(3550)	97/2	38-158	≤30	6.7	33
PCB-1254	8082(3550)	97/2	40-122	≤30	2.7	33
PCB-1260	8082(3550)	97/2	39-138	≤30	5.6	33
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081(3550)/8082(3550)	97/2	10-114	NA	NA	NA
Surrogate-Decachlorobiphenyl (DCB)	8081(3550)/8082(3550)	97/2	27-128	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Monochlorobiphenyls	680/(3550)	97/93	30-130	≤50	0.68	10
Dihlorobiphenyls	680/(3550)	97/93	30-130	≤50	0.76	10
Trichlorobiphenyls	680/(3550)	97/93	30-130	≤50	0.68	10
Tetrachlorobiphenyls	680/(3550)	97/93	40-140	≤50	1.3	20
Pentachlorobiphenyls	680/(3550)	97/93	40-140	≤50	0.83	20
Hexachlorobiphenyls	680/(3550)	97/93	40-140	≤50	0.89	20
Heptachlorobiphenyls	680/(3550)	97/93	40-140	≤50	1.6	30
Octachlorobiphenyls	680/(3550)	97/93	40-140	≤50	0.95	30
Nonachlorobiphenyls	680/(3550)	97/93	30-130	≤50	1.9	50
Decachlorobiphenyl	680/(3550)	97/93	30-130	≤50	1.9	50
Surrogate- Decachlorobiphenyl-13C12	680/(3550)	97/93	30-130	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acetone	8260(5035)	97/2	14-189	≤40	2.3	50
	8260(5035ext)	97/2	14-189	≤40	230	5000
Benzene (MS)	8260(5035)	97/2	64-144	≤25	0.41	5.0
	8260(5035ext)	97/2	64-144	≤25	41	500
Bromodichloromethane	8260(5035)	97/2	71-140	≤40	0.34	5.0
	8260(5035ext)	97/2	71-140	≤40	34	500
Bromoform	8260(5035)	97/2	59-143	≤40	0.44	5.0
	8260(5035ext)	97/2	59-143	≤40	44	500
Bromomethane (Methyl bromide)	8260(5035)	97/2	35-181	≤65	2.0	10
	8260(5035ext)	97/2	35-181	≤65	200	1000
2-Butanone (MEK)	8260(5035)	97/2	54-166	≤40	2.8	25
	8260(5035ext)	97/2	54-166	≤40	280	2500
Carbon disulfide	8260(5035)	97/2	10-160	≤65	0.29	5.0
	8260(5035ext)	97/2	10-160	≤65	29	500
Carbon tetrachloride	8260(5035)	97/2	65-129	≤40	0.47	5.0
	8260(5035ext)	97/2	65-129	≤40	47	500
Chlorobenzene (MS)	8260(5035)	97/2	56-152	≤25	0.58	5.0
	8260(5035ext)	97/2	56-152	≤25	58	500
Chloroethane	8260(5035)	97/2	65-129	≤40	1.3	10
	8260(5035ext)	97/2	65-129	≤40	130	1000
Chloroform	8260(5035)	97/2	62-117	≤40	0.26	5.0
	8260(5035ext)	97/2	62-117	≤40	26	500
Chloromethane	8260(5035)	97/2	10-186	≤65	1.2	10
	8260(5035ext)	97/2	10-186	≤65	120	1000
Dibromochloromethane	8260(5035)	97/2	73-127	≤40	0.28	5.0
	8260(5035ext)	97/2	73-127	≤40	28	500
1,1-Dichloroethane	8260(5035)	97/2	64-112	≤40	0.28	5.0
	8260(5035ext)	97/2	64-112	≤40	28	500
1,2-Dichloroethane	8260(5035)	97/2	66-128	≤40	1.0	5.0
	8260(5035ext)	97/2	66-128	≤40	100	500
cis-1,2-Dichloroethene	8260(5035)	97/2	64-123	≤40	0.31	5.0
	8260(5035ext)	97/2	64-123	≤40	31	500
trans-1,2-Dichloroethene	8260(5035)	97/2	60-161	≤25	0.57	5.0
	8260(5035ext)	97/2	60-161	≤25	57	500
1,1-Dichloroethene (MS)	8260(5035)	97/2	44-157	≤25	0.77	5.0
	8260(5035ext)	97/2	44-157	≤25	77	500
1,2-Dichloropropane	8260(5035)	97/2	47-146	≤65	1.0	5.0
	8260(5035ext)	97/2	47-146	≤65	100	500

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
cis-1,3-Dichloropropene	8260(5035)	97/2	50-146	≤65	0.49	5.0
	8260(5035ext)	97/2	50-146	≤65	49	500
trans-1,3-Dichloropropene	8260(5035)	97/2	57-143	≤65	0.65	5.0
	8260(5035ext)	97/2	57-143	≤65	65	500
Ethylbenzene	8260(5035)	97/2	60-131	≤40	0.55	5.0
	8260(5035ext)	97/2	60-131	≤40	55	500
2-Hexanone	8260(5035)	97/2	47-145	≤40	3.7	25
	8260(5035ext)	97/2	47-145	≤40	370	2500
4-Methyl-2-pentanone (MIBK)	8260(5035)	97/2	54-167	≤49	3.2	25
	8260(5035ext)	97/2	54-167	≤49	320	2500
Styrene	8260(5035)	97/2	56-121	≤40	0.43	5.0
	8260(5035ext)	97/2	56-121	≤40	43	500
1,1,2,2-Tetrachloroethane	8260(5035)	97/2	36-157	≤40	0.80	5.0
	8260(5035ext)	97/2	36-157	≤40	80	500
Tetrachloroethene	8260(5035)	97/2	61-142	≤40	0.44	5.0
	8260(5035ext)	97/2	61-142	≤40	44	500
Toluene (MS)	8260(5035)	97/2	67-142	≤25	0.48	5.0
	8260(5035ext)	97/2	67-142	≤25	48	500
1,1,1-Trichloroethane	8260(5035)	97/2	11-148	≤40	0.98	5.0
	8260(5035ext)	97/2	11-148	≤40	98	500
1,1,2-Trichloroethane	8260(5035)	97/2	66-123	≤40	0.65	5.0
	8260(5035ext)	97/2	66-123	≤40	65	500

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Trichloroethene (MS)	8260(5035)'	97/2	41-134	≤22	0.72	5.0
	8260(5035ext)	97/2	41-134	≤22	72	500
Vinyl chloride	8260(5035)	97/2	18-169	≤65	0.98	10
	8260(5035ext)	97/2	18-169	≤65	98	1000
Xylenes (total)	8260(5035)	97/2	50-150	≤40	1.5	5.0
	8260(5035ext)	97/2	50-150	≤40	150	500
o-Xylene	8260(5035)	97/2	22-154	≤40	0.50	5.0
	8260(5035ext)	97/2	22-154	≤40	50	500
m+p-Xylene	8260(5035)	97/2	62-123	≤40	1.0	5.0
	8260(5035ext)	97/2	62-123	≤40	100	500
Surrogate - Toluene-d8	8260(5035)	97/2	58-148	NA	NA	NA
	8260(5035ext)	97/2	58-148	NA	NA	NA
Surrogate - p-Bromofluorobenzene	8260(5035)	97/2	70-136	NA	NA	NA
	8260(5035ext)	97/2	70-136	NA	NA	NA
Surrogate -Dibromofluoromethane	8260(5035)	97/2	66-148	NA	NA	NA
	8260(5035ext)	97/2	66-148	NA	NA	NA
Surrogate - 1,2-Dichloroethane-d4	8260(5035)	97/2	38-151	NA	NA	NA
	8260(5035ext)	97/2	38-151	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	8260(5035)	97/2	58-148	NA	NA	NA
	8260(5035ext)	97/2	58-148	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL-A (ug/kg)
Acenaphthene (MS)	8270(3550)	97/2	28-102	≤25	13	330
Acenaphthylene	8270(3550)	97/2	54-140	≤25	13	330
Anthracene	8270(3550)	97/2	39-106	≤30	12	330
Benzo(a)anthracene	8270(3550)	97/2	29-111	≤25	9.6	330
Benzoic acid	8270(3550)	97/2	10-150	≤50	21	1700
Benzo(b)fluoranthene	8270(3550)	97/2	19-126	≤25	17	330
Benzo(k)fluoranthene	8270(3550)	97/2	26-125	≤38	13	330
Benzo(g,h,i)perylene	8270(3550)	97/2	10-121	≤50	19	330
Benzo(a)pyrene	8270(3550)	97/2	34-116	≤29	11	330
Bis(2-chloroethoxy) methane	8270(3550)	97/2	13-108	≤50	30	330
Bis(2-chloroethyl) ether	8270(3550)	97/2	10-130	≤50	9.6	330
Bis(2-chloroisopropyl) ether	8270(3550)	97/2	36-166	≤50	9.9	330
Bis(2-ethylhexyl) phthalate	8270(3550)	97/2	23-124	≤40	46	330
4-Bromophenyl phenyl ether	8270(3550)	97/2	53-127	≤40	11	330
Butyl benzyl phthalate	8270(3550)	97/2	24-120	≤40	30	330
-Chloroaniline	8270(3550)	97/2	10-150	≤50	17	660
4-Chloro-3-methylphenol (MS) (p-Chloro-m-cresol)	8270(3550)	97/2	25-107	≤25	10	330
2-Chloronaphthalene	8270(3550)	97/2	60-118	≤40	11	330
2-Chlorophenol (MS)	8270(3550)	97/2	23-114	≤25	11	330
4-Chlorophenylphenyl ether	8270(3550)	97/2	20-118	≤33	10	330
Chrysene	8270(3550)	97/2	35-130	≤27	12	330
2-Methyl phenol (o-Cresol)	8270(3550)	97/2	14-95	≤50	13	330
3- Methyl phenol (m- Cresol)	8270(3550)	97/2	12-102	≤50	61	330
4-Methyl phenol (p-Cresol)	8270(3550)	97/2	12-102	≤50	61	330
3- and 4-Methyl phenol (m- and p-Cresol)	8270(3550)	97/2	12-102	≤50	61	330
Dibenz(a,h)anthracene	8270(3550)	97/2	40-147	≤28	15	330
Dibenzofuran	8270(3550)	97/2	10-150	≤50	12	330
Di-n-butylphthalate	8270(3550)	97/2	28-125	≤50	15	330
1,2-Dichlorobenzene	8270(3550)	97/2	10-99	≤40	11	330
1,3-Dichlorobenzene	8270(3550)	97/2	10-100	≤42	8.8	330
1,4-Dichlorobenzene (MS)	8270(3550)	97/2	10-125	≤40	11	330
3,3'-Dichlorobenzidine	8270(3550)	97/2	10-189	≤100	16	660
2,4-Dichlorophenol	8270(3550)	97/2	16-104	≤40	10	330
Diethylphthalate	8270(3550)	97/2	31-113	≤40	15	330
2,4-Dimethylphenol	8270(3550)	97/2	10-95	≤25	10	330
Dimethylphthalate	8270(3550)	97/2	20-116	≤40	11	330
6-Dinitro-2-methylphenol	8270(3550)	97/2	43-135	≤93	29	1700
4-Dinitrophenol	8270(3550)	97/2	10-126	≤87	22	1700

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,4-Dinitrotoluene (MS)	8270(3550)	97/2	26-107	≤30	13	330
2,6-Dinitrotoluene	8270(3550)	97/2	38-107	≤40	16	330
Di-n-octylphthalate	8270(3550)	97/2	22-135	≤50	19	330
Fluoranthene	8270(3550)	97/2	23-121	≤25	11	330
Fluorene	8270(3550)	97/2	32-107	≤40	12	330
Hexachlorobenzene	8270(3550)	97/2	44-105	≤40	10	330
Hexachlorobutadiene	8270(3550)	97/2	10-112	≤40	11	330
Hexachlorocyclopentadiene	8270(3550)	97/2	D-132	≤50	18	330
Hexachloroethane	8270(3550)	97/2	16-91	≤40	13	330
Indeno(1,2,3-cd)pyrene	8270(3550)	97/2	18-157	≤83	20	330
Isophorone	8270(3550)	97/2	17-107	≤60	12	330
2-Methylnaphthalene	8270(3550)	97/2	19-105	≤50	9.2	330
Naphthalene	8270(3550)	97/2	53-125	≤25	11	330
2-Nitroaniline	8270(3550)	97/2	22-110	≤50	14	1700
3-Nitroaniline	8270(3550)	97/2	10-117	≤50	11	1700
4-Nitroaniline	8270(3550)	97/2	10-136	≤50	13	1700
Nitrobenzene	8270(3550)	97/2	9-104	≤40	13	330
2-Nitrophenol	8270(3550)	97/2	10-101	≤40	8.8	330
4-Nitrophenol (MS)	8270(3550)	97/2	10-117	≤45	30	1700
N-Nitrosodiphenylamine/ Diphenylamine	8270(3550)	97/2	40-122	≤50	98	330
N-Nitroso-di-n-propylamine MS)	8270(3550)	97/2	11-117	≤35	14	330
Pentachlorophenol (MS)	8270(3550)	97/2	10-120	≤44	28	1700
Phenanthrene	8270(3550)	97/2	38-113	≤25	12	330
Phenol (MS)	8270(3550)	97/2	17-103	≤25	19	330
Pyrene (MS)	8270(3550)	97/2	18-136	≤25	11	330
1,2,4-Trichlorobenzene (MS)	8270(3550)	97/2	17-105	≤28	11	330
2,4,5-Trichlorophenol	8270(3550)	97/2	39-123	≤27	13	330
2,4,6-Trichlorophenol	8270(3550)	97/2	20-116	≤40	9.2	330
Surrogate - Nitrobenzene-d5	8270(3550)	97/2	12-125	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	8270(3550)	97/2	24-118	NA	NA	NA
Surrogate - p-Terphenyl-d14	8270(3550)	97/2	18-153	NA	NA	NA
Surrogate - Phenol-d5	8270(3550)	97/2	10-142	NA	NA	NA
Surrogate - 2-Fluorophenol	8270(3550)	97/2	10-118	NA	NA	NA
Surrogate - 2,4,6-Tribromophenol	8270(3550)	97/2	14-121	NA	NA	NA

SEVERN TRENT SERVICES

Biological Tissues Parameters

PARAMETER	METHOD	ACC (%REC)	PREC (%RPD)	MDL (ug/kg)	RL (ug/kg)
Indeno(1,2,3-cd)pyrene	8270(3550)	18-157	<=83	60	1000
Isophorone	8270(3550)	17-107	<=60	36	1000
2-Methylnaphthalene	8270(3550)	19-105	<=50	28	1000
Naphthalene	8270(3550)	53-125	<=25	33	1000
2-Nitroaniline	8270(3550)	22-110	<=50	42	5000
3-Nitroaniline	8270(3550)	10-117	<=50	33	5000
4-Nitroaniline	8270(3550)	10-136	<=50	39	5000
Nitrobenzene	8270(3550)	9-104	<=40	39	1000
2-Nitrophenol	8270(3550)	10-101	<=40	26	1000
4-Nitrophenol (MS)	8270(3550)	10-117	<=45	90	5000
N-Nitrosodiphenylamine	8270(3550)	40-122	<=50	290	1000
N-Nitroso-di-n-propylamine MS)	8270(3550)	11-117	<=35	42	1000
Pentachlorophenol (MS)	8270(3550)	10-120	<=44	84	5000
Phenanthrene	8270(3550)	38-113	<=25	36	1000
Phenol (MS)	8270(3550)	17-103	<=25	57	1000
Pyrene (MS)	8270(3550)	18-136	<=25	33	1000
1,2,4-Trichlorobenzene (MS)	8270(3550)	17-105	<=28	33	1000
2,4,5-Trichlorophenol	8270(3550)	39-123	<=27	39	1000
2,4,6-Trichlorophenol	8270(3550)	20-116	<=40	28	1000
Surrogates					
Nitrobenzene-d5	8270(3550)	12-125	NA	NA	NA
2-Fluorobiphenyl	8270(3550)	24-118	NA	NA	NA
p-Terphenyl-d14	8270(3550)	18-153	NA	NA	NA
Phenol-d5	8270(3550)	10-142	NA	NA	NA
2-Fluorophenol	8270(3550)	10-118	NA	NA	NA
2,4,6-Tribromophenol	8270(3550)	14-121	NA	NA	NA

RL based on an extraction weight of 10g and and a final extract volume of 1.0mL.

Herbicides by GC/Electron Capture					
2,4-D (MS)	8151	19-153	<=47	4.8	25
Dalapon	8151	10-170	<=40	69	6000
2,4-DB	8151	20-160	<=40	19	25
Dicamba	8151	20-160	<=40	3.6	60
Dichlorprop	8151	30-170	<=40	8.1	300
Dinoseb	8151	10-130	<=50	25	300
MCPA	8151	10-130	<=50	2220	6000
MCPP	8151	10-130	<=50	1200	6000
2,4,5-T (MS)	8151	14-143	<=59	3	25
2,4,5-TP (Silvex) (MS)	8151	27-120	<=51	2.7	25
Surrogate					
2,4-Dichlorophenyl acetic acid (DCAA)	8151	30-189	NA	NA	NA

RL based on an extraction weight of 10g and and a final extract volume of 10mL.

SEVERN TRENT SERVICES

Biological Tissues Parameters

PARAMETER	METHOD	ACC (%REC)	PREC (%RPD)	MDL (ug/kg)	RL (ug/kg)
Semivolatiles by GC/MS					
Acenaphthene (MS)	8270(3550)	28-102	<=25	39	1000
Acenaphthylene	8270(3550)	54-140	<=25	39	1000
Anthracene	8270(3550)	39-106	<=30	36	1000
Benzo(a)anthracene	8270(3550)	29-111	<=25	29	1000
Benzoic acid	8270(3550)	10-150	<=50	63	5000
Benzo(b)fluoranthene	8270(3550)	19-126	<=25	51	1000
Benzo(k)fluoranthene	8270(3550)	26-125	<=38	39	1000
Benzo(g,h,i)perylene	8270(3550)	10-121	<=50	57	1000
Benzo(a)pyrene	8270(3550)	34-116	<=29	33	1000
Bis(2-chloroethoxy) methane	8270(3550)	13-108	<=50	90	1000
Bis(2-chloroethyl) ether	8270(3550)	10-130	<=50	29	1000
Bis(2-chloroisopropyl) ether	8270(3550)	36-166	<=50	30	1000
Bis(2-ethylhexyl) phthalate	8270(3550)	23-124	<=40	140	1000
4-Bromophenyl phenyl ether	8270(3550)	53-127	<=40	33	1000
Butyl benzyl phthalate	8270(3550)	24-120	<=40	90	1000
4-Chloroaniline	8270(3550)	10-150	<=50	51	2000
4-Chloro-3-methylphenol (MS) (p Chloro-m-cresol)	8270(3550)	25-107	<=25	30	1000
2-Chloronaphthalene	8270(3550)	60-118	<=40	33	1000
2-Chlorophenol (MS)	8270(3550)	23-114	<=25	33	1000
4-Chlorophenylphenyl ether	8270(3550)	20-118	<=33	30	1000
Chrysene	8270(3550)	35-130	<=27	36	1000
2-Methyl phenol (o-Cresol)	8270(3550)	14-95	<=50	39	1000
3- and 4-Methyl phenol (m and p-Cresol)	8270(3550)	12-102	<=50	180	1000
Dibenz(a,h)anthracene	8270(3550)	40-147	<=28	45	1000
Dibenzofuran	8270(3550)	10-150	<=50	36	1000
Di-n-butylphthalate	8270(3550)	28-125	<=50	45	1000
1,2-Dichlorobenzene	8270(3550)	10-99	<=40	33	1000
1,3-Dichlorobenzene	8270(3550)	10-100	<=42	26	1000
1,4-Dichlorobenzene (MS)	8270(3550)	10-125	<=40	33	1000
3,3'-Dichlorobenzidine	8270(3550)	10-189	<=100	48	2000
2,4-Dichlorophenol	8270(3550)	16-104	<=40	30	1000
Diethylphthalate	8270(3550)	31-113	<=40	45	1000
2,4-Dimethylphenol	8270(3550)	34973	<=25	30	1000
Dimethylphthalate	8270(3550)	20-116	<=40	33	1000
4,6-Dinitro-2-methylphenol	8270(3550)	43-135	<=93	87	5000
2,4-Dinitrophenol	8270(3550)	10-126	<=87	66	5000
2,4-Dinitrotoluene (MS)	8270(3550)	26-107	<=30	39	1000
2,6-Dinitrotoluene	8270(3550)	38-107	<=40	48	1000
Di-n-octylphthalate	8270(3550)	22-135	<=50	57	1000
Fluoranthene	8270(3550)	23-121	<=25	33	1000
Fluorene	8270(3550)	32-107	<=40	36	1000
Hexachlorobenzene	8270(3550)	44-105	<=40	30	1000
Hexachlorobutadiene	8270(3550)	10-112	<=40	33	1000
Hexachlorocyclopentadiene	8270(3550)	D-132	<=50	54	1000
Hexachloroethane	8270(3550)	16-91	<=40	39	1000

B-2 Volatile Compounds by GC/MS (EPA 8260B)

VOLATILE COMPOUNDS BY GC/MS (EPA 8260B)

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Approved by:

C. Wayne Potter — Nov 12, 1999

Title: STL-SL QA Manager Date

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures that can be used to determine the concentration of volatile organic compounds (VOC) in water, wastewater, soils/sediments, wastes, oils, sludges, and solids. The attached quantitation report lists the target compounds, an example of the retention time order of each target compound, the quantitation and confirmation ions of the target compounds, and internal standard assignments.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria for each target compound are listed in Section 5 of the current revisions of the STL Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF METHOD

2.1 Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 Aqueous samples may be purged at ambient conditions (recommended) or at 40C (optional). Five to twenty-five milliliter aliquots of the sample may be purged. The calibration standards and the associated QC must be analyzed under the same conditions and volume.

2.3 Low-level (nominally <1mg/kg) soil samples are purged at 40C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards are purged under the same conditions.

2.4 High level soils (nominally >1mg/kg) and waste samples are extracted with methanol-1mL of methanol per gram of sample. An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

2.5 This method is based on the guidance in SW-846 Methods 8260B and 5035.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedure that you do not understand or that will put yourself or others in a potentially hazardous situation.

3.2 Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, eye protection, or other equipment should be used. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

3.4 The exit vent of the split injector must have a carbon trap in-line to collect the volatile compounds that are vented during the injection of the sample. The traps should be changed a minimum of every three months and disposed of in accordance with STL-SL SOP CA70: *Waste Management.*

4.0 INTERFERENCES

4.1 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.

4.2 The volatiles lab must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator blanks should be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in STL-SL SOP AN70: *Segregation of Low and High Concentration Volatile and Semivolatile Samples.*

4.3 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-terachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the lab can clearly demonstrate matrix problems. Mass 58 is recommended for quantitation of acetone due to the elution of a hydrocarbon at the same retention time.

4.4 The analysis of highly contaminated samples (>1mg/L or >1mg/kg) can affect succeeding analyses. Carry-over can occur when low concentration samples are analyzed after high concentration samples. Trap replacement and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when carryover is suspected to demonstrate that the system is free from contamination.

4.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If contamination of the fittings is suspected, the fittings may be heated at 105 C for one hour or replaced.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

5.1 Liquid samples are collected with no headspace in 40mL vials equipped with Teflon-lined caps. The samples are acidified at the time of collection with about 0.10mL of concentrated HCl per 40mL of sample. The acid prevents the biological degradation of the aromatic compounds and prevents the dehydrohalogenation of some of the chlorinated alkanes. The sample must be iced at the time of collection and refrigerated at 4C (less than 6C with no frozen samples) in the lab until analysis.

Check each sample vial at the time of receipt for the presence of "bubbles". If the bubbles are less than 3mm in diameter, the vial is acceptable. If the bubble is greater than 3mm, use another vial. Notify the department supervisor or project manager if there are no acceptable vials for analysis.

A "sacrificial" vial or the vial used for screening analysis is used to check the sample pH. If the sample pH is greater than 2, notify the department supervisor or project manager. If directed by supervisor or project manager, hydrochloric acid may be added through the septum to bring the pH <2. Do not add more than 400uL (0.40mL) of 1:1 HCl to a VOC vial. If pH cannot be adjusted to <=2 without destroying the integrity of the sample, the sample must be analyzed within 7 days of collection.

The holding time for samples preserved with HCl is 14 days for all target compounds. The holding time for un-preserved samples is 7 days.

5.2 Soils: Soils are routinely collected in duplicate in Encore samplers. A "bulk" sample is also routinely collected in a 125-mL jar fitted with a Teflon-lined cap. The bulk sample can be used for the methanol extraction if the concentration of the sample collected in the Encore exceeds the working range of the analytical system.

Soils collected in Encore samplers must be analyzed within 48 hours of collection or must be transferred within 48 hours of collection to sealed vials containing sodium bisulfate solution or methanol. If the sample contains high levels of carbonates, the sample is preserved with water and frozen until the time of analysis. The procedure for preparing soil samples is given in Section 9.2.

The hold time of the preserved sample is 14 days from the date of collection. The hold time for frozen samples is 14 days from the date of collection.

5.3 High level soil and waste samples are collected in glass containers (usually 125-mL clear glass) equipped with Teflon-lined caps. Soil samples may also be submitted as core samples contained in Encore samplers, metal or plastic "tubes", or in 40-mL VOA vials. The samples are iced at the time of collection and stored at 4C (less than 6C with no frozen samples). The holding time for soil and waste samples subjected to methanol extraction is14 days from date of collection; that is, the extraction and analysis must be completed within 14 days of collection.

5.4 TCLP leachate samples are collected with no headspace in Tedlar bags or syringes. The leachate samples are acidified at the time of collection (after the leaching procedure) with about 0.10mL of concentrated HCl per 40mL of sample and stored at 4C (less than 6C with no frozen samples) from the time leaching is completed until the analysis. The acidified leachate sample must be analyzed within 14 days of the leaching procedure. If the sample is not acidified, the leachate must be analyzed within 7 days of the leaching procedure.

NOTE: Samples that are suspected of having very high concentrations of VOC should be segregated from the "routine" samples and stored in a manner that will minimize sample and laboratory contamination. See STL-SL SOP AN70. If possible, keep the field QC in the same storage refrigerator as the samples.

6.0 APPARATUS AND MATERIALS

The apparatus and materials listed in this section may vary from lab to lab. The items listed are to give guidance and to provide a general overview of the equipment employed in this analysis.

6.1 Mass spectrometer: equipped with a capillary direct interface and a split/splitless injector or molecular jet separator

6.2 Gas chromatograph, compatible with the MS and purge and trap systems. If the GC is equipped with an injector that is operated in the split mode, the exit vent must have a carbon trap in-line to collect the volatile compounds that are vented during the transfer from the purge and trap device. The carbon traps should be changed a minimum of every three months.

6.3 Purge and trap device Tekmar 3000 Liquid Concentrator or equivalent

6.4 Supelco Vocarb 3000 trap or equivalent, Other traps may be used as long as the target compounds can be detected at the required quantitation limit.

6.5 Archon soil analyzer for low level soils, compatible with Tekmar purge and trap instruments. The instrument must be capable of automatically adding water and internal standard to the container while maintaining the septum seal, heating the sample to 40C, and spinning the stir bar to mix the sample during the purging step.

6.5 Data System compatible with the analytical system

6.6 Microsyringes: 10ul, 25ul, 50ul, 100ul, 250ul, 500ul, 2.5mL

6.7 Gastight syringe: 5mL, 25mL with luerlock tip

6.8 Volumetric flasks: 1.0mL, 10mL, 100mL

6.9 Recommended Columns

J&W DB-624: 60m x 0.32mm ID, 1.8um film
J&W DB-624: 20m x 0.18mm ID, 1.8um film

7.0 REAGENTS

Reagents must be tracked in accordance with STL-SL SOP AN44: *Reagent Traceability.*

7.1 Reagent water-free of volatile contaminants (obtained by purging with inert gas or carbon filtration)

7.2 Methanol-Burdich and Jackson, Purge and Trap grade

7.3 Sodium bisulfate-reagent grade. This salt is hydroscopic and should be stored in a dessicator.

7.4 Soil preservation solution- Slowly add, while stirring, 200g of sodium bisulfate to a 1.0-L volumetric containing about 700mL of reagent water. After the salt has dissolved, dilute to volume with reagent water, transfer to a storage container, and store the solution in an area free from VOC-especially water-soluble solvents such as acetone. The reagent should be tested prior to use by the analysis of a blank containing 5mL of the solution. The reagent is acceptable if it meets the same criteria as a method blank.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all stock solutions or neat compounds. All preparation steps must be in accordance with STL-SL SOP AN41: *Standard Material Traceability.*

8.1 Preparation of Stock Standards from Neat Compounds

The lab should attempt to obtain a certified primary standard or secondary standard before preparing stock standards from neat materials. If primary stock standards must be prepared in-house, the target concentration range is from 2000ug/mL to 10000ug/mL. SL-SOP AN43: *Standard Preparation* gives the general instructions for the preparation of the stock solutions from neat materials.

8.2 Preparation of the Working Standard from Stock Standards

The working standard is prepared from the primary stock standards that are either prepared from neat compounds or purchased as certified solutions. The working standard contains one or more of the target compounds at a concentration suitable for preparing the calibration standards, generally 10-200ug/mL. A known volume of the working standard is then added to a known volume of reagent water to make the calibration standard.

The standards and standard concentrations listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document the standard preparation procedures in the standard traceability log.

8.3 Preparation of the Calibration Standards from the Working Standards

The calibration standards are the standards that are analyzed on the instrument. The calibration standard is made by adding a known volume of the working standard to a known volume of reagent water. The instrument must be calibrated using a minimum of five calibration standards. The lowest level standard must be at the reporting limit and the rest of the standards will define the working range of the analytical system.

8.3.1 Add 5.0mL of reagent water to a 5mL-glass syringe or 25ml of reagent water to a 25-ml glass syringe.

8.3.2 Add a known volume of the working standard to 5.0mL or 25ml of reagent water.

NOTE: The calibration standards for the low level soils are prepared using the same procedures as for the 5mL water purge except that the standards are purged at 40C. The lab has the option of using blank sand in the calibration standards.

The calibration standards listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document these standard preparation procedures in the standard traceablity log. A 5mL-purge volume may be used for low level (nominal RL of 1ug/L) if the instrument has sufficient sensitivity to detect the targets and the calibration criteria is met.

9.0 SAMPLE PREPARATION

Composite samples can be prepared using the guidance provided in STL-SL-SOP AN70.

9.1 Aqueous samples are analyzed directly by purge and trap/GC-MS. No sample preparation is necessary except to homogenize the sample prior to subsampling. The pH of liquid samples is checked and recorded prior to analysis to determine if the sample has been properly preserved.

9.2 Preparation of Soil Samples (5035)

9.2.1 Remove the Encore samples and the bulk sample from the storage area.

9.2.2 Test an aliquot of the bulk sample for the presence of carbonates.

- Transfer 5g of sample from the bulk sample to a 40mL vial..
- Add 5ml of the sodium bisulfate solution and shake the vial .
- If the sample exhibits effervescence, the Encore samples should be preserved as described above using 5mL of volatile-free water in place of the sodium bisulfate solution and placed in a freezer at –10C. The analytical hold time for frozen samples is 14 days from collection.
- If no effervescence is noted, the Encore samples may be preserved with 5mL soil preservation solution.

9.2.3 Add a stir bar to a vial and weigh the vial and record its tare weight(or tare the vial and stir bar weight by pressing the autotare button).

9.2.4 Transfer the sample from the Encore sampler to the tared vial and record the weight of the sample log.

If the sample effervesced during the carbonate test (9.2.2), add 5.0mL of reagent water and freeze at –10C. The hold time is 14 days from collection.

If not, add 5.0mL of the soil preservation solution, seal the vial, and store the sample at 4C until the time of analysis. The preserved sample must be analyzed within 14 days of collection.

NOTE: A preparation blank is prepared when Encore samples are transferred. The preparation blank contains the same reagents as the samples-either 5mL of reagent water or 5mL of soil preservation solution.

9.3 A methanol extraction is prepared when the concentration of the target compounds (by direct purge) exceeds the working range of the calibration curve. The bulk sample, collected in the 125-mL sample container, can be used to prepare the methanol extraction. Carry out the preparation quickly to minimize the loss of volatiles.

-Mix the sample with a stainless steel spatula and transfer 10g (+/- 0.5g) to a glass vial.

-Add 8uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 10g and 100% percent solids, is calculated:

$$Ct(ug/kg, dw) = \frac{0.008mL \otimes 2500ug/mL}{0.010g \otimes solids} = 2000ug/kg, dw$$

-Shake the sample for two minutes. Allow the solvent to separate from the solids portion of the sample and transfer a 1-2mL aliquot of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

-For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The lab control standard is prepared by adding 8uL of the surrogate spiking solution and 8uL of the matrix spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The matrix spikes are prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) and 8uL of the matrix spiking solution (2500ug/mL) to 10-g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for two minutes. Analyze 125uL of the extract or a smaller volume if the VOC concentration is high.

-Add 125uL of the extract (or a smaller volume if the VOC concentration exceeds the linear range of the system with 125uL) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

9.4 Methanol Extraction for Wastes

Carry out the preparation quickly to minimize the loss of volatiles.

9.4.1 Mix the sample with a stainless steel spatula and transfer 1g (+/- 0.2g) to a glass vial.

9.4.2 Add 10uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. If the sample is completely soluble in the methanol, dilute to a final volume of 10mL. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 1.0g , is calculated:

$$Ct(ug/kg) = \frac{0.010mL \otimes 2500ug/mL}{0.0010g \otimes solids} = 25000ug/kg$$

9.4.2 Shake the sample for one minute. Allow the solvent to separate from the solids portion of the sample and transfer 1mL to 2mL of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) to 10mL of purge and trap grade methanol. Assume a sample weight of 1.0g. Analyze 100uL of the extract.

The lab control standard is prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 5.0mL of purge and trap grade methanol. Assume a sample weight of 5.0g. Analyze 100uL of the extract.

The matrix spikes are prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 1g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for one minute.

Add 100uL of the extract (or a smaller volume) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

NOTE: Waste samples may require significant dilution prior to analysis.

10.0 PROCEDURE

The following instrument conditions are recommended. The actual conditions may vary due to differences in instrumentation. The lab must document the instrument conditions in the maintenance log, the data system, or on the analysis log.

10.1 Instrument Conditions

10.1.1 GC Conditions

GC conditions may vary according to the environment and condition of each instrument. The lab must document the instrument conditions to assure consistent results and to aid in trouble-shooting the analytical system. Each lab is responsible for assuring that the conditions necessary to achieve adequate separation and sensitivity of the target analytes are maintained.

10.1.1.1 Example GC temperature program

Initial column temperature: 35 C for 3 minutes
Column temperature program 1: 20C per minute
Intermediate column temperature: 70C for 4 minutes
Column temperature program 2: 10C per minute
Final column temperature: 200C for 5.25 minutes

10.1.1.2 Column flow: Approximately 5-10mL/minute helium with a make-up of 20-25mL/minute helium. Total flow into the jet separator should be about 30mL/minute. The vacuum gauge on the jet separator will read about 0.5Torr.

If no jet separator is used and the column is plumbed directly into the source, the column flow should be adjusted to 0.5-1.0ml/min and a split ratio (desorb to column flow) of about 40:1 established. Smaller bore capillary columns (0.18 to 0.32mm) are required if the column is plumbed directly into the source

10.1.1.3 Mass Spectrometer and interface parameters

Jet separator temperature: 240C
Mass spectrometer interface: 240C
Mass spectrometer source temperature: factory set at 300C
range: 35-300amu, with a minimum scan cycle of 1 scan per second

10.1.2 Purge and Trap Conditions
The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

10.1.2.1 "Three ring trap"-charcoal, Tenax, silica gel
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 180C
Bake time: 8 minutes at 225C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

10.1.2.1 VOCARB 3000 trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 225C
Bake time: 8 minutes at 250C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water-soluble targets-ketones, ethers, bromoform-may be low and the reporting limit may not be achieved on a routine basis.

10.2 BFB Tune Check

10.2.1 Fifty nanograms of 4-BFB must be analyzed at the beginning of each 12-hour clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage. If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

10.2.2 Evaluation of the 4-BFB peak.

10.2.2.1 The chromatogram should exhibit acceptable baseline behavior and the 4-BFB peak should be symmetrical. A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.

10.2.2.2 The spectrum of the 4-BFB must meet the criteria listed in the attached SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 5 scans from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.

10.2.2.3 The following records must be kept for each 4-BFB analysis that meets the criteria:
- the date, time, and data file of the analysis
- a spectrum of the scan or averaged scans
- a tabulation of the ion abundances of the scan

10.2.2.4 The 4-BFB analysis should be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window should be established for each instrument. Response outside of this window suggests instrumental problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , or other anomalies.

10.2.2.5 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

10.3 Initial Calibration

After the 4-BFB criteria has been met, the initial calibration standards are analyzed. Prepare the initial calibration standards according to the example recipes in the SOP appendices or lab-specific recipe. The lab must document the "recipe" used to prepare the calibration standards. The lowest level calibration standard must be at or below the routine RL and the other calibration standards will define the working range of the system.

10.3.1 Remove the plunger from the syringe and fill the barrel to overflowing with reagent water (syringe valve in the "red" position).

10.3.2 Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL or 25mL)

10.3.3 Briefly remove the syringe valve and inject the standards and internal standards into the syringe.

NOTE: Use the internal standard (IST) mix when preparing the calibration standards for analysis. The surrogates are already included in the standard mixes.

10.3.4 Load the standard(s) onto the purge and trap device and begin the analysis. All pertinent information concerning the standards must be recorded on the analysis log. The standards must be clearly identified and traceable to the preparation steps.

NOTE: The standards for low-level soil samples are prepared in the same manner as the 5mL standards. The standards for the low-level soils are purged at 40C. The lab has the option of using blank sand or soil in the calibration standards and the blank in the low level soil analysis.

10.3.5 After the acquisition has taken place, evaluate the calibration standards to ensure that each target compound, surrogate, and internal standard has been correctly identified. The analyst must be careful to complete this step before proceeding.

10.3.6 After each target compound, surrogate, and internal standard has been correctly identified, the relative response factor for each target compound and surrogate is calculated using the data system or using a PC spreadsheet as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where
Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (see the attached quantitation report for a list of the compounds that are associated with the various internal standards)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug on-column)
Cis = concentration or mass on-column of the internal standard (ug/L or ug/kg OR ng or ug on-column)

The average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$RRFavg = \frac{RRF1 + RRF2 + ... + RRFn}{n}$$

where n = number of calibration levels

Calculate the standard deviation (SD) for the target compounds and surrogates at all calibration levels:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RFi - RFavg)^2}{n-1}}$$

where
Rfi = response factor of a target compound in the individual calibration level
Rfavg = average response factor
n= number of calibration levels

10.3.7 Calculate the relative standard deviation (% RSD) of the calibration levels for each target:

$$\%\,RSD = \frac{standard\ deviation}{RRFavg} \otimes 100$$

10.3.8 The results of the initial calibration are evaluated against the Calibration Check Compound (CCC) criteria and the System Performance Check Compound (SPCC) criteria, which are listed below. The CCC and SPCC criteria must be met before samples can be analyzed.

Calibration Check Compounds – CCC Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
<=30% RSD	<=20% difference from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

10.3.9 After the initial calibration criteria (CCC and SPCC) have been met, each target is evaluated for linearity.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug)
Cis = concentration of the internal standard (ug/L or ug/kg OR ng or ug)

If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples.. Regression curves may be forced through zero but it is recommended that the curve be evaluated without forcing through zero first and then with the curve forced through the origin. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest o highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3.10 After the initial calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP or other specified QAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compounds must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of samples can take place.

If the method blank repeatedly fails to meet the criteria, contact the immediate supervisor to determine the cause of the problem and to determine a course of action. This action may include re-cleaning the sparging tubes (with soap, hot water, and methanol), purging the effected autosampler ports with heated methanol, flushing the purge and trap ALS concentrator with methanol, replacing the trap, changing the transfer line, and changing the column. A method blank is then analyzed after taking the corrective action to demonstrate that the contamination has been eliminated. Once the system is determined to be free from contamination, sample analysis may begin. Method blanks may be required after the analysis of samples that contain very high levels of VOC.

10.4 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of 50ng of 4-BFB. This criteria must be met before the analysis of the calibration check standards can take place.

10.4.1 After the tune criteria has been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a nominal concentration of 50ug/L-kg for 5ml/5g samples and 10ug/L for 25mL with ketones and poor purgeables at higher concentrations. The CCC and SPCC criteria (Section 10.3.8) must be met before the analysis of the method blank and samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration
Cccv = measured concentration using the selected quantitation method

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If this criteria is not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.4.2 The calibration standard (CCV) must also be evaluated for internal standard retention time and response.

If the retention time of any internal standard changes by more than 30 seconds from the retention times of the internal standards in the initial calibration, the analytical system must be inspected for problems and corrective action instituted.

If the extracted ion current profile (EICP) area for any of the internal standards changes by more than a factor of two (-50% to +100%) from the last calibration check standard, the analytical system must be inspected for problems and corrective action instituted. If the CCV is the first one after the initial calibration, compare the ISTD response to the corresponding level in the ICAL.

10.4.3 After the continuing calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compound must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of client samples can take place.

10.5 Aqueous Sample Analysis-5.0mL to 25mL

The analyst must use the same volume as was used for the calibration standards-if a 5mL sample is used, it must be quanted off of the 5mL calibration curve; if a 25ml sample is used, it must be quanted off of the 25mL calibration curve. Samples are analyzed only after the tune criteria, the calibration (initial or continuing) criteria has been met, and the method blank criteria has been met. See the SOP Summary for the analytical sequence.

10.5.1 Remove the samples to be analyzed from the refrigerator and allow the samples to come to ambient temperature.

10.5.2 Put on a pair of gloves before transferring the sample from the vial to the syringe. The sample is most likely preserved with acid or may contain toxic or hazardous chemicals or biologically active components that may cause skin irritations. ***Gloves must be worn when handling samples.***

10.5.3 Mix the contents of the vial by inverting the vial several times. Check to see if there are air bubbles present in the sample. If air bubbles are present, use another vial if available. Make a note on the analysis log if the sample used contained bubbles and notify the supervisor and/or the project manager.

10.5.5 Remove the plunger from the glass syringe. Attach a syringe valve to the syringe Luer-tip to prevent sample from spilling out of the syringe when sample is added.

10.5.5 Open the vial of the well-mixed sample and gently pour the sample into the syringe barrel. The sample should fill the barrel of the syringe and overflow to allow trapped air bubbles to escape.

10.5.6 Replace the plunger into the syringe barrel. Try not to let air bubbles get into the barrel. If air bubbles are present, turn the syringe up, open the syringe valve , and expel the air while adjusting the volume to 5.0mL or 25mL. If no air bubbles were trapped, adjust the syringe to volume.

NOTE: For TCLP leachate samples, use 1.25mL of sample (1:4 dilution).

10.5.7 Open the syringe valve and inject the internal standard/surrogate (ISSU) mix into the sample.

10.5.8 Transfer the sample from the syringe to the purge and trap device. Record all of the sample identification information on the analysis log. Check the pH of the sample with pH paper and record the pH on the instrument log or other appropriate log.

10.5.9 Analyze the samples using the purge and trap and GC/MS conditions used for the initial and continuing calibration standards.

10.5.10 Determine the concentration of the samples and QC items. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

A dilution is made when a volume of the sample is mixed with the reagent water to a final volume of 5.0mL or 25ml,depending on which curve is being used. The dilution factor is calculated by dividing the volume of sample into the volume used for the calibration curve.

$$DF = \frac{final\ volume\ of\ dilution(mL)}{volume\ of\ sample\ used(mL)}$$

For example, if 1.0mL of sample is diluted to final volume of 5.0mL, the dilution factor is 5. (5.0/1.0 = 5). If 1.0mL of sample is diluted to a final volume of 25mL, the dilution factor is 25 (25/1=25).

The following table gives some dilution factors:

Volume of Sample (mL)	Volume of Reagent Water (mL)	Final Volume (mL)	Dilution factor
5.0	0	5.0	1
2.5	2.5	5.0	2
1.0	4.0	5.0	5
0.5	4.5	5.0	10
0.10	4.9	5.0	50
25.0	0	25.0	1
5.0	20.0	25.0	5
2.5	22.5	25.0	10
1.0	24.0	25.0	25
0.50	24.5	25.0	50
0.10	24.9	25.0	250

NOTE: The same volume of internal standard/surrogate mix (ISSU) is added to the dilution as was added to the undiluted sample.

10.6 Low Level Soil Samples by Heated Purge and Trap (Method 5035)

The soil analytical system is calibrated using the same concentrations as the 5mL purge. The tune, initial and continuing calibration criteria, and the method blank criteria must be met before samples are analyzed. Standards and QC items must be analyzed under the same heated purge and trap conditions.

Remove the samples to be analyzed (Section 9.2) from the refrigerator or freezer and allow the sample to come to ambient temperature. Inspect the vial for cracks or obvious breaches in the septum. Load the samples on to the soil-purging unit and analyze according to the sequence described in Appendix B.

Liquid field QC for soils (trip blank, field blank, etc.) should be analyzed with the associated soil samples, using the same preparation and analytical procedures, including the heated purge. Report the results for liquid trip blanks as ug/L.

10.7 Analysis of Methanol Extracts of Soils and Wastes

The methanol extraction is used when the concentration of one or more target compounds exceeds the linear range of the low-level purge technique (>1000ug/kg). or if the concentration of VOC in the soil or waste samples is high. Samples are analyzed only after the 4-BFB criteria, the calibration criteria (initial and continuing), and the method blank criteria has been met. Medium level soil extracts are quanted using the ambient purge calibration curve. Sample preparation steps are included in Section 9.

10.7.1 Remove the plunger from the 5.0-mL syringe and fill the barrel to overflowing with reagent water(syringe valve in the "red" position). Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL)

10.7.2 Briefly remove the syringe valve and inject the sample extract and 5uL of the internal standard (IST) solution into the syringe. Use 125ul of the extract for soils and 100uL of the extract for wastes. Smaller aliquots are used if the concentration of target analytes exceed the working range of the system.

NOTE: Use the internal standard (IST) mix when preparing the medium level samples. Recall that the surrogates have already been added to the sample during the methanol extraction step (Section 9).

10.7.3 Load the sample on to the purge and trap device and begin the analysis. All pertinent information concerning the samples must be recorded on the analysis log. The samples must be clearly identified and traceable to the extraction log. These conditions must be the same as was used for the initial and continuing calibration standards-ambient purge for aqueous samples.

10.7.4 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, a smaller aliquot of the methanol extract is reanalyzed to bring the highest target within the upper half of the calibration curve. Follow the guidelines in Section 10.4.10 for reporting dilutions.

NOTE: It is possible to dilute the surrogates in the sample extract below the linear range of the calibration curve. The minimum extract aliquot that can be used to provide a quantifiable result for the surrogates and matrix spikes is 0.0025mL (2.5uL).

SOIL: 10g to 10mL MeOH	WASTES: 1g to 10mL MeOH	Surrogates- Theoretical ng on-column
125uL(0.125mL)	100uL (0.100mL)	250
62.5uL(0.0625mL)	50uL(0.050mL)	125
25uL(0.025mL)	25uL(0.020mL)	50
12.5uL(0.0125mL)	10ul(0.010mL)	25
2.5uL(0.0025mL)	2.0uL(0.0020mL)	5.0-quantiation limit
<2.5uL(0.025mL)	<2.0uL(0.0020mL)	<5.0ng- below the quantitation limit-diluted out

NOTE: Some instrument quantitation limits may be higher than the limit listed in the table. The volume of extract should be adjusted accordingly.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis of Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1 Two criteria must be met in order to identify a target compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{retention\ time\ of\ the\ target\ compound}{retention\ time\ of\ the\ associated\ internal\ standard}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. These ions due to the non-target compound should be subtracted from the sample component spectrum ass part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification. Tentative identifications of non-targets will be made only by analysts having completed the training specified in the training schedule.

11.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.2.2 The relative intensities of the major ions should agree within +/-30%.

11.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult other analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculation is

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes DF$$

where
Cis = concentration of the internal standard, ug/L
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
DF = dilution factor

Soils by Heated P/T

$$TIC\,(ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{5.0g}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
W = weight of sample analyzed, g
solids = decimal equivalent of percent solids

Soils by Methanol Extraction

$$TIC\,(ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{Vcal}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = decimal equivalent of the percent solids(percent solids/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

where
Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

11.3 Calculations for Samples-Internal Standard Technique

Aqueous Samples- relative response factor :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes DF$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
DF = dilution factor

Aqueous Samples: regression curve

$$concentration(ug/L) = concentration(curve) \otimes DF$$

where
DF = dilution factor

The reporting limit (RL) is calculated:

$$RL(ug/L) = RLqap \otimes DF$$

where
DF = dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

Soils by Heated P/T- relative response factor :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{5.0g}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/kg)
RRFavg = average response factor of the compound being measured
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

Soils by Heated P/T: regression curve

$$conc(ug/kg,dw) = Ccurve(ug/kg) \otimes \frac{5.0g}{(W)(solids)}$$

where
Ccurve = concentration from curve(ug/kg)
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The reporting limit (RL) is calculated:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

Methanol Extraction Soils and Wastes- relative response factor

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{Vcal}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = (percent solids)/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

Methanol Extraction of Soils and Solids- regression curve:

$$conc(ug,kg,dw) = Ccurve(ug/L) \otimes \frac{Vcal}{(W)(solids)}$$

where
Vcal = volume that calibration curve is based on (0.005L or 0.025L)
W = weight of sample added to the reagent water (g)-defined above

The reporting limit (RL)is calculated:

$$RL = RLgap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the reagent water (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in Section 3.1.3 of STL-SL SOP AN02: *Analytical Batching*. Note that the method blank for liquid samples and low-level soils is clock-specific and that the method blank for medium level soil samples is extraction batch-specific.

STL-SLSOP AN02: *Analytical Batching* describes the procedure for evaluating batch-specific QC. This criteria is summarized in the attached 8260 SOP Summary.

STL-SL SOP AN02 also contains the calculations for accuracy and precision and the calculations for the theoretical concentrations of surrogates, lab spikes, and matrix spikes.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration is the preparation and analysis of spiked reagent water. Section 8.3 of EPA Method 8260A gives the general procedure for the performance of the IDOC and Table 6 of EPA Method 8260A gives the acceptance criteria for the accuracy and precision.

12.3 Method Detection Limit

The method detection limit is determined in accordance with STL-SL SOP CA90.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. Section 10 of the STL-SL QAPs provide guidance on preventive maintenance.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time. See instrument manufacturers' manuals for guidance on locating and repairing instrument problems.

15.0 REFERENCES

1. *Savannah Laboratories' Comprehensive Quality Assurance Plan* and *Savannah Laboratories' Corporate Quality Assurance Plan*, current revisions.

2. Method s 5035, 8000B, and 8260B. *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846.including Update III*[U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Appendix A

VOLATILES BY GC/MS WORKING STANDARDS -EXAMPLE

These standards can be used to prepare the working standards for EPA Method 8260 to report the TCL (target compound list) compounds and the extended list of target compounds generally associated with EPA 8260. The standards are prepared in purge and trap grade methanol and are stored at 4C with minimum headspace.

Working Standard 1 (TCL WS-1)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #2	2000	12.5	25
VOA Cal #3	2000	12.5	25
VOA Cal #4	2000	12.5	25
1,2,-DCB	5000	5.0	25
1,3-DCB	5000	5.0	25
1,4-DCB	5000	5.0	25
2-CEVE	1000	125	125

Working Standard 2 (TCL WS-2)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #1	5000	25	125
8260 Surrogates	2500	10	25

Working Standard for GASES (TCL GASES)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
502.2 Cal 1	2000	12.5	25

Appendix A

Working Standard 3 (8260 WS-3)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
8260 Custom Mix #1	200	125	25
8260 Custom Mix #2	200	125	25
1,1,2,2-Tetrachloroethane	2000	12.5	25

Appendix A

Internal Standard (8260 ISTD)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50

Internal Standard/Surrogate (8260 ISSU)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50
8260 Surrogate	2500	20	50

Tune Evaluation Standard (4-BFB)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
4-BFB	5000	10	50

Matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
Matrix Spiking Solution	2500	20	50

TCLP matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
TCLP Spiking Solution	2000	16	125

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS - EXAMPLES

The following calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	TCL-1 *	TCL-2 *	TCL-3 *	TCL-4 *	TCL-5 *	TCL-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	8260-1 *	8260-2 *	8260-3 *	8260-4 *	8260-5 *	8260-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

CONCENTRATIONS OF THE CALIBRATION STANDARDS-5.0mL OR 5.0g

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
TCL-1,8260-1	5ug/l-kg	25ug/l-kg
TCL-2,8260-2	10ug/l-kg	50ug/l-kg
TCL-3,8260-3	25ug/l-kg	125ug/l-kg
TCL-4,8260-4	50ug/l-kg	250ug/l-kg
TCL-5,8260-5	100ug/l-kg	500ug/l-kg
TCL-6,8260-6	200ug/l-kg	1000ug/l-kg

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS-25mL Purge Volume-EXAMPLES

These calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The standards are based on a volume of 25mL to achieve lower quantitation limits for the target compounds. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	25TCL -1*	25TCL -2*	25TCL-3*	25TCL -4*	25TCL-5*	25TCL-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	258260-1*	258260 -2*	258260-3*	258260-4*	258260-5*	258260-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

CONCENTRATIONS OF THE CALIBRATION STANDARDS

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
25TCL-1,25-8260-1	1.0ug/l	5.0ug/l
25TCL-2,25-8260-2	2.0ug/l	10ug/l
25TCL-3,25-8260-3	5.0ug/l	25ug/l
25TCL-4,25-8260-4	10ug/l	50ug/l
25TCL-5,25-8260-5	20ug/l	100ug/l
25TCL-6,25-8260-6	40ug/l	200ug/l

Appendix B
8260 SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Container	Hold Time
Aqueous	None; 4C	40mL no headspace	7 days
	HCl pH<2; 4C	40mL-no headspace	14 days
Soil/solid(low level)	Iced at collection; 5mL sodium bisulfate added upon arrival in lab; store at 4C	5-g Encore Sampler	14 days
Soil/solid(low level) -high carbonates	Iced at collection; 5mL water added upon arrival in lab; store at –10C	5-g Encore Sampler	14 days
Soil/solid(high level)	None; 4C	Glass 125mL	14 days
TCLP	HCl pH<2; 4C	Tedlar bag or syringe	14 days

*storage temperature is 4C with a control criteria of less than 6C with no frozen samples

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
4-BFB 50ng on column Clock starts at injection	4-BFB 50ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification (50ug/L or 50ug/kg)) RL Standard-low point on cal curve (if necessary)
Method blank	Method blank
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

See SL SOP AN02, Section 3.1.3, for the batch/clock options for LCS and MS/MSD.

Recommended Internal Standards:
1,2-dichloroethane-d4; 1,4-difluorobenzene; chlorobenzene-d5; 1,4-dichlorobenzene-d4

Surrogates/System Monitoring Compounds:
dibromofluoromethane; toluene-d8; 4-bromofluorobenzene

LCS/MS: CQAP Subset:
1,1-dichloroethene; benzene; trichloroethene; toluene; chlorobenzene

Appendix B
8260 SOP SUMMARY

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION BROMOFLUOROBENZENE (BFB)	
m/e	Abundance Criteria
50	8.0-40.0% of mass 95
75	30.0-66.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	50-120%% of mass 95
175	4.0-9.0% of mass 174
176	93.0-101.0% of mass 174
177	5.0-9.0% of mass 176

(1) *8260 criteria taken from CLP OLMO4.0 (January 1998)

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
Less than or equal to 30% RSD	Less than or equal to 20% difference or drift from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

See Sections 10.3 and 10.4 for ICAL and CCV linearity checks and criteria.

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
MS Tune Check - 50ng 4-BFB	Before initial and continuing calibration standards - every 12 hours	Mass abundances within method acceptance criteria	-Evaluate chromatogram and spectrum - Reanalyze - Retune MS and reanalyze - Remake standard and reanalyze - Perform instrument maintenance and reanalyze
Initial Calibration – minimum five point curve with lowest point at or below the Reporting Limit (RL)	Initially; after major instrument maintenance; whenever continuing calibration check fails. Prior to analysis of method blank and samples	Method criteria for CCC/SPCC (see -Calibration Acceptance Criteria – Table presented earlier in this document)	- Evaluate chromatograms, spectra, and integrations - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument maintenance and recalibrate
Continuing Calibration check - midpoint standard	Every 12 hours before analysis of method blank and samples	Method criteria for CCC/SPCC (see Calibration Acceptance Criteria - Table presented earlier in this document)	- Evaluate chromatogram, spectra, integrations - Reanalyze standard - Remake and reanalyze standard - Recalibrate - Perform instrument maintenance and recalibrate
Method Blank	Every 12 hours (per clock) before sample analyses	All reported targets <RL	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Lab Control Sample (LCS) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Matrix Spike/Matrix Spike Duplicate (MS/MSD) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogates	All samples, blanks, LCS, MS	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Internal Standard Area	Evaluate all standards and samples	-Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence -Retention time of internal standard must be +/-30 seconds from internal standard in initial calibration -Areas in samples should be evaluated for gross error . Consult supervisor.	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze

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Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Reporting Limit Standard -1x to 2x the RL	(Optional) Daily. Required for Florida DEP	Detected with reasonable response	-Evaluate chromatogram, spectra, and integrations -Reanalyze -Remake standard and reanalyze -Retune and recalibrate -Perform instrument maintenance and recalibrate
Initial Demonstration of Capability	Per analyst	Method criteria	-Reanalyze targets that do not meet criteria
Method Detection Limit (MDL)	See STL-SL SOP CA90	See STL-SL SOP CA90	-Reanalyze and re-evaluate

Appendix C
EXAMPLE QUANTITATION REPORT

-quantitation ions
-internal standard and target compound association

Savannah Laboratories

COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m
Quant Method : ISTD Target Version : 3.50
Last Update : 12-Aug-1999 11:49 Number of Cpnds : 106
Data Type : MS DATA

Global Integrator : HP RTE

Chromat Events	Values
Initial:Thresh Units	0.000000
Initial:Area Thresh	200.000000
Initial:Max Peaks	100.000000
Initial:Bunching	2.000000
Initial:Smoothing	1.000000
Initial:Start Thresh	0.057000
Initial:Stop Thresh	0.142000
Initial:Baseline Reset	5.000000
Initial:Set Valley	100.000000

Compound	RT	RT Window	RF	Mass
1 DICHLORODIFLUOROMETHANE	2.291	1.842-2.740	6.813e-01	85.00
	2.291	1.842-2.740		87.00
	2.291	1.842-2.740		101.00
2 CHLOROMETHANE	2.589	2.140-3.038	1.054e+00	50.00
	2.595	2.146-3.044		52.00
3 VINYL CHLORIDE	2.729	2.280-3.178	1.429e+00	62.00
	2.723	2.274-3.172		64.00
4 BROMOMETHANE	3.173	2.724-3.622	3.097e+00	94.00
	3.173	2.724-3.622		96.00
	3.167	2.718-3.616		79.00
5 CHLOROETHANE	3.319	2.870-3.768	7.702e-01	64.00
	3.319	2.870-3.768		66.00
6 TRICHLOROFLUOROMETHANE	3.672	3.223-4.121	1.210e+00	101.00
	3.672	3.223-4.121		103.00
	3.672	3.223-4.121		105.00
7 TRICHLOROTRIFLUOROETHANE (1	3.672	3.223-4.121	1.173e+00	101.00
	3.672	3.223-4.121		151.00
	3.672	3.223-4.121		103.00
8 ACROLEIN	3.988	3.434-4.542		56.00
	3.988	3.434-4.542		55.00
9 ACETONITRILE	4.070	3.516-4.624		41.00
	4.070	3.516-4.624		40.00

Savannah Laboratories

COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

Compound	RT	RT Window	RF	Mass
10 1 1-DICHLOROETHENE	4.317	3.868-4.765	1.053e+00	96.00
	4.317	3.868-4.765		61.00
	4.323	3.874-4.772		98.00
11 ACETONE	4.359	3.910-4.808	1.171e-01	58.00
	4.359	3.910-4.808		43.00
12 DIMETHYL SULFIDE	4.557	4.003-5.111		47.00
	4.557	4.003-5.111		62.00
	4.557	4.003-5.111		45.00
13 CARBON DISULFIDE	4.609	4.160-5.057	4.138e+00	76.00
	4.609	4.160-5.057		78.00
14 IODOMETHANE	4.666	4.112-5.220		141.90
	4.666	4.112-5.220		126.90
15 METHYL MERCAPTAN	4.827	4.273-5.381		47.00
	4.827	4.273-5.381		48.00
	4.827	4.273-5.381		45.00
16 ACRYLONITRILE	4.876	4.322-5.430		53.00
	4.876	4.322-5.430		52.00
	4.876	4.322-5.430		51.00
17 METHYLENE CHLORIDE	4.895	4.446-5.343	1.289e+00	84.00
	4.888	4.440-5.337		49.00
	4.888	4.440-5.337		86.00
18 3-CHLORO-1-PROPENE	4.946	4.392-5.500		41.00
	4.946	4.392-5.500		76.00
19 METHYL T-BUTYL ETHER	5.223	4.774-5.672	2.979e-03	73.00
	5.223	4.774-5.672		57.00
20 trans-1 2-DICHLOROETHENE	5.235	4.786-5.684	1.213e+00	96.00
	5.241	4.792-5.690		61.00
	5.235	4.786-5.684		98.00
21 1 1-DICHLOROETHANE	5.734	5.285-6.183	1.785e+00	63.00
	5.734	5.285-6.183		65.00
	5.752	5.303-6.201		83.00
22 VINYL ACETATE	5.771	5.322-6.219	1.308e+00	43.00
	5.771	5.322-6.219		86.00
23 2-BUTANONE	6.446	5.997-6.895	4.896e-01	43.00
	6.446	5.997-6.895		72.00
24 cis-1 2-DICHLOROETHENE	6.458	6.009-6.907	1.297e+00	96.00
	6.458	6.009-6.907		61.00
	6.446	5.997-6.895		98.00
25 2 2-DICHLOROPROPANE	6.458	6.009-6.907	7.527e-01	77.00
	6.458	6.009-6.907		41.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

	Compound	RT	RT Window	RF	Mass
	26 BROMOCHLOROMETHANE	6.750	6.301-7.199	5.114e-01	127.90
		6.750	6.301-7.199		49.00
		6.750	6.301-7.199		129.90
	27 CHLOROFORM	6.835	6.386-7.284	1.687e+00	83.00
		6.835	6.386-7.284		85.00
		6.841	6.392-7.290		47.00
	28 PROPIONITRILE	6.885	6.331-7.439		54.00
		6.885	6.331-7.439		55.00
$	29 DIBROMFLUOROMETHANE	7.048	6.599-7.497	8.084e-01	112.90
		7.030	6.581-7.479		81.00
		7.048	6.599-7.497		110.90
	30 1 1 1-TRICHLOROETHANE	7.103	6.620-7.586	3.268e-01	97.00
		7.103	6.620-7.586		99.00
		7.109	6.626-7.592		61.00
	31 1 1-DICHLOROPROPENE	7.316	6.833-7.799	3.671e-01	75.00
		7.316	6.833-7.799		110.00
		7.316	6.833-7.799		77.00
	32 HEXANE	7.325	6.771-7.879		57.00
		7.325	6.771-7.879		41.00
		7.325	6.771-7.879		43.00
	33 CARBON TETRACHLORIDE	7.328	6.845-7.811	2.509e-01	116.90
		7.328	6.845-7.811		118.90
		7.328	6.845-7.811		120.90
	34 TETRAHYDROFURAN	7.410	6.856-7.964		42.00
		7.410	6.856-7.964		71.00
		7.410	6.856-7.964		72.00
*	35 1,2-DICHLOROETHANE-d4	7.480	7.031-7.929		65.00
		7.480	7.031-7.929		67.00
		7.492	7.043-7.941		102.00
	36 CHLOROPRENE	7.492	6.938-8.046		53.00
		7.492	6.938-8.046		88.00
	37 1 2-DICHLOROETHANE	7.590	7.106-8.073	2.546e-01	62.00
		7.577	7.094-8.060		49.00
		7.590	7.106-8.073		64.00
	38 BENZENE	7.590	7.106-8.073	1.335e+00	78.00
		7.583	7.100-8.067		50.00
		7.583	7.100-8.067		51.00
	39 METHACRYLONITRILE	7.959	7.405-8.513		67.00
		7.959	7.405-8.513		52.00

Savannah Laboratories

COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

	Compound	RT	RT Window	RF	Mass
*	40 1,4-DIFLUOROBENZENE	8.052	7.569-8.535		114.00
		8.052	7.569-8.535		63.00
		8.058	7.575-8.541		88.00
	41 TRICHLOROETHENE	8.435	7.952-8.918	2.915e-01	129.90
		8.435	7.952-8.918		95.00
		8.429	7.946-8.912		131.90
	42 1 2-DICHLOROPROPANE	8.733	8.250-9.216	3.098e-01	63.00
		8.727	8.244-9.210		76.00
		8.733	8.250-9.216		65.00
	43 ISOBUTANOL	8.883	8.329-9.437		43.00
		8.883	8.329-9.437		41.00
	44 DIBROMOMETHANE	8.904	8.420-9.387	1.437e-01	93.00
		8.904	8.420-9.387		173.80
		8.904	8.420-9.387		95.00
	45 BROMODICHLOROMETHANE	9.104	8.621-9.587	3.080e-01	83.00
		9.110	8.627-9.593		85.00
		9.098	8.615-9.581		129.00
	46 ETHYL ACETATE	9.303	8.749-9.857		43.00
		9.303	8.749-9.857		45.00
		9.303	8.749-9.857		88.00
	47 2-CHLOROETHYL VINYL ETHER	9.506	9.023-9.989	1.457e-01	63.00
		9.512	9.029-9.995		65.00
		9.500	9.017-9.983		106.00
	48 CYCLOHEXANE	9.592	8.945-10.239		56.00
		9.592	8.945-10.239		84.00
	49 cis-1,3-DICHLOROPROPENE	9.749	9.266-10.232	4.493e-01	75.00
		9.749	9.266-10.232		77.00
		9.743	9.260-10.226		110.00
	50 2-HEXANONE	9.950	9.205-10.695	5.456e-01	43.00
		9.950	9.205-10.695		58.00
$	51 TOLUENE-d8	10.163	9.680-10.646	1.031e+00	98.00
		10.163	9.680-10.646		100.00
		10.151	9.668-10.634		70.00
	52 TOLUENE	10.260	9.777-10.743	7.674e-01	92.00
		10.260	9.777-10.743		91.00
		10.254	9.771-10.737		65.00
	53 HEPTANE	10.487	9.840-11.134		43.00
		10.487	9.840-11.134		57.00
		10.487	9.840-11.134		71.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

	Compound	RT	RT Window	RF	Mass
	54 trans-1,3-DICHLOROPROPENE	10.558	10.075-11.041	3.927e-01	75.00
		10.564	10.081-11.047		77.00
		10.558	10.075-11.041		110.00
	55 1 1 2-TRICHLOROETHANE	10.844	10.361-11.327	2.219e-01	83.00
		10.850	10.367-11.333		97.00
		10.844	10.361-11.327		99.00
	56 TETRACHLOROETHENE	11.106	10.361-11.851	5.192e-01	163.90
		11.118	10.373-11.863		165.90
		11.112	10.367-11.857		167.90
	57 1 3-DICHLOROPROPANE	11.112	10.629-11.595	4.788e-01	76.00
		11.112	10.629-11.595		78.00
		11.112	10.629-11.595		41.00
	58 4-METHYL-2-PENTANONE (MIBK	11.215	10.732-11.698	1.708e-01	43.00
		11.215	10.732-11.698		57.00
		11.215	10.732-11.698		58.00
	59 DIBROMOCHLOROMETHANE	11.471	10.726-12.216	5.124e-01	128.90
		11.471	10.726-12.216		126.90
		11.477	10.732-12.222		130.90
M	60 1,2-DICHLOROETHENE (total)	11.485	7.016-8.164	1.255e+00	96.00
	61 1 2-DIBROMOETHANE	11.672	11.188-12.155	2.421e-01	107.00
		11.672	11.188-12.155		109.00
	62 METHYL METHACRYLATE	12.235	11.588-12.882		69.00
		12.235	11.588-12.882		41.00
	63 1-CHLOROHEXANE	12.409	11.664-13.154	1.251e+01	41.00
		12.409	11.664-13.154		43.00
		12.409	11.664-13.154		55.00
*	64 CHLOROBENZENE-d5	12.420	11.675-13.165		82.00
		12.420	11.675-13.165		117.00
		12.420	11.675-13.165		119.00
	65 CHLOROBENZENE	12.462	11.717-13.207	1.691e+00	112.00
		12.462	11.717-13.207		77.00
		12.456	11.711-13.201		51.00
	66 1 1 1 2-TETRACHLOROETHANE	12.584	11.839-13.329	5.385e-01	130.90
		12.584	11.839-13.329		132.90
		12.584	11.839-13.329		119.00
	67 ETHYL BENZENE	12.633	11.887-13.378	3.045e+00	91.00
		12.633	11.887-13.378		106.00
		12.627	11.881-13.372		51.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

	Compound	RT	RT Window	RF	Mass
	68 m,p-XYLENE	12.821	12.076-13.566	1.187e+00	106.00
		12.827	12.082-13.573		91.00
		12.827	12.082-13.573		77.00
	69 DIMETHYL DISULFIDE	13.378	12.731-14.025		94.00
		13.378	12.731-14.025		45.00
		13.378	12.731-14.025		79.00
	70 o-XYLENE	13.497	12.751-14.242	1.144e+00	106.00
		13.497	12.751-14.242		91.00
		13.503	12.757-14.248		77.00
	71 STYRENE	13.515	12.770-14.260	1.885e+00	104.00
		13.515	12.770-14.260		78.00
		13.515	12.770-14.260		103.00
	72 BROMOFORM	13.831	13.086-14.576	3.683e-01	172.80
		13.825	13.080-14.570		170.80
		13.831	13.086-14.576		174.80
	73 ISOPROPYLBENZENE	14.123	13.137-15.109	3.229e+00	105.00
		14.123	13.137-15.109		120.00
		14.123	13.137-15.109		77.00
$	74 p-BROMOFLUOROBENZENE	14.391	13.646-15.136	7.764e-01	95.00
		14.385	13.639-15.130		174.00
		14.385	13.639-15.130		176.00
	75 1 1 2 2-TETRACHLOROETHANE	14.622	13.877-15.367	7.407e-01	83.00
		14.622	13.877-15.367		85.00
		14.616	13.871-15.361		168.00
	76 BROMOBENZENE	14.659	13.673-15.644	7.575e-01	156.00
		14.659	13.673-15.644		77.00
		14.659	13.673-15.644		158.00
	77 1 2 3-TRICHLOROPROPANE	14.701	13.715-15.687	2.250e-01	110.00
		14.701	13.715-15.687		112.00
	78 n-PROPYLBENZENE	14.841	13.855-15.827	9.029e-01	120.00
		14.841	13.855-15.827		91.00
		14.841	13.855-15.827		65.00
	79 2-CHLOROTOLUENE	14.999	14.013-15.985	7.182e-01	126.00
		14.999	14.013-15.985		91.00
		14.999	14.013-15.985		63.00
	80 n-BUTYL ACETATE	15.096	13.997-16.195		56.00
		15.096	13.997-16.195		43.00
	81 1 3 5-TRIMETHYLBENZENE	15.145	14.159-16.131	2.424e+00	105.00
		15.145	14.159-16.131		120.00
		15.145	14.159-16.131		77.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

	Compound	RT	RT Window	RF	Mass
	82 4-CHLOROTOLUENE	15.182	14.196-16.167	7.548e-01	126.00
		15.182	14.196-16.167		91.00
		15.182	14.196-16.167		63.00
M	83 XYLENE (total)	15.547	15.083-16.231	1.197e+00	106.00
	84 tert-BUTYLBENZENE	15.723	14.737-16.709	2.362e+00	119.00
		15.723	14.737-16.709		91.00
		15.723	14.737-16.709		134.00
	85 1 2 4-TRIMETHYLBENZENE	15.814	14.829-16.800	2.418e+00	105.00
		15.814	14.829-16.800		120.00
		15.814	14.829-16.800		77.00
	86 ETHYL METHACRYLATE	15.843	14.744-16.942		69.00
		15.843	14.744-16.942		41.00
		15.843	14.744-16.942		39.00
	87 sec-BUTYLBENZENE	16.119	15.133-17.104	3.458e+00	105.00
		16.119	15.133-17.104		134.00
		16.119	15.133-17.104		91.00
	88 1 3-DICHLOROBENZENE	16.325	15.340-17.311	1.404e+00	146.00
		16.319	15.334-17.305		148.00
		16.313	15.327-17.299		111.00
	89 p-ISOPROPYLTOLUENE	16.374	15.388-17.360	2.676e+00	119.00
		16.374	15.388-17.360		134.00
		16.374	15.388-17.360		91.00
*	90 1,4-DICHLOROBENZENE d4	16.429	15.443-17.415		152.00
		16.429	15.443-17.415		150.00
		16.423	15.437-17.408		115.00
	91 1 4-DICHLOROBENZENE	16.471	15.486-17.457	1.431e+00	146.00
		16.471	15.486-17.457		148.00
		16.478	15.492-17.463		111.00
$	92 1,2-DICHLOROBENZENE D4	17.104	16.118-18.090	1.403e+00	150.00
		17.104	16.118-18.090		152.00
		17.104	16.118-18.090		115.00
	93 n-BUTYLBENZENE	17.122	16.136-18.108	2.737e+00	91.00
		17.122	16.136-18.108		92.00
		17.122	16.136-18.108		134.00
	94 1 2-DICHLOROBENZENE	17.141	16.155-18.126	1.237e+00	146.00
		17.141	16.155-18.126		148.00
		17.135	16.149-18.120		111.00
	95 1-CHLOROHEXANE	18.372	17.273-19.471		41.00
		18.372	17.273-19.471		43.00
		18.372	17.273-19.471		55.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

Compound	RT	RT Window	RF	Mass
96 1 2-DIBROMO-3-CHLOROPROPAN	18.552	17.566-19.537	1.133e-01	75.00
	18.552	17.566-19.537		156.90
	18.552	17.566-19.537		154.90
97 5-METHYL-2-HEXANONE (MIAK)	18.722	17.623-19.821		43.00
	18.722	17.623-19.821		58.00
	18.722	17.623-19.821		57.00
98 1 2 4-TRICHLOROBENZENE	20.146	19.160-21.131	8.787e-01	179.90
	20.146	19.160-21.131		181.90
	20.146	19.160-21.131		145.00
99 trans-1,4-DICHLORO-2-BUTEN	20.446	19.065-21.827		53.00
	20.446	19.065-21.827		88.00
	20.446	19.065-21.827		89.00
100 HEXACHLOROBUTADIENE	20.541	19.556-21.527	4.492e-01	224.80
	20.541	19.556-21.527		222.80
	20.541	19.556-21.527		189.90
101 NAPHTHALENE	20.675	19.689-21.661	2.234e+00	128.00
	20.675	19.689-21.661		102.00
	20.675	19.689-21.661		51.00
102 alpha PINENE	20.750	19.651-21.849		93.00
	20.750	19.651-21.849		92.00
	20.750	19.651-21.849		91.00
103 beta PINENE	20.750	19.651-21.849		93.00
	20.750	19.651-21.849		92.00
	20.750	19.651-21.849		91.00
104 1 2 3-TRICHLOROBENZENE	21.241	20.496-21.986	7.900e-01	180.00
	21.241	20.496-21.986		182.00
105 PENTACHLOROETHANE	22.047	20.948-23.146		167.00
	22.047	20.948-23.146		130.00
106 2-methylnapthalene	30.000	29.500-30.500		142.00
	30.000	29.500-30.500		141.00
	30.000	29.500-30.500		0.00

B-3 Semi-Volatile Compounds by GC/MS (EPA 8270C)

SEMI-VOLATILE COMPOUNDS BY GC/MS (8270C)

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Approved by:

R. Wayne Cole Jan 11, 2000

Title: STL-SL Technical Manager Date

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of various semi-volatile organic compounds (SVOC) in groundwater, TCLP and SPLP leachates, soils, sediments, waste, and solid sample extracts. The attached quantitation report (Appendix B) lists the routine target compounds, the retention times of the target compounds, the characteristic ions of the target compounds, and the internal standard association of each target compound.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits for the target compounds are given in Section 5 of the current revision of the STL Savannah Laboratories' *Laboratory Quality Plan.*

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the mass spectra determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 This procedure is based on the guidance provided in SW-846 Method 8270C.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated area.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

3.4 The exit vent of the splitless injector must have a carbon trap in-line to collect the semivolatile compounds that are vented during the injection of the extract. The traps should be changed every three months and disposed of in accordance with STL-SL SOP CA70: *Waste Management.*

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products such as diesel or waste oil.

4.3 Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days from TCLP leaching procedure	40 days

Refrigerator temperature acceptance criterion is less than 6C with no frozen samples.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph- Hewlett-Packard (HP) 5890 or equivalent with compatible autosampler, splitless injector, and direct capillary interface. The exit vent of the splitless injector must have a carbon trip in-line to collect the semivolatile compounds that are vented during the injection of extracts. The carbon traps should be changed every three months.

6.2 Mass spectrometer- HP5971, HP5972, HP5973 or equivalent

6.3 Recommended Capillary column-HP-5MS, 30m x 0.25mm ID x 0.25um film thickness or equivalent column.

6.4 Data system- compatible with GC/MS system

6.5 Microsyringes- appropriate volumes

6.6 Volumetric flasks, Class A-appropriate volumes

6.7 Autosampler vials and crimper, compatible with autosampler

7.0 REAGENTS

Reagents must be tracked in accordance with STL-SL SOP AN44: *Reagent Traceability.*

7.1 Methylene chloride-pesticide residue grade, for preparation of standards

7.2 Acetone-pesticide residue grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with STL-SL SOP AN41: *Standard Material Traceability*. General guidance on the preparation of standards is given in sTL-SL SOP AN43: *Standard Preparation.*

The lab should purchase certified solutions from STL-SL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See STL-SL SOP AN43 for guidance for standard preparation from neat materials.

8.1 Preparation of the Stocks from Neat Standards

The steps for the preparation of primary stock standards from neat materials are given in STL-SL SOP *AN43: Standard Preparation.* The standards should be prepared in methylene chloride but may require other solvents to dissolve the material.

8.2 Preparation the calibration standards from the stock standards

A minimum of five calibration standards are prepared. The concentrations of the stock standards are in the 1000-10000ug/mL range. The recommended standards are listed in Section 10.2. The lowest level standard should be at the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve and seven calibration points are required for a polynomial regression curve (see SW-846 Method 8000B). Internal standards should be added to each standard to give a final concentration of 40 ug/mL.

Each lab should develop controlled recipes that can be posted or maintained in appropriate logbooks.

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP	Extraction Technique
Aqueous, TCLP leachates	EX30	Continuous Liquid-liquid Extraction
Aqueous, TCLP leachates	EX35	Separatory Funnel
Soils/Sediments	EX40	Sonication
Wastes	EX42	Waste dilution

9.2 The sample concentration procedures are given in STL-SL SOP EX 50: Zymark Nitrogen Concentration.

9.3 Gel permeation chromatography (GPC-STL-SL SOP EX61) may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products.

10.0 PROCEDURE

10.1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must optimize and document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:

HP-5MS 30m x 0.25mm ID x 0.25um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:

Initial column temperature: 45 C for 3 minutes
Column temperature program: 10C per minute
Final column temperature: 300C (until at least one minute past the elution time of Benzo (g,h,i) perylene).

GC injector parameters

Injector temperature: 250-270°C
Injection type: split, approximately 1:10 or splitless injection
Injector liner: 4mm ID quartz or 4mm glass, deactivated (single "Gooseneck")
Sample injection volume: 1-2uL

Mass Spectrometer and interface parameters

Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: 35-500amu, with a scan time of 1.0 scans per second or greater

10.2 Calibration

A minimum of five calibration standards are prepared and analyzed. The recommended TCL standards are 10, 50, 80, 100, 200, and 350ug/mL. The recommended Appendix IX concentrations are 10, 20, 50, 80, 100, 120, and 160ug/mL. The lowest level standard should be at or below the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve and seven calibration points are required for a polynomial regression curve (see SW-846 Method 8000B).

10.2.1 Fifty nanograms of DFTPP must be analyzed at the beginning of each 12-hour clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios. The DFTPP analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of DFTPP. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage.

10.2.1.1 Prepare a 50 ng/uL solution of tune/ column evaluation standard containing each of the following compounds at 50 ug/mL in methylene chloride: DFTPP, pentachlorophenol, p,p'-DDT, and benzidine.

10.2.1.2 Analyze a 1uL aliquot of the tune/column evaluation solution.

10.2.1.3 Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary (Appendix A). Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.2.1.4 Benzidine and pentachlorophenol should be present at their normal responses with no peak tailing visible. This is a good check on the system: if pentachlorophenol (a CCC) does not respond well, the calibration standard should not be analyzed. Perform injector port and column maintenance and reanalyze the tune/column evaluation standard.

The percent breakdown of p,p'- DDT is calculated using the following equation. The percent breakdown should not exceed 20%.

$$\%Breakdown = \frac{(areaDDE + areaDDD)}{(areaDDT + areaDDE + areaDDD)} x\,100$$

Areas from the total ion chromatogram are used to calculate DDT breakdown.

10.2.2 After the DFTPP criteria and column evaluation criteria have been met, the initial calibration standards are analyzed

10.2.2.1 Prepare the initial calibration standards. The lowest calibration standard should be at the RL and the rest of the standards will define the working range. See section 10.2 for guidance regarding calibration levels.

10.2.2.2 Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts.

10.2.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.

10.2.4 Calculate the relative response factor for each compound as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured
(See the attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration of the compound being measured (ug/mL)
Cis = concentration of the internal standard (40ug/mL)

Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

10.2.5 Calculate the average relative response factor (RRF_{avg}) for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3.... + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

NOTE: As noted previously, some target compounds may have fewer than five calibration standard levels.

10.2.6 Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_1} (RRF_i - RRF_{avg})^2}{n-1}}$$

10.2.7 Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards.

$$\%RSD = \frac{SD}{RRF_{avg}} x\,100$$

10.2.8 Evaluation of the Initial Calibration

The initial calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in the SOP Summary (Appendix A). The %RSD criteria for CCC and minimum RRF for SPCC must be met before the analysis of sample extracts can begin.

If the CCC and SPCC criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the initial calibration must be repeated. The analyst must be aware of the 12-hour clock for the DFTPP analysis. The DFTPP criteria must be met prior to the analysis of the calibration standards.

10.2.8 After the initial calibration criteria (CCC/SPCC) have been met, each target is evaluated for linearity.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration of the target compound being measured (ug/mL)
Cis = concentration of the internal standard (ug/mL)

A linear, quadratic, or higher order regression fit may be used to define the concentration/response relationship. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples. Regression curves may be forced through zero but it is recommended that the curve be evaluated without forcing through zero first and then with the curve forced through the origin. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire concentration range.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of the tune/column evaluation solution (10.2.1.1). The tune and column evaluation criteria (10.2.1.3 and 10.2.1.4) must be met before the analysis of the calibration check standards can take place.

10.3.1 After the tune and column evaluation criteria have been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a mid level concentration. The CCC and SPCC criteria (SOP Summary, Appendix A) must be met before the analysis of samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration (ug/mL)
Cccv = measured concentration using the selected quantitation method (ug/mL)

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If these criteria are not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.3.2 The continuing calibration verification standard (CCV) must also be evaluated for internal standard retention time and response.

If the retention time of any internal standard changes by more than 30 seconds from the last 12-hour calibration check, the analytical system must be inspected for problems and corrective action instituted.

If the extracted ion current profile (EICP) area for any of the internal standards in the CCV changes by more than a factor of two (-50% to +100%) from the last initial calibration sequence, the analytical system must be inspected for problems and corrective action instituted.

10.4 Samples are analyzed only after the DFTPP criteria, column evaluation criteria and the calibration verification criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the tune/column evaluation standard and a mid-level calibration standard.

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- Minimum of five cal levels	Mid point calibration verification Optional RL: Standard-low point on cal curve
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

10.4.1 Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.4.2 Add 20-uL of the internal standard mix (2000 ug/mL) to each 1.0 mL aliquot of the sample extract. The concentration of the internal standard in the extract is 40 ug/mL.

10.4.3 Mix the contents of the autosampler vial by inverting several times.

10.4.4 Analyze the samples using the same analytical conditions used for the initial and continuing calibration standard. Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

10.4.5 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100uL of a sample extract are diluted to final volume of 1.0mL, the dilution factor is 10. (1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

uL extract-Vext	uL MeCl2	volume of dilution (Vdil-uL)	uL ISTD (2000ug/mL)-Vistd	DF
1000	0	1000	20	1
500	500	1000	10*	2
200	800	1000	16*	5
100	900	1000	18*	10
50	950	1000	19*	20
20	980	1000	20*	50

*assumes dilution of a 1.0mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 40ug/mL using 20ul of a 2000ug/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 40ug/mL. The following equation can be used to determine the volume of the 2000ug/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$Vistd\,(uL) = 20uL - \left(\frac{Vext}{Vdil} \otimes 20ul\right)$$

Vistd = volume of 2000ug/mL internal standard to add to the diluted extract (uL)
Vext = volume of extract used to prepare the dilution (uL)
Vdil = final volume of the dilution (uL)-1000uL (1.0mL)

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

11.1.1 Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from the daily calibration standard or a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1.1 Two criteria must be met in order to positively identify a compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{\textit{retention time of the target compound}}{\textit{retention time of the associated internal standard}}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. Ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification..

11.1.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.1.2.2 The relative intensities of the major ions should agree within +/-30%.

11.1.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.1.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.1.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.1.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult senior analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.1.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculations assume that the same volume is injected for standards and samples.

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{V} \otimes DF$$

where

Cis =	concentration of the internal standard (ug/mL)
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract (mL)
V =	volume of sample extract (L)
DF =	dilution factor

Soils

$$TIC\ (ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Cis =	concentration of the internal standard, ug/mL
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract mL
W =	weight of sample analyzed (kg)

solids = decimal equivalent of percent solids

11.2 Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples and that the standards and samples have the same concentration of internal standard.

11.2.1 Aqueous Samples

11.2.1.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.2 If a regression curve is used, the concentration is given:

$$concentration(ug/L) = Ccurve \otimes \frac{F}{V} \otimes DF$$

where

C_{curve} =	concentration from curve (ug/mL)
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.3 The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Vqap}{V} \otimes DF$$

where

F =	final volume of extract (mL)
Fqap =	1.0mL
Vqap =	1.0L
V =	volume of sample extracted
DF =	dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

11.2.2 Soils

11.2.2.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100
DF =	dilution factor

11.2.2.2 If the regression curve is used, the concentration is given:

$$conc(ug/kg,dw) = Ccurve \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ccurve =	concentration from curve(ug/mL)
W =	weight of sample extracted (kg)
F =	final volume of extract (mL)
solids =	(percent solids)/100)
DF =	dilution factor

11.2.2.3 The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Wqap}{(W)(solids)} \otimes DF$$

where

F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100

The SL CQAP assumes Wqap = 30g, solids = 1, Fqap = 1.0mL, and DF = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching*. STL-SL SOP AN02 also describes the procedure for evaluating batch-specific QC. The QA/QC criteria are summarized in the SOP Summary (Appendix A).

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must participate in the analysis of samples by this procedure in accordance with STL-SL SOP CA92: *Evaluation of IDOCs.*

12.3 Method Detection Limit

The method detection limit is determined in accordance with STL-SL SOP CA90.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. See Section 10 of the current STL-SL Laboratory Quality Manual.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time.

15.0 REFERENCES

15.1 *STL Savannah Laboratories' Laboratory Quality Manual* current revisions.

15.2 Method 8270C:*Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

APPENDIX A
8270C SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days	40 days

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification standard RL Standard (lowest point on calibration curve if required by client or state-specific QAP)
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (DFTPP)	
m/e	Ion Abundance Criteria (1)
51	30-80% of mass 442
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	25-75% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5.0-9.0% of mass 198
275	10-30% of mass 198
365	Greater than 0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15.0-24.0% of mass 442

(1) 8270 criteria taken from CLP OLMO4.0 (January 1998). The use of alternate criteria is expressly allowed in SW-846 Method 8270C.

APPENDIX A
8270C SOP SUMMARY

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Phenol, 1,4-Dichlorobenzene, 2-Nitrophenol, 2,4-Dichlorophenol, Hexachlorobutadiene, 4-Chloro-3-methylphenol, 2,4,6-Trichlorophenol, Acenapthene, N-Nitrosodiphenylamine, Pentachlorophenol, Fluoranthene, Di-n-octylphthalate, Benzo(a) pyrene

System Performance Check Compounds-SPCC
N-Nitrosodi-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol, 4-Nitrophenol

Initial Calibration	Continuing Calibration*
CCC: <= 30% RSD	CCC: <= 20% difference from initial calibration
SPCC: RRFavg >= 0.050	SPCC: RRF>= 0.050

*If CCC and/or SPCC do not meet the stated criteria, all targets that are reported must meet the CCC criteria.

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

The results for all target compounds are evaluated for linearity. If the %RSD is less than 15%, the calibration is assumed linear through the origin and the average response factor can be used for quantitation. If the average response factor for the target exceeds 15% (including any CCC), the analyst must use the calibration curve option.

NOTE: The lab has the option of using a regression curve for all analytes.

A linear, quadratic, or higher order regression fit may be used to define the concentration/response relationship. If the correlation coefficient of the linear regression curve or the coefficient of determination of a higher order fit is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range. The minimum number of calibration standards required for a regression curve are given in the following table:

Type of curve	Minimum Number of Calibration Points
Linear (first order)	5
Quadratic (second order)	6
Polynomial(third)	7

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 50ng Pentachlorophenol - 50ng Benzidine – 50ng p,p'-DDT 50ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
		Pentachlorophenol and benzidine - present at usual response with no peak tailing visible p,p'-DDT - %breakdown <20%	-Reanalyze -Perform injector port maintenance and reanalyze -Cut more than usual length of column and reanalyze -Replace column
Initial Calibration	After Tune Check and when calibration verification standard fails acceptance criteria. All initial calibration standards	CCC: %RSD < 30% SPCC: RRFavg > 0.050 Use regression curve for quantitation if %RSD for any target compound exceeds 15%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples	CCC: %Difference <= 20% Or %Drift <= 20% SPCC: RRF >= 0.050	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence Areas in samples should be evaluated for gross error . Consult supervisor Retention time of internal standard must be +/-30 seconds from internal standard in previous CCV.	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available -Recalibrate

QC Item	Frequency	Acceptance Criteria	Corrective Action
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within STL-SL LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in LQM	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Lab Control Standard (LCS) - QAP subset	Per batch See STL SOP AN02	Within STL-SL LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See AN02	Within STL-SL LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
RL Standard (reporting limit)	Daily (optional)-lowest point on calibration curve if required by client or state-specific QAP	Detected at reasonable sensitivity	-Evaluate integrations and spectra; -Reanalyze -Prepare new standard and reanalyze
Initial Demonstration of Capability (IDOC)	Each work group	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	Annually for each routine matrix See STL SL SOP CA90	Evaluate according to STL-SL SOP CA90	Evaluate according to STL-SL SOP CA90

STL Savannah Laboratories

APPENDIX B- EXAMPLE QUANTITATION REPORT

Savannah Laboratories

Semivolatile REPORT SW-846 Method 8270C

Data file : /chem/SM/MSE5973.i/1e0107.b/eq612.d
Lab Smp Id: SMTCL-200 Client Smp ID: SMTCL-200
Inj Date : 07-JAN-2000 08:56
Operator : Inst ID: MSE5973.i
Smp Info : 1/7/00
Misc Info : SMTCL-200 SMC03-7-1
Comment :
Method : /chem/SM/MSE5973.i/1e0107.b/e-b8270C-m.m
Meth Date : 07-Jan-2000 10:19 jstingl Quant Type: ISTD
Cal Date : 28-DEC-1999 19:45 Cal File: eq582.d
Als bottle: 2 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: tl99(350).sub
Target Version: 3.40
Processing Host: cserver1

		Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (NG)	AMOUNTS ON-COL (NG)
*	1	1,4-Dichlorobenzene-d4	152	5.109	5.109	(1.000)	138890		
	2	1,4-Dioxane	88	2.284	2.284	(0.447)	365537	200.000	180
	3	Pyridine	79	2.573	2.573	(0.504)	990777	200.000	170
	4	N-Nitrosodimethylamine	42	2.563	2.563	(0.502)	372916	200.000	180
$	5	2-Fluorophenol	112	3.784	3.784	(0.741)	783911	200.000	180
$	6	Phenol-d5	99	4.768	4.768	(0.933)	1100575	200.000	180
	7	Aniline	93	4.788	4.788	(0.937)	1279048	200.000	170
	8	Phenol	94	4.778	4.778	(0.935)	1157650	200.000	180
	9	Bis(2-chloroethyl)ether	63	4.861	4.861	(0.951)	629821	200.000	180
	10	2-Chlorophenol	128	4.912	4.912	(0.962)	921621	200.000	190
	11	1,3-Dichlorobenzene	146	5.068	5.068	(0.992)	1002450	200.000	190
	12	1,4-Dichlorobenzene	146	5.120	5.120	(1.002)	1011714	200.000	190
	13	Benzyl Alcohol	108	5.295	5.295	(1.036)	621327	200.000	180
	14	1,2-Dichlorobenzene	146	5.337	5.337	(1.045)	943597	200.000	190
	15	2-Methylphenol	107	5.440	5.440	(1.065)	742922	200.000	190
	16	bis(2-Chloroisopropyl)ether	45	5.471	5.471	(1.071)	1127568	200.000	190
	17	N-Nitroso-di-n-propylamine	70	5.637	5.637	(1.103)	652256	200.000	180
	18	3&4-Methylphenol	107	5.606	5.606	(1.097)	1071208	200.000	180
	19	Hexachloroethane	117	5.678	5.678	(1.111)	369084	200.000	190
*	20	Naphthalene-d8	136	6.620	6.620	(1.000)	576109		
$	21	Nitrobenzene-d5	82	5.772	5.772	(0.872)	999920	200.000	210
	22	Nitrobenzene	77	5.792	5.792	(0.875)	964588	200.000	200
	23	Isophorone	82	6.082	6.082	(0.919)	1688698	200.000	180
	24	2-Nitrophenol	139	6.175	6.175	(0.933)	524138	200.000	230
	25	2,4-Dimethyphenol	122	6.237	6.237	(0.942)	774066	200.000	180
	26	Bis(2-chloroethoxy)methane	93	6.362	6.362	(0.961)	1061177	200.000	180

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (NG)	ON-COL (NG)
	27 Benzoic acid	122	6.434	6.434	(0.972)	535495	200.000	180
	28 2,4-Dichlorophenol	162	6.475	6.475	(0.978)	777505	200.000	200
	29 1,2,4-Trichlorobenzene	180	6.569	6.569	(0.992)	813165	200.000	200
	30 Naphthalene	128	6.651	6.651	(1.005)	2635688	200.000	220
	31 4-Chloroaniline	127	6.755	6.755	(1.020)	1098581	200.000	180
	32 Hexachlorobutadiene	225	6.889	6.889	(1.041)	426857	200.000	200
	33 4-Chloro-3-methylphenol	107	7.417	7.417	(1.120)	827216	200.000	190
	34 2-Methylnaphthalene	142	7.572	7.572	(1.144)	1802569	200.000	190
	35 1-Methylnaphthalene	142	7.717	7.717	(1.166)	1730785	200.000	190
*	36 Acenaphthene-d10	164	9.094	9.094	(1.000)	329747		
	37 Hexachlorocyclopentadiene	237	7.893	7.893	(0.868)	529072	200.000	230
	38 2,4,6-Trichlorophenol	196	8.018	8.018	(0.882)	561125	200.000	200
	39 2,4,5-Trichlorophenol	196	8.069	8.069	(0.887)	595405	200.000	200
$	40 2-Fluorobiphenyl	172	8.131	8.131	(0.894)	1881161	200.000	190
	41 2-Chloronaphthalene	162	8.256	8.256	(0.908)	1714878	200.000	190
	42 2-Nitroaniline	65	8.463	8.463	(0.931)	551375	200.000	210
	43 Dimethylphthalate	163	8.804	8.804	(0.968)	1947578	200.000	180
	44 2,6-Dinitrotoluene	165	8.897	8.897	(0.978)	459158	200.000	230
	45 Acenaphthylene	152	8.866	8.866	(0.975)	2701962	200.000	180
	46 3-Nitroaniline	138	9.094	9.094	(1.000)	549348	200.000	210
	47 Acenaphthene	154	9.146	9.146	(1.006)	1648463	200.000	190
	48 2,4-Dinitrophenol	184	9.239	9.239	(1.016)	265203	200.000	300
	49 4-Nitrophenol	65	9.373	9.373	(1.031)	383510	200.000	200
	50 Dibenzofuran	168	9.384	9.384	(1.032)	2380681	200.000	180
	51 2,4-Dinitrotoluene	165	9.477	9.477	(1.042)	643843	200.000	210
	52 2,3,4,5-Tetrachlorophenol	232	9.632	9.632	(1.059)	557848	200.000	220
	53 2,3,4,6-Tetrachlorophenol	232	9.653	9.653	(1.061)	528217	200.000	200
	54 Diethylphthalate	149	9.891	9.891	(1.088)	1910197	200.000	180
	55 Fluorene	166	9.912	9.912	(1.090)	1962664	200.000	230
	56 4-Chlorophenyl-phenylether	204	9.943	9.943	(1.093)	906483	200.000	200
	57 4-Nitroaniline	138	10.046	10.046	(1.105)	572192	200.000	200
$	58 2,4,6-Tribromophenol	330	10.315	10.315	(1.134)	303242	200.000	230
*	59 Phenanthrene-d10	188	11.350	11.350	(1.000)	621681		
	60 4,6-Dinitro-2-methylphenol	198	10.098	10.098	(0.890)	369634	200.000	300
	61 N-Nitrosodiphenylamine	169	10.139	10.139	(0.893)	1441356	200.000	180
	62 1,2-Diphenylhydrazine	77	10.181	10.181	(0.897)	2095566	200.000	180
	63 4-Bromophenyl-phenylether	248	10.688	10.688	(0.942)	539491	200.000	190
	64 Hexachlorobenzene	284	10.895	10.895	(0.960)	581783	200.000	200
	65 Pentachlorophenol	266	11.185	11.185	(0.985)	364412	200.000	200
	66 Phenanthrene	178	11.392	11.392	(1.004)	2871313	200.000	220
	67 Anthracene	178	11.464	11.464	(1.010)	2895262	200.000	220
	68 Carbazole	167	11.754	11.754	(1.036)	2838733	200.000	190
	69 Di-n-Butylphthalate	149	12.458	12.458	(1.098)	3186408	200.000	200
	70 Fluoranthene	202	13.286	13.286	(1.171)	2950951	200.000	240
	71 Benzidine	184	13.534	13.534	(0.887)	1713183	200.000	210
*	72 Chrysene-d12	240	15.263	15.263	(1.000)	357835		
	73 Pyrene	202	13.607	13.607	(0.891)	2729353	200.000	200

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (NG)	ON-COL (NG)
$	74 Terphenyl-d14	244	13.886	13.886	(0.910)	1726251	200.000	190
	75 Butylbenzylphthalate	149	14.621	14.621	(0.958)	1010594	200.000	170
	76 3,3'-Dichlorobenzidine	252	15.263	15.263	(1.000)	783655	200.000	210
	77 Benzo(a)Anthracene	228	15.242	15.242	(0.999)	1960776	200.000	190
	78 Bis(2-ethylhexyl)phthalate	149	15.490	15.490	(1.015)	1351209	200.000	160
	79 Chrysene	228	15.304	15.304	(1.003)	1797418	200.000	180
*	80 Perylene-d12	264	17.229	17.229	(1.000)	416323		
	81 Di-n-octylphthalate	149	16.349	16.349	(1.071)	2333973	200.000	180
	82 Benzo(b)fluoranthene	252	16.743	16.743	(0.972)	2436436	200.000	210
	83 Benzo(k)fluoranthene	252	16.784	16.784	(0.974)	1959383	200.000	230
	84 Benzo(a)pyrene	252	17.167	17.167	(0.996)	2003135	200.000	210
	85 Indeno(1,2,3-cd)pyrene	276	18.689	18.689	(1.224)	2303273	200.000	180
	86 Dibenzo(a,h)anthracene	278	18.730	18.730	(1.087)	1854734	200.000	190
	87 Benzo(g,h,i)perylene	276	19.092	19.092	(1.108)	1923577	200.000	190

B-4 Organochlorine Pesticides and PCBs by GC

ORGANOCHLORINE PESTICIDES AND PCBs BY GC
(Methods 608 and 8081A/8082)

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Approved by:

Title: STL-SL Technical Manager Jan 7, 2000
Date

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of various chlorinated pesticides and polychlorinated biphenyls (PCBs) in liquid and soil extracts. Appendix A gives an example of the retention times of the routine target compounds. A summary of the method QC requirements is provided in the Appendix C.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria are given in Section 5 of the current revision of the STL Savannah Laboratories Laboratory Quality Manual.

The procedures for chlorinated pesticides (8081A) and PCBs (8082) are given as separate methods separated in the latest update of SW-846. Until this update, the pesticides and PCBs were combined in both SW-846 Methods 8080 and 8081. The extraction and the analysis are combined in this SOP 1) to reduce the time of extraction and analysis; and 2) to reduce the amount of solvent used in the procedures (one extraction instead of two). If interferences or high levels of non-PCB compounds are present, a portion of the extract will be subjected to the acid cleanup and reanalyzed. Note that if the list of target analytes includes only a limited list of components (i.e. Toxaphene, Chlordane, or PCBs), these procedures may be abbreviated to address only the analytes of interest.

2.0 SUMMARY OF METHOD

2.1 Aqueous and leachate samples are extracted with methylene chloride using a continuous liquid/liquid extractor (SOP EX30) or separatory funnel (SOP EX35). Soils are extracted with 1:1 hexane/acetone or 1:1 acetone/methylene chloride using a sonic dismembrator (SOP EX40). The solvent is evaporated, the residue exchanged into hexane, and the sample adjusted to a final volume of 10mL or less. The preparation may also incorporate Florisil, copper (sulfur), acid (PCBs and Toxaphene only), or gel permeation chromatography (GPC) cleanups. Analysis of the extract is routinely performed on a GC equipped with dual capillary columns (different phases) connected to dual electron capture (EC) detectors, allowing simultaneous detection and confirmation of the target compounds. GC/MS confirmation can also be employed if analyte concentration is sufficiently high or if the sample extract is concentrated to an appropriate final volume. Quantitation may be performed using the external or internal standards calibration technique. The procedures for waste dilution are given in STL-SL SOP EX42.

2.2 This method is based on the guidance in SW-846 Methods 8000B, 8081A, and 8082 and 40 CFR 136 Method 608.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially hazardous situations.

3.2 The analyst should wear an apron or lab coat, gloves, and eye protection when handling extracts. Dilutions should be performed under a hood or in a well-ventilated area.

3.3 The analyst must be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in the analysis of pesticides and PCBs. Many of these compounds are suspected carcinogens.

4.0 INTERFERENCES

4.1 Glassware should be scrupulously cleaned and solvent-rinsed in accordance with STL-SL SOP AN60 to minimize artifacts and/or elevated baselines in gas chromatograms. Any vessel that comes in contact with the extract is a potential source for contamination. Method blanks that are extracted and analyzed with each batch of samples will provide clues to the source of contamination form the glassware and reagents.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. See Section 9 for a table summary of the cleanups that may be employed to eliminate or reduce interferences. If matrix interferences continue after a clean up has been performed, the sample is diluted as needed for data analysis. If a cleanup is used, the method blank must also be subjected to the cleanup.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

Aqueous samples are collected in 1-L glass containers with Teflon-lined caps. Soil/sediment samples are collected in wide mouth glass jars equipped with Teflon lined caps. No preservative is added. The samples are iced at the time of collection and maintained at 4°C (less than 6C with no frozen samples) until extraction. Extraction must be performed within 7 days for aqueous samples and within 14 days of sampling for soils/solids. The extracts must be stored at 4°C (less than 6C) and must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC), temperatures programmable, equipped with single or dual electron capture (EC) detectors and a compatible autosampler. The GC should be capable of housing two columns and have make-up gas lines for each detector

6.2 Data system compatible with the GC, with appropriate software or integration capabilities

6.3 The following column pairs are recommended

DB-5 fused silica capillary column 30 M x 0.53 mm ID x 1.5 um film (J&W or equivalent)
DB-608 fused silica capillary column 30 M x 0.53 mm ID x 0.83 um film (J&W or equivalent)
<Or>
DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-17 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
<Or>
DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-1701 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)

6.4 Microsyringes, appropriate volumes

6.5 Volumetric flasks, Class A, appropriate volumes

6.6 Autosampler vials, septa, and caps; compatible with the autosampler

7.0 REAGENTS

Hexane- pesticide grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with STL-SL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in STL - SL SOP AN43:*Standard Preparation.*

The lab should purchase certified solutions from STL-SL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See STL-SL SOP AN43 for guidance for standard preparation.

Calibration Standard Recipes

The standard concentrations given in Appendix B are provided for guidance. The recipes used for standard preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. Note that the preparation of some of the calibration standards will include intermediate level standards.

The lowest level calibration standard should be at or below the equivalent of the reporting limit as defined in the STL-SL LQM or client QAPP. The remaining standards will define the working range of the analytical system. When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

9.0 SAMPLE PREPARATION

The sample preparation and cleanup procedures are described in the following SOPs:

PROCEDURE	MATRIX	STL-SL SOP
Continuous Liquid-liquid extraction	aqueous and leachates	EX30
Separatory funnel extraction	aqueous and leachates	EX35
Ultrasonic extraction	soils and sediments	EX40
Waste dilution	Waste samples (oils, products, etc)	EX42
Zymark extract concentration	Aqueous, leachate, and soil extracts	EX50

CLEAN-UP PROCEDURE	STL-SL SOP	APPLICATION	EFFECTIVENESS
Florisil	EX62	pest/PCBs	Eliminates polar non-target compounds
Sulfuric acid / permanganate	EX60	PCBs and Toxaphene	Eliminates some unsaturated hydrocarbon interferences
Copper	EX60	pest/PCBs	Eliminates elemental sulfur
GPC	EX61	pest/PCBs	Eliminates high molecular weight non-target compounds and sulfur

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions
The instrument conditions listed in this section are for guidance. The actual conditions used by the lab must be documented in the instrument maintenance log, data system, or run log. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

10.1.1 Two configurations may be used for the analysis of pesticides and PCBs. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column, a two-hole ferrule, or a glass tee to provide simultaneous detection and confirmation of the target analytes. The following stationary phase pairs are recommended:

DB-5 fused silica capillary column 30 M x 0.53 mm ID x 1.5 um film (J&W or equivalent)
DB-608 fused silica capillary column 30 M x 0.53 mm ID x 0.83 um film (J&W or equivalent)

DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-17 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)

DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-1701 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)

A guard column is recommended to help protect the analytical columns.

10.1.2 Example GC Parameters

Injector: 220 – 240°C
Detector: 300 – 320 °C
Carrier Gas Flow: Helium at 5 mL/min (per column)
Make-up Gas Flow: Nitrogen at 25 mL/min (per detector)-see manufacturer's recommended flows

Example chromatogram temperature program:

Initial Temp:	160 C
Initial Hold:	4.0 min
Program Rate:	10 C/min
Final Temp:	270 C (hold for 10 minutes)
Injected Volume:	2-4uL - 1-2uL per column (single injection into guard column and "Y" splitter)

NOTE: These conditions and parameters are given for guidance. The conditions and parameters may be modified to optimize the analytical system.

10.2 Column Evaluation

The column(s) must be evaluated prior to the analysis of the calibration standards. The column evaluation is performed by injecting Endrin and p,p'-DDT and calculating the percent breakdown of these compounds.. The column evaluation does not have to be performed if PCBs only are the target compounds. PCBs are stable and not subject to breakdown in the injection port.

If the instrument has not been in use for more than one day, a "priming" analysis may be beneficial. The analysis of a relatively high concentration pesticide or PCB standard may help to stabilize the response of the very sensitive EC detector. Inject a standard that is about 10x the concentration of the highest calibration standard and allow the instrument to cycle through the temperature program. It is not necessary to acquire the data but the baseline should be monitored before and after the priming analysis to gauge the condition of the detector. A hexane blank should be analyzed after the analysis of the priming standard and before the %breakdown check.

NOTE: The "priming" standard should be injected manually to avoid contaminating the autosampler syringe.

Inject the Endrin/DDT breakdown standard. Check the peak integrations and calculate the breakdown as follows:

$$\%\text{Breakdown Endrin} = \frac{\text{Area(Endrin Aldehyde + Endrin Ketone)}}{\text{Area(Endrin + Endrin aldehyde + Endrin Ketone)}} \otimes 100$$

$$\%\text{Breakdown DDT} = \frac{\text{Area(DDE + DDD)}}{\text{Area(DDT + DDE + DDD)}} \otimes 100$$

The breakdown for each compound must be less than 15%. If the breakdown exceeds 15%, the instrument will require column and/or injector port maintenance. The maintenance may include but is not limited to replacing the septum, clipping the front of the column, and replacing the glass injector sleeve.

10.2 Initial Calibration

The external standard calibration technique is routinely employed for the determination of the concentration of pesticides and PCBs. The lab also has the option of using internal standard calibration. Pentachloronitrobenzene (PCNB) or 2-Nitro-1-bromobenzeneis are suggested for use as internal standards.

10.2.1 Prepare and analyze the calibration standards. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in the SOP Summary.

Note that the following offers two (2) options for calibration and quantitation – average CF or regression curve. Only one need be chosen per analyte.

When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

10.2.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?
>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?
>Are the peaks symmetrical? Is there tailing or fronting?
>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.2.3 Average CF or RF Option:
Calculate the calibration factor (CF) or the response factor (RF) of each calibration standard, the average calibration or response factor and the relative standard deviation using the following equations:

Calibration factor-external standard

$$CF = \frac{Rt}{Ct}$$

where
Rt = response (area or height) of the target compound
Ct = concentration of the target compound, usually expressed as ug/mL

Response factor-internal standard

$$RF = \frac{Ac}{Ais} \otimes \frac{Cis}{Cc}$$

where

Ac = area of the target compound
Ais = area of the internal standard
Cc = concentration or mass on-column of the target compound (ug/mL)
Cis = concentration or mass on-column of the internal standard (ug/mL)

Average Calibration Factor or Response Factor

$$CF_{avg} = \frac{CF_1 + CF_2 + CF_3 + \ldots CFn}{n}$$

Relative Standard Deviation

$$\%\,RSD = \frac{standard\ deviation}{\overline{CF_{avg}}} \otimes 100$$

$$Standard\ \ Deviation = \sqrt{\frac{\sum_{i=1}^{n}(CF_i - \overline{CF_{avg}})^2}{n-1}}$$

Where

CF_i = calibration factor (or response factor) of the individual calibration level
CF_{avg}= average calibration factor (or response factor)

10.2.4 Initial Calibration Criteria:

600-series: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear and the average calibration factor may be used for quantitation.

8000-series: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear and the average calibration factor may be used for quantitation.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average calibration factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required since the lab has demonstrated that the lowest standard in the calibration curve (the equivalent of the RL) can be detected.

10.2.5 Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples. For 8000-series methods, a minimum of five points is required for a linear regression, six points for a second order curve, and seven or more for higher order fits. It is recommended that only linear and quadratic (second order) curves be used for quantitation. See STL-STL SL SOP AN67 for guidance on evaluation of calibration curves.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

10.3 Calibration Verification

Calibration is verified at the frequency given in the SOP Summary. Note that the following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive for the compound(s) failing high are detected, these samples may be reported.

10.3.1 Analyze the mid-level standard(s). Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration:

$$PercentDrift = \left| \frac{Cccv - Ctrue}{Ctrue} \right| \otimes 100$$

where
Cccv = concentration of CCV (ug/mL) quanted from regression curve or CFavg or RFavg
Ctrue = true concentration of the CCV (ug/mL)

$$PercentDifference = \left| \frac{CFccv - CFavg}{CFavg} \right| \otimes 100$$

where
CFccv = calibration factor (or RF) of CCV
CFavg = average calibration factor (or RFavg) from initial calibration

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10.3.2 Continuing Calibration Verification Criteria

Response Criteria:
If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

600-series: If the percent drift or percent difference is less than or equal to 15%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

8000-series: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor is used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average calibration factor or regression curve from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

All samples analyzed must be bracketed by acceptable CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

Retention Time Criteria
The retention time for the CCV must fall within the daily retention time window as defined in STL-SL SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses.*

10.4 Sample Analysis Sequence
The analytical sequences for the 600- and 8000-series methods are given in the SOP Summary in Appendix C.

10.4.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.4.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted in hexane and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve.

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve (i.e., a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client). Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in STL-SL SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses.* If internal standard calibration is used, the determination of absolute retention time windows is not required. Relative retention times, as described in Section 11.1.4, are used to identify the target compounds.

11.0 DATA ANALYSIS/CALCULATIONS

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times), the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration, and the integration of the peaks. Manual integration must be documented in accordance with STL-SL SOP AN65. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data.

The judgement and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?

11.1 Qualitative analysis

Identification of the surrogates and target compounds is based on retention time. The retention time (RT) windows calculated around the CCV retention times are used for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peaks areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks, which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed above (10.4.2).

11.1.1 The surrogates should be evaluated first to check for shifts in retention times and to evaluate the surrogate recovery. The recovery criteria are given in Section 5 of the STL-Laboratory Quality Manual (LQM).

A minimum of two surrogates are spiked into each sample and QC item prior to preparation. 2,3,4,6-tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are the recommended surrogates. DCB should be evaluated as the primary surrogate; TCMX is evaluated if there is matrix interference with DCB.

Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Generally, it is expedient to calculate the recovery of both surrogates on one chromatogram. If these recoveries are deemed acceptable, the other channel is not evaluated for recovery. If interferences are suspected, evaluate recoveries on the other channel. Note that given this nature, an extract is considered acceptable if one of the 4 potentially calculated recoveries is acceptable.

NOTE: If the recovery of the surrogate(s) is above the upper control limit and no target compounds are detected in the sample, results may be reported. Refer to section 13 of the current Laboratory Quality Manual regarding this issue.

11.1.2 Evaluate each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, and the overall shapes of the peaks.

If the concentration is below the detection limit, the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.3.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range, the analysis at a dilution is not necessary.

11.1.3 If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds.

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

If the relative percent difference is less than 40%, the presence of the target compound is confirmed and the higher concentration is reported. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A large relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. If interference is detected, flag the result to note the disparity between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution.

The following table summarizes the general guidance for the evaluating of chromatographic data.

PEAK INFORMATION	ACTION	REPORT(1)
No peaks found on primary or secondary column		Report < RL
Peak found within RTW on primary column	Peak is tentatively identified as the target. If, in the professional judgement of the analyst and supervisor, the peak within the RTW can be reasonably excluded as a target, the result may be reported as a "non detect".	If concentration < RL, report < RL If concentration > RL, evaluate confirmation column.
Peak found within RTW on confirmation column	Peak is tentatively confirmed as the target. If, in the professional judgement of the analyst and supervisor, the peak within the RTW can be reasonably excluded as a target, the result may be reported as a "non-detect".	If concentration < RL, report <RL If concentration >RL, calculate %RPD -if %RPD <=40, report highest concentration of primary and confirmation analyses -if %RPD >40, report the lowest result or the result that is most reasonable for the sample matrix. and flag result to note the disparity Case narrative or note to PM may be required for complex matrices.

(1)RL may be the STL-SL Reporting Limit in Table 5 of the LQM or may be defined by the client QAP or contract.

The analyst must clearly show how the reported sample results were determined. Manual calculations and integrations must be documented as described in STL SOP AN65.

11.1.4 Identification "Tools"

Analysis by GC/MS (scan or SIM) may be used to confirm the presence of the target compounds (see STL-SL SOP SM06: *Guidelines for SIM Analysis by GC/MS.)*

11.1.4.1 Relative Retention Time

The retention time of a surrogate compound provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification. Another useful tool that employs a similar idea to the RRT is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual cues to the identification of the target compound.

11.1.4.2 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience.

11.1.5 Qualitative Analysis of Multiple Peak Compounds

Identification of multicomponent pesticides/PCBs is based on the recognition of their chromatographic patterns. Quantitation is performed using the area of characteristic peaks in the sample and standard using external calibration procedures.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be warranted. Suggested cleanup options are given in Section 4.

11.1.5.1 PCBs as Aroclors

PCBs are generally reported as Aroclors. The Aroclors have varying levels of PCB congeners with the last two numbers in the Aroclor designation indicating the weight percent of chlorine. For example, AR1221 is 21% chlorine by weight; AR1260 is 60% chlorine by weight. The 12- in the Aroclor designation represents the biphenyl molecule. The exception to this naming convention is AR1016, which is about 42% chlorine by weight. (Note that AR1026 and AR1242 have similar chromatograms - both Aroclors have almost the same weight of chlorine by weight and nearly the same PCB congeners.)

Aroclors are identified by matching the pattern of the sample with standards analyzed under the same analytical conditions. Interference may occur due to the presence of non-target analytes or due to "weathering" of the Aroclor in the environment. The presence of multiple Aroclors will also complicate the identification and quantitation of the Aroclors. Many matrix interferences may be reduced or eliminated by treating the sample extract with copper, sulfuric acid, and permanganate prior to analysis. STL SL SOP EX60 details this procedure.

NOTE: Do not use the acid or permanganate cleanup on the entire extract if pesticides are also to be reported as many of the pesticides are not stable in acid or strong oxidizer.

When a pattern matching an Aroclor is encountered, it may be quantitated using either the 3-5 characteristic peaks (recommended) or total area response. Total area quantitation should only be used as detailed below. Residues of either AR1016 or AR1260 are quantitated using the average RF/CF determined during initial calibration. The other Aroclors are quantitated against the RF/CF determined from their single-point analysis during initial calibration. Samples should be diluted when the amount of PCB in a sample extract exceeds the calibration range defined in initial calibration. Note that the AR1660 standard defines the working range for all the Aroclors (i.e. if AR1660 was calibrated from 0.10ug/mL to 5.0ug/ml, and a sample extract was analyzed containing 10ug/ml of AR1232, that extract would require dilution to get the amount of AR1232 to be less than 5.0ug/ml.)

In the 3-5 peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of PCB in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 3-5 resulting concentrations are averaged to provide the final result for the sample.

"Weathering" is the loss of part of the Aroclor pattern due to biological or chemical degradation of individual PCBs. When weathering is suspected, try to match the later eluting peaks first. Flag the results for a weathered Aroclor pattern as tentatively identified and make a note in the case narrative if provided.

The presence of multiple Aroclors can be a problem to identify since most Aroclors have at least a few PCBs in common. The easiest case would be to have early and late eluting Aroclors present. The most difficult cases will involve the presence of Aroclors with the same relative chlorine level. In cases where the identification of Aroclors cannot be clearly determined, quantify the Aroclors against the Aroclor that most closely matches the pattern of the sample using the total area of all peaks within the pattern range. The result should be flagged and noted in the case narrative if provided.

NOTE: When choosing individual peaks for quantitation, compare their responses in the sample and standard. If the peaks chosen do not correlate well (i.e. ratios to other peaks are close) between the sample and standard, review the chromatograms for other possible peaks for quantitation.

11.1.5.2 Toxaphene

Toxaphene is a mixture of chlorinated camphenes, which has a complex and characteristic pattern when analyzed by GC-ECD. A single Toxaphene standard is analyzed during the initial calibration for the purpose of pattern identification in samples. When a Toxaphene residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for Toxaphene should be analyzed. Alternatively, single points may be prepared with Toxaphene concentrations within 2x the Toxaphene quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the Toxaphene standard(s), the samples are re-analyzed using these standard(s) for quantitation. Note that when analysis of Toxaphene-containing samples occurs over an extended time, the calibration factor should be verified or regenerated every 12 hours.

If the sample and standard chromatograms agree well, Toxaphene is quantitated using 5 characteristic peaks (similar to the PCB approach, above). If the sample and standard pattern do not agree as well (i.e. individual peak ratios do not agree as well, but all the major Toxaphene components in the standard are present in the sample), a total area integration is more appropriate. To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the retention times of the first and last eluting Toxaphene components in the standard. Note that in order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample and that extraneous peaks or humps (contributed by non-Toxaphene components) are NOT included in the quantitation.

When Toxaphene is determined using the 5 peak approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionately larger or smaller in the sample compared to the standard.

In the 5-peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 5 resulting concentrations are averaged to provide the final result for the sample.

11.1.5.3 Technical Chlordane –

Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The following components are significant: α and γ Chlordane, trans-Nonachlor, Heptachlor, and Heptachlor-epoxide. The α and γ Chlordane isomers are the most prevalent and their detection as single components is a good indicator that Technical Chlordane may be present.

The following sections discuss three specific options: reporting Technical Chlordane, reporting Chlordane (not otherwise specified-"NOS"), and reporting the individual Chlordane components that can be identified under their individual CAS numbers.

When the GC pattern of the residue resembles that of the Technical Chlordane standard, quantitate Chlordane residues by comparing the area of 3 to 5 major peaks. Heptachlor and heptachlor epoxide should not be included in this quantitation but rather should be quantitated and reported separately.

The GC pattern of a Chlordane residue in a sample may differ considerably from that of the Technical Chlordane standard. In such instances, it may not be practical to relate a sample chromatogram back to the lab's Technical Chlordane Standard. Therefore, depending on the objectives of the analysis, the analyst may choose to report the sum of all the identifiable Chlordane components as "Chlordane (n.o.s.)" under the CAS number 57-74-9. This option should only be used at the direction of or in consultation with a project manager.

The third option is to quantitate the peaks of a-Chlordane and y-Chlordane separately against the appropriate reference materials, and report these individual components.. This option should be used only after consultation with the project manager. Note that quantitation values determined from the individual chlordane isomers will be substantially lower than those reported for the technical product.

When a Technical Chlordane residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for technical chlordane should be analyzed. Alternatively, single points may be prepared with technical chlordane concentrations within 2x the Technical Chlordane quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the technical chlordane standard(s), the samples are re-analyzed using these standard(s) for quantitation. Note that when analysis of technical chlordane-containing samples occurs over an extended time, the calibration factor should be verified or regenerated every 12 hours. Note that these procedures are not necessary if the lab is reporting chlordane as the α and γ chlordane isomers, not as the technical product.

11.2 Calculations-External Standard

Aqueous/Liquid Samples

If the regression curve option is used, the sample concentration is calculated:

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{V}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

If the calibration factor option is chosen for quantitation:

$$Concentration(ug/L) = \frac{response}{CF_{avg}} \otimes \frac{F \otimes DF}{V}$$

where
Response = area (or height) of the target
CF_{avg} = average calibration factor (ICAL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

Soils/Solids Samples

Regression Curve

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
W = weight of sample extracted (kg)
solids = (percent solids)/100

If the calibration factor option is chosen for quantitation:

$$Concentration(ug/kg,dw) = \frac{response}{CF_{avg}} \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
response = area (or height) of the target
CF_{avg} = average calibration factor
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)
W = weight of sample extracted (kg)
solids = (percent solids)/100

11.3 Internal Standard Calibration

Aqueous Samples

$$Concentration(ug/L) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{V} \otimes DF$$

where

Ac = area of the target compound
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
V = volume of sample extracted (l)
DF =dilution factor

Soil Samples

$$Concentration(ug/kg,dw) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{W \otimes solids} \otimes DF$$

where

Ac = area of the target compounds
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
W =weight of sample extracted (kg)
Solids = (percent solids)/100
DF =dilution factor

12. 0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The **analytical batch** is discussed in STL-SL SOP AN02: *Analytical Batching*, and these criteria are summarized in the SOP Summary included in Appendix C. Calculation of QC data is also given in AN02.

12.2 The **method detection limit (MDL)** must be determined annually in each matrix of concern in accordance with STL-SL SOP CA90: *Procedure for Determination of Method Detection Limit (MDL).*

12.3 Each analyst must participate (individually or as part of a work group) in the analysis and evaluation of QC samples to demonstrate minimum proficiency in this procedure. The IDOC samples are processed in the same manner as routine samples and evaluated according to STL-SL SOP CA92.

13.0 PREVENTIVE MAINTENANCE

No items are included in this revision. See Section 10 of the current STL-SL LQM.

14.0 TROUBLESHOOTING

No items are included in this revision.

15.0 REFERENCES

1. *Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

2. *Code of Federal Regulations, Title 40, Part 136*; U.S. Government Printing Office: Washington, DC, July 1, 1988.

3. STL Savannah Laboratories' *Laboratory Quality Manual*, current revision.

Appendix A

TABLE 1

Compound	RT COL 1	RT COL 2
Tetrachloro-m-xylene	5.26	6.65
Alpha-BHC	7.39	7.71
Gamma-BHC (Lindane)	8.63	8.65
Beta-BHC	8.80	8.39
Heptachlor	9.54	10.61
Delta-BHC	9.83	9.19
Aldrin	10.38	11.53
Heptachlor epoxide	11.96	12.51
Gamma-Chlordane	12.31	13.04
Alpha-Chlordane	12.72	13.41
Endosulfan I	12.86	13.42
4,4'-DDE	13.39	13.84
Dieldrin	13.63	14.05
Endrin	14.45	14.53
4,4'-DDD	14.75	14.83
Endosulfan I	14.97	14.78
4,4'-DDT	15.54	15.78
Endrin aldehyde	15.76	15.21
Endosulfan sulfate	15.98	15.71
Dibutyl chlorendate	16.73	17.80
Methoxylchlor	17.60	17.04
Endrin ketone	17.79	16.82
Decachlorobiphenyl	21.36	22.36
Technical chlordane	MR	MR
Toxaphene	MR	MR
AR 1221	MR	MR
AR1232	MR	MR
AR1016	MR	MR
AR1242	MR	MR
AR1248	MR	MR
AR1254	MR	MR
AR1260	MR	MR
Isodrin	11.85	12.45
Chlorobenzilate	14.51	14.79
AR1268	MR	MR

MR = multi-peak/multi-response compounds COL1 = DB608 COL2 = DB-5

Appendix B:CHLORINATED PESTICIDES AND PCBs STANDARDS

Routine Targets

Target compound	ISMA-1 (ug/mL)	ISMA-2 (ug/mL)	ISMA-3 (ug/mL)	ISMA-4 (ug/mL)	ISMA-5 (ug/mL)
TCMX, DCB (surr)	0.0025	0.0050	0.010	0.020	0.040
g-BHC(Lindane), Heptachlor, Heptachlor epoxide, Endosulfan I	0.0050	0.010	0.020	0.030	0.050
Dieldrin, p,p'-DDT, Endosulfan II, Endrin aldehyde, Methoxychlor	0.010	0.020	0.040	0.060	0.10

Target compound	ISMB-1 (ug/mL)	ISMB-2 (ug/mL)	ISMB-3 (ug/mL)	ISMB-4 (ug/mL)	ISMB-5 (ug/mL)
TCMX,DCB (surr)	0.0025	0.0050	0.010	0.020	0.040
a-BHC, b-BHC, d-BHC, a-Chlordane, g-Chlordane,	0.0050	0.010	0.020	0.030	0.050
p,p'-DDE, Endrin p,p'-DDD, Endosulfan sulfate, Endrin ketone	0.010	0.020	0.040	0.060	0.10

DDT/Endrin Breakdown Evaluation Standard

Pesticide Evaluation Standard	CONC (ug/mL)
Endrin,	0.020
P,P'-DDT	0.020

Appendix IX Targets

Target compound	ISMB-1 (ug/mL)	ISMB-2 (ug/mL)	ISMB-3 (ug/mL)	ISMB-4 (ug/mL)	ISMB-5 (ug/mL)
TCMX,DCB (surr)	0.0025	0.0050	0.010	0.020	0.040
Isodrin	0.0050	0.010	0.020	0.030	0.050
Chlorobenzilate	0.050	0.10	0.20	0.50	1.0
Kepone	0.025	0.050	0.10	0.20	0.50

APPENDIX B

Technical Chlordane Five-point Curve

STOCK STANDARD	TCHLOR -1*	TCHLOR -2*	TCHLOR -3*	TCHLOR -4*	TCHLOR -5*
Technical Chlordane	0.100	0.20	0.40	0.60	0.80
DCB, TCMX (surr)	0.0025	0.0050	0.010	0.020	0.040

Toxaphene Five-point Curve- 0.10, 0.25, 0.50, 1.0, 2.0ug/mL

STOCK STANDARD	TOX -1*	TOX -2*	TOX -3*	TOX -4*	TOX -5*
Toxaphene	0.10	0.25	0.50	1.0	2.0
DCB, TCMX (surr)	0.0025	0.0050	0.010	0.020	0.040

PCBs as Aroclors

AR1660 Standards

Calibration Std	AR1016(ug/mL)	AR1260(ug/mL)	surrogates
AR1660-1	0.10	0.10	0.0025
AR1660-2	0.20	0.20	0.0050
AR1660-3	0.50	0.50	0.010
AR1660-4	1.0	1.0	0.020
AR1660-5	2.0	2.0	0.040

Single Point Aroclor Calibration Standards

Calibration Standard	Single Pont Concentration (ug/mL)	Surrogate Concentrations (ug/mL)
AR1221	1.0	0.020
AR1232	1.0	0.020
AR1242	1.0	0.020
AR1248	1.0	0.020
AR1254	1.0	0.020

If required, five point curves for AR1221, AR1232, AR1242, AR1248, and AR1254 are prepared at the same concentrations as the AR1660 curve.

Appendix C

608/8081/8082 METHOD SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	None; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days (after leaching procedure)	40 days

*Storage temperature is 4C with a control criteria of less than 6C with no frozen samples

EXTRACTION

Aqueous: Approximately 1L of sample (contents of container) using continuous or separatory funnel extraction at pH 5-9 with methylene chloride; exchange to hexane and concentrate to final volume of 10mL

Soil/Solids: Approximately 30g of sample using sonication with 1:1 acetone/hexane or 1:1acetone/methylene chloride; Concentrate to final volume of 10mL in hexane

Wastes: Approximately 1g of sample diluted to final volume of 10mL with hexane

ANALYSIS

Dual capillary columns with dual EC; 2-5uL injection into glass tee or y-splitter; external or internal standard calibration

SEQUENCE-600-series

Endrin/p,p'-DDT breakdown evaluation (daily-every 24 hours)
Initial Calibration- 3 point single peaks compounds 1 point all Aroclors 1 point toxaphene 1 point technical chlordane
Initial Calibration Verification (ICV)
Sample analyses
Continuing calibration verification (CCV)-daily-every 24 hours Single peak compounds AR1660
RL standard(optional; required by state or client QAP)
Sample analyses

Sequence continues until all samples have been analyzed or the CCV fails the acceptance criteria. If a multi-peak target compound is detected, the extract is reanalyzed with a 3-point curve.

SURROGATE(S):
Tetrachloro-m-xylene- 0.20ug/L
Decachlorobiphenyl-0.20ug/L

BATCH QC
Method blank
LCS/LCSD- full target of single peak analytes @ 0.20ug/L
MS/MSD- full target of single peak analytes @ 0.20ug/L

Appendix C

SEQUENCE-8000-series

STANDARD/SAMPLES
Endrin/p,p'-DDT breakdown (every 12 hours)
Initial Calibration- 5 point single peaks 5 point Ar1660(note1) 1 point Toxaphene 1 point tech Chlordane 1 point remaining Aroclors
Up to twenty sample extracts
Endrin/p,p'-DDT breakdown (every 12 hours)
Continuing calibration check midpoint single peaks and midpoint Ar1660(note1)
RL Standard(optional)-lowest point on the calibration curve if required by state or client QAP
Up to twenty sample extracts
Continuing calibration check-midpoint single peaks and midpoint Ar1660(note1)

The sequence continues until all samples have been analyzed or until the calibration verification fails the acceptance criteria. All samples extract analyses must be bracketed by acceptable verification standards.

Note 1-A mixture of Ar1016 and Ar1260 will be used to calibrate and verify the response for PCBs and to verify the response for Toxaphene and technical Chlordane in the continuing calibration.

SURROGATE (S):
Tetrachloro-m-xylene- 0.20ug/L
Tecachlorobiphenyl-0.20ug/L

BATCH QC
Method blank
LCS- LQM subset
MS/MSD- LQM subset

Parameter	Aqueous(ug/L)	Soils(ug/kg)
Lindane	0.20	6.0
Aldrin	0.20	6.0
Heptachlor	0.20	6.0
Dieldrin	0.50	15
Endrin	0.50	15
p,p'-DDT	0.50	15

Appendix C

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
p,p'-DDT and Endrin breakdown check -required for 8000-series, recommended for 600-series	Initially and every 12 hours	% breakdown of both compounds less than 20%	-re-analyze check solution -perform injector port and/or column maintenance and re-analyze
Initial Calibration- 600-series:3 point minimum with lowest point at RL 8000-series:5 point minimum with lowest point at RL	Initially prior to sample analysis, when major instrument maintenance performed, or when CCV fails	**600-series:** 1) RSD of each target <= 10%; OR 2) plot regression curve CC>=0.99 for each target **8000-series:** 1) RSD of each target <= 20%; OR 2) plot regression curve CC>=0.99 for each target (see previous page for exceptions)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification(CCV)	After every ten to twenty sample analyses and at the end of the sequence if external standard calibration is used	**600-series:** <=10% Difference/Drift **8000-series:** Percent difference or drift <= 15% (see previous page for exceptions) Retention time of CCV must fall within the daily retention time window	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards

Appendix C

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Method blank	Per batch	All targets reported less than RL in Section 5 of the STL-SL LQM	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in LQM
Lab control sample (LCS)- Subset of targets in STL SL LQM	Per batch (If MS/MSD cannot be performed , the LCS must be performed in duplicate)	Recoveries within STL-SL LQM Section 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in LQM
Matrix spike(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within STL-SL LQM Section 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in LQM
Internal Standard Response	All samples, method blanks, and QC	Response with a factor of two from the midpoint standard in the initial calibration sequence	-Evaluate chromatogram and integrations. -Reanalyze or dilute and reanalyze -Flag data
Surrogates	All samples, method blanks, and QC	Recoveries within STL-SL LQM Table 5 limits See section 11.1.1. for specifics	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in LQM
Reporting limit(RL) standard-standard at the reporting limit used to verify sensitivity of the instrument	Daily(optional-see specific state or client requirements for frequency)	Detected with reasonable sensitivity	-Reanalyze RL standard -Remake and reanalyze RL standard -Perform instrument or column maintenance, recalibrate, and reanalyze associated samples

STL Savannah Laboratories

Appendix C

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Retention time window determination	See guidance in STL-SL SOP AN66	See guidance in STL-SL SOP AN66	Use guidance in STL-SL SOP AN66: *Determination of Retention Time Windows in Gas Chromatographic Analyses*
Initial demonstration of Capability (the analyst has to perform the IDOC for either of the analogous 600 or 8000 series methods- not both)	Per work group or analyst	Within the 600- or 8000-series method limits (see STL-SL SOP CA92)	-Reanalyze QC sample for the targets that failed to meet the criteria (see STL-SL SOP CA92)
Method detection limit(MDL)	See STL-SL SOP CA90	Evaluate data using criteria STL-SL SOP CA90	-Evaluate data. Check calculations. -Reanalyze MDL samples.

B-5 Polychlorinated Biphenyls (PCBs) by GC/MS (Method 8082)

Approval Signature: R. Wayne Robbins

Title: Corporate QA Manager Date: July 15, 1999

Polychlorinated Biphenyls (PCBs) by GC/MS (Method 680)

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of polychlorinated biphenyls (PCBs) in groundwater, soils, sediments, wastes, and biological tissues by GC/MS. PCBs are reported by the level of chlorination: monochlorobiphenyls, dichlorobiphenyls, trichlorobiphenyls, etc., up to decachlorobiphenyl.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits for each target compound is given in Section 5 of the current revisions of the Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is spiked with a surrogate and extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS operated in the Selected Ion Monitoring Mode (SIM). Windows are established to monitor for the characteristic masses of the various PCB congeners. Qualitative identification of the target compounds in the extract is based on the presence of the peak within the SIM window and the mass ratio between the primary and confirmation ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion. Results are reported as total monochlorobiphenyls, total dichlorobiphenyls, etc.

The default identification and quantitation procedure will be to use only the quantitation and confirmation ions for the PCB congeners. Interference check ions, as described in Section 11.2.3, will not be used routinely to evaluate peaks as PCB congeners unless specified in a client QAP or agency requirement or the sample concentration is near a critical quantitation limit. Samples evaluated according to the default quantitation procedures may be slightly high biased.

2.2 This procedure is based on the guidance provided in EPA Method 680. The method has been modified to include the use of a carbon-13 labeled analogue of decachlorobiphenyl (13C12-DCB) as the surrogate in place of the labeled BHC and DDT compounds. 13C12-DCB can be subjected to the optional acid cleanup (the unlabeled analogue, decachlorobiphenyl, is routinely used in SW-846 Method 8082). A window-defining mix containing the first and last eluting isomers of each level of chlorination is used as an aid to establish and verify that the SIM windows are properly set. Preparation procedures are also referenced for soils and biological tissues, which are not included in EPA Method 680.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated area.

3.2 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products such as diesel or waste oil. GPC cleanup may be necessary for biological tissues.
Acid cleanup may be employed as an additional cleanup tool.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	Preservative/ Storage	Container	Sample Hold Time[1]	Extract Hold Time[1]
Aqueous	none; 4C +/- 2C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C +/- 2C	500-mL	14 days	40 days
Biological	Frozen	Glass or wrapped in aluminum foil	6 months	40 days

[1] Holding times are advisory - no holding times are defined in method 680.

6.0 APPARATUS AND MATERIALS

6.1 GC/MS System with compatible data system autosampler, splitless injector, and direct capillary interface.

6.2 Recommended Capillary column-HP-5MS, 30m x 0.25mm ID, 0.50um film thickness. Equivalent columns can be used.

6.3 Microsyringes-

6.4 Volumetric flasks, Class A-appropriate volumes

6.5 Analytical balance

7.0 REAGENTS

7.1 Hexane

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in SL SOP AN43:*Standard Preparation.*

The lab should purchase certified solutions from SL-approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SL SOP AN43 for guidance for standard preparation from neat materials.

8.1 The recommended calibration standards are listed in Appendix A, Table 1. Prepare these standards at the stated concentrations in hexane.

8.2 The surrogate compound, 13C12-Decachlorobiphenyl, is prepared at a concentration of 1.0ug/mL and 1.0mL of this solution is spiked into all samples and QC items prior to extraction.

8.3 The matrix spiking solution contains one PCB congener of each chlorination level except for the nonachlorobiphenyls. The solution is prepared at the indicated concentrations in acetone and 1.0mL of this solution is spiked into all lab spikes and matrix spikes.

COMPOUND	CONCENTRATION (ug/mL)
2-Chlorobiphenyl	2.0
2,3-Dichlorobiphenyl	2.0
2,4,5-Trichlorobiphenyl	2.0
2,2',4,6-Tetrachlorobiphenyl	4.0
2,2',3,4,5'-Pentachlorobiphenyl	4.0
2,2',4,4',5,6'-Hexachlorobiphenyl	4.0
2,2',3,4',5,6,6'-Heptachlorobiphenyl	6.0
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	6.0
Decachlorobiphenyl	10

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP Number	Extraction Technique
Aqueous	EX30	Continuous Liquid-liquid Extraction
Aqueous	EX35	Separatory Funnel
Soils/Sediments and Biological	EX40, PS15	Sonication

9.2 The sample concentration procedures are given in SL SOP EX 50: Zymark Nitrogen Concentration.

9.3 Gel permeation chromatography (SL SOP EX61) may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products. Acid cleanup (SL SOP EX60) is recommended as a routine cleanup prior to analysis. Sulfur cleanup may be necessary if the sample extract contains high levels of sulfur.

10.0 PROCEDURE

10.1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:

HP-5MS 30m x 0.25mm ID, 0.50um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:

Initial column temperature: 45 C for 1 minutes
Column temperature program 1 : 20C per minute to 150C, hold 1 minute
Column temperature program 2 : 10C per minute to 310C, hold until DCB and 13C12-DCB elute

GC injector parameters

Injector temperature : 250-260 °C
Injector : splitless
Inlet purge time : 0.8 minutes
Injector liner : 4mm ID quartz or 4mm glass, deactivated
Sample injection volume : 2uL

Mass Spectrometer and interface parameters

Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: SIM (see Table 3 in Appendix B for ions to monitor)
Mass range for DFTPP analysis: 35-500amu at 1 scan per second or less.

10.2 Tune Criteria

Twenty nanograms of DFTPP are analyzed at the beginning of each 12 hour clock as a check on the "tune" of the mass spectrometer. The DFTPP analysis is performed using scan analysis and the same tune parameters are used for the SIM analysis of the calibration standards and samples.

10.2.1 Prepare a 10ng/uL solution of DFTPP column evaluation standard. The standard must also contain p,p'-DDT (4,4'-DDT).

10.2.2 Analyze a 1uL aliquot of the 10ng/uL DFTPP solution using the same temperature program that is used for SIM analysis of the calibration standards, samples, and QC samples.

10.2.3 Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.3 Window-Defining Solution and SIM Parameters

10.3.1 Analyze 1uL of the window-defining solutions in the scan mode from 45amu to 500amu at > 1 scan per second. Use the same temperature program that will be used for the SIM analysis of PCBs. The window defining solutions may be analyzed separately or may be combined into a single solution.

10.3.2 Determine the retention times of the first and last eluting congeners at each level of chlorination. The quantiation and confirmation masses are listed in Table 3 of Appendix B.

10.3.3 Set the SIM parameters as follows. Refer to Table 3 of Appendix B for the ion sets.

-Begin data acquisition with ion set #1 before the elution of PCB congener #1, Stop the acquisition of ion set #1 and begin acquisition of ion set #2 approximately 10 seconds before the elution of PCB congener #104.
-Stop the acquisition of ion set #2 and begin the acquisition of ion set #3 approximately 10 seconds after the elution of PCB congener #77..
-Stop the acquisition of ion set #3 and begin the acquisition of ion set #4 approximately 10 seconds after the elution of 4,4'-DDT. (the retention time of 4,4'-DDT is determined from the scan analysis of the DFTPP solution that is analyzed at the beginning of each 12-hour clock.).
-Stop the acquisition of ion set #4 and begin the acquisition of ion set #5 approximately 10 seconds before the elution of PCB congener #208.

10.4 Initial Calibration
After the SIM windows are established and verified and the DFTPP criteria has been met, the initial calibration standards are analyzed. Note that a single PCB congener of each chlorination level is used for calibration and quantiation. Decachlorobiphenyl is used to quantify nonachlorobiphenyls.

10.4.1 Prepare the initial calibration standards. The lowest calibration standard should be at the RL and the rest of the standards will define the working range.

10.4.2 Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts. The routine volume is 1uL.

10.4.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.

10.4.4The relative response factor for each compound is calculated as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion of the calibration congener
Ais = area of the characteristic ion for Chrysene-d10
Cx = concentration of the compound being measured (ug/mL)
Cis = concentration of the internal standard (40ug/mL)

NOTE: Use Chrysene-d10 as the internal standard unless matrix interferences are encountered. If phenthrene-d10 must be used, the calibration must be re-evaluated and verified using the second internal standard

10.4.5 Calculate the average relative response factor (RRF_{avg}) for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3.... + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

10.4.6Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_1} (RRF_i - RRF_{avg})^2}{n-1}}$$

10.4.7Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards.

$$\%RSD = \frac{SD}{RRF_{avg}} x\ 100$$

- If the %RSD of each target compound is less than or equal to 20%, the average response factor can be used for quantitation of samples.
- If the %RSD of the target compound is greater than 20%, the curve should be evaluated for errors and one or more standards re-analyzed. Take corrective action until the %RSD of each target is less than 20%.

10.4.8 Performance Criteria
In addition to meeting the calibration criteria, the following performance criteria must also be met for the mid-level standard.

-Mass abundance ratios of all calibration congeners within acceptance range
(see Table 4,Appendix B)
-Baseline separation of PCB congener #87 from congeners #154 and #77
-Signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241
-decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498

10.5 Continuing Calibration Verification
Samples are analyzed only after the DFTPP criteria and the calibration acceptance criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the DFTPP and a mid-level calibration standard prior tot he analysis of samples and after the samples by the analysis and evaluation of a mid-level standard.

10.5.1 The percent difference or percent drift between the continuing calibration RRF and the average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$\%difference = \left| \frac{RRF - RRFavg}{RRFavg} \right| \otimes 100$$

where
RRF = relative response factor from CCV
RRFavg = average relative response factor from initial calibration curve

If the percent difference is less than or equal to 20% for each target compound, the initial calibration is verified.

If the continuing calibration criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the analysis of the continuing calibration standard must be repeated. The analyst must be aware of the 12-hour clock-DFTPP criteria must be met prior to the analysis of the calibration standards. If the continuing calibration standard repeatedly fails the calibration verification criteria, the initial calibration curve must be reanalyzed and reevaluated.

The performance criteria given in Section 10.4.8 must also be met prior to the analysis of samples.

10.6 Sample Analysis
Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.6.1 Add 30uL of the internal standard mix (25ug/mL) to each 1-mL aliquot of the sample extract. The concentration of the internal standard in the extract is 0.75 ng/uL.

10.6.2 Mix the contents of the autosampler vial by inverting several times.

10.6.3 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

10.6.4 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100uL of a sample extract is diluted to final volume of 1.0mL, the dilution factor is 10. (1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

uL extract-Vext	uL MeCl2	volume of dilution (Vdil-uL)	uL ISTD (25ug/mL)-Vistd	DF
1000	0	1000	30	1
500	500	1000	15*	2
200	800	1000	24	5
100	900	1000	27	10
50	950	1000	28.5	20
20	980	1000	30	50

*assumes dilution of a 1.0mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 0.75ug/mL using 30ul of a 25.0ug/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 0.75ug/mL. The following equation can be used to determine the volume of the 25.0 ug/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$Vistd(uL) = 30uL - \left(\frac{Vext}{Vdil} \otimes 30ul\right)$$

Vistd = volume of 25.0ug/mL internal standard to add to the diluted extract (uL)
Vext = volume of extract used to prepare the dilution (uL)
Vdil = final volume of the dilution (uL)-1000uL (1.0mL)

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

The default procedure will be to use only the quantitation and confirmation ions for identification and quantification of PCB congeners. Interference check ions, as described in Section 11.2.3, will not be used routinely to evaluate peaks as PCB congeners unless specified in a client QAP or agency requirement or the sample concentration is near a critical quantitation limit.

11.1.1 Examine the Selected Ion Current Profiles (SICP) for the internal standards. Confirm that the RT and response of the internal standards are within the acceptance criteria specified in the SOP Summary. If the internal standard retention times have changed significantly or the peaks cannot be located, stop and analysis and correct the problem. Reanalyze any associated samples.

11.1.2 Evaluate the peaks for candidates to be identified as PCBs. A peak is tentatively identified as a PCB if

-The peak falls within the retention time range bordered by the first and last eluting isomer of that chlorination level

-The ratio of the quantitation and confirmation ions are present and the area ratios fall within the acceptance criteria in Appendix B, Table 4. The scans must maximize within one scan of each other. Examine the data for the presence of a coeluting PCB of higher chlorination if both ions and the M-70 ions are present and the ratio does fall within the acceptance limits.

-The areas for the quantiation and confirmation ions must be greater than three times the background noise and must fall within the working range of the calibration curve (must not saturate the detector

-At least one ion in the M-70 cluster must be present

11.1.3 Evaluate each PCB candidate in the Cl-3 to Cl-7 range for the presence of coeluting PCBs containing one or two additional chlorines. An intense M+35 ion at the retention time may indicate a PCB with one additional chlorine and the presence of an intense M+70 would indicate a co-eluting PCB containing two additional chlorines. Use the information in Tables 5 and 6 of Appendix B to correct for the interfering ion(s).

For example, if a Cl-7-PCB and a Cl-5-PCB coelute, the Cl-7-PCB will contribute to the quantiation and confirmation ions for the Cl-5-PCB. Cl-7-PCB produces a cluster of three ions by the loss of two chlorines-ions 322,324, and 326. Two of these ions-324 and 326-are also ions contained in the molecular ion cluster of Cl-5-PCB. To determine the ion 326 and 324 areas produced only by the Cl-5-PCB, calculate the contribution to each and subtract it from the measured areas. See Tables 5 and 6 in Appendix B for the percentage of the interference peak to subtract from the quantiation and confirmation ions. In this example, 164% of the area measured for ion 322 should be subtracted from the area measured for ion 324 and 108% of the area measured for ion 322 should be subtracted from ion 326.

NOTE: A coeluting PCB with one more chlorine will affect only the quantiation ion (Table 6). The interference from a coeluting PCB containing one more chlorine, due to the natural abundance of 13C12, is small and can usually be neglected except when measuring the area of a small amount of a PCB coeluting with a large amount of a another PCB containing one more chlorine.

11.2 Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples.

11.2.1 Aqueous Samples

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where

Ax =	sum of areas of the characteristic ion of the PCB chlorination level being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Vqap}{V} \otimes DF$$

where

F =	final volume of extract (mL)
Fqap =	1.0mL
Vqap =	1.0L
V =	volume of sample extracted
DF =	dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

11.2.2 Soils

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	sum of areas of the characteristic ion of the PCB chlorination level being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100
DF =	dilution factor

The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Wqap}{(W)(solids)} \otimes DF$$

where

F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100

The SL CQAP assumes Wqap = 30g, solids = 1, Fqap = 1.0mL, and DF = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching*. SL SOP AN02 also describes the procedure for evaluating batch-specific QC. The attached SOP summary and Table 13.1 in the SL Corporate QA Plan provide guidance for evaluating sample data.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each laboratory must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration are the preparation and analysis of spiked reagent water. The criteria for IDOC accuracy and precision are the accuracy and precision criteria listed in Table 5 of the SL QAP.

12.3 Method Detection Limit

The method detection limit is determined by each lab annually in accordance with SL SOP CA90:*Procedure for Determination of Method Detection Limit (MDL)*.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. See Section 10 of the current SL Quality Assurance Plan.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time.

15.0 REFERENCES

15.1 *Savannah Laboratories' Comprehensive Quality Assurance Plan* and Savannah Laboratories' *Corporate Quality Assurance Plan*, current revisions.

15.2 *Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry*. November 1985. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH

APPENDIX A

TABLE 1 CALIBRATION STANDARDS

CALIBRATION COMPONENTS	CAL 1	CAL 2	CAL3	CAL4	CAL5
Calibration Congener					
2-chlorobiphenyl (1)	0.10	0.50	1.0	2.0	5.0
2,3-dichlorobiphenyl(5)	0.10	0.50	1.0	2.0	5.0
2,4,5-trichlorobiphenyl(29)	0.10	0.50	1.0	2.0	5.0
2,2',4,6-tetrachlorobiphenyl(50)	0.20	1.0	2.0	4.0	10
2,2',3,4,5'-Pentachlorobiphenyl (87)	0.20	1.0	2.0	4.0	10
2,2',4,4',5,6'-hexachlorobiphenyl(154)	0.20	1.0	2.0	4.0	10
2,2',3,4',5,6,6'-heptachlorobiphenyl(188)	0.30	1.5	3.0	6.0	15
2,2',3,3',4,5,5',6,6'-octachlorobiphenyl(200)	0.30	1.5	3.0	6.0	15
Decachlorobiphenyl	0.50	2.5	5.0	10	25
Retention Time Congeners					
3,3',4,4-tetrachlorobiphenyl(77)	0.20	1.0	2.0	4.0	10
2,2',4,6,6'-Pentachlorobiphenyl (104)	0.20	1.0	2.0	4.0	10
2,2',3,3',4,5,5',6,7'-nonachlorobiphenyl(208)	0.40	2.0	4.0	8.0	20
Surrogate					
13C12-Decachlorobiphenyl	0.50	2.5	5.0	10	25
Internal Standards					
Phenathrene-d10	0.75	0.75	0.75	0.75	0.75
Chrysene-d12	0.75	0.75	0.75	0.75	0.75

TABLE 2 –First and Last Eluting Isomers

Congener	First Eluting Isomer	Last Eluting Isomer
Cl-1	2-chlorobiphenyl	4-Chlorobiphenyl
Cl-2	2,6-dichlorobiphenyl	4,4'-dichlorobiphenyl
Cl-3	2,2',6-trichlorobiphenyl	3,4,4'-trichlorobiphenyl
Cl-4	2,2',6,6'-tetrachlorobiphenyl	3,3',4,4'-tetrachlorobiphenyl
Cl-5	2,2',4,6,6'-pentachlorobiphenyl	3,3',4,4',5-pentachlorobiphenyl
Cl-6	2,2',4,4',6,6'-hexachlorobiphenyl	3,3',4,4',5,5'-hexachlorobiphenyl
Cl-7	2,2',3,4',5,6,6'-heptachlorobiphenyl	2,3,3',4,4',5,5'-heptachlorobiphenyl
Cl-8	2,2',3,3',5,5',6,6'-octachlorobiphenyl	2,3,3',4,4',5,5',6-octachlorobiphenyl
Cl-9	2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl

APPENDIX B
SIM IONS

Table 3-Ions for SIM Acquisition

ION Set 1 (a)	ION Set 2 (b)	ION Set 3 (c)	ION Set 4 (d)	ION Set5 (e)
152	186	248	240	356
153	188	249	241	358
186	220	254	288	360
187	222	256	290	390
188	254	288	322	392
189	255	290	324	394
190	256	322	326	424
220	258	323	356	425
221	288	324	357	426
222	289	326	358	428
224	290	328	360	430
255	292	357	362	432
256	294	358	391	462
258	323	360	392	464
290	324	362	394	466
292	326	392	396	496
294	328	394	398	498
	358	396	428	499
	360	398	430	500
	362		432	502

(a) Cl-1 to Cl-4 and Phenthrene-d10
(b) Cl-3 to-Cl-6
(c) Cl5 to Cl-7
(d) Cl-6 to Cl-8 and Chrysene-d12
(e) Cl-8 to Cl-10 and 13C12-DCB

TABLE 4-Approximate Retention Times for PCB Isomer Groups and Calibration Congeners

PCB Isomer Group	Approximate RRT Range	Calibration Congener	Approximate Calibration Congener RRT
Cl-1	0.30-0.35	2-chlorobiphenyl (1)	0.30
Cl-2	0.38-0.50	2,3-dichlorobiphenyl(5)	0.43
Cl-3	0.46-0.64	2,4,5-trichlorobiphenyl(29)	0.54
Cl-4	0.55-0.82	2,2',4,6-tetrachlorobiphenyl(50)	0.56
Cl-5	0.64-0.92	2,2',3,4,5'-entachlorobiphenyl (87)	0.80
Cl-6	0.75-1.1	2,2',4,4',5,6'-hexachlorobiphenyl(154)	0.82
Cl-7	0.88-1.2	2,2',3,4',5,6,6'-heptachlorobiphenyl(188)	0.88
Cl-8	0.99-1.21	2,2',3,3',4,5,5',6,6'-octachlorobiphenyl(200)	1.03
Cl-9	0.16-1.28	Decachlorobiphenyl	1.3
Cl-10	1.3	Decachlorobiphenyl	1.3

RRT = retention time relative to Chrysene-d12

Table 4 Quantitation and Interference Check Ions

PCB Isomer Group	Quant ION	Confirmation ION	Expected Ratio(a)	Acceptable Ratio(a)	M-70 Confirmation ION	Interference Check ION M+70	Interference Check ION M+35
Cl-1	188	190	3.0	2.5-3.5	152	256	222
Cl-2	222	224	1.5	1.3-1.7	152	292	256
Cl-3	256	258	1.0	0.8-1.2	186	326	290
Cl-4	292	290	1.3	1.1-1.5	220	360	326
Cl-5	326	324	1.6	1.4-1.8	354	394	360
Cl-6	360	362	1.2	1.0-1.4	288	430	394
Cl-7	394	396	1.0	0.8-1.2	322	464	430
Cl-8	430	428	1.1	0.9-1.3	356	498	464
Cl-9	464	466	1.3	1.1-1.5	390		498
Cl-10	498	500	1.1	0.9-1.3	424		
Chrysene-d12	240	241	5.1	4.3-5.9			
Phenathrene-d10	188	189	6.6	6.0-7.2			
13C12-DCB (surrogate)	510	512					

(a) ratio of quantitation ion to confirmation ion

TABLE 5-Corrections for Interference of PCB Containing Two Additional Chlorines

PCB Isomer Group	Quant ION	Confirmation ION	Ion Measured to Determine Interference	Percent Ion area to be subtracted from QUNAT ION Area	Percent Ion area to be subtracted from CONFIRMATION ION Area
Cl-3	256	258	254	99	33
Cl-4	292	290	288	65	131
Cl-5	326	324	322	108	164
Cl-6	360	362	356	161	71
Cl-7	394	396	390	225	123

TABLE 6-Corrections for Interference of PCB Containing One Additional Chlorine

PCB Isomer Group	Quant ION	Ion Measured to Determine Interference	Percent Ion area to be subtracted from QUNAT ION Area
Cl-2	222	221	13.5
Cl-3	256	255	13.5
Cl-4	292	289	17.4
Cl-5	326	323	22.0
Cl-6	360	357	26.5
Cl-7	394	391	30.9
Cl-8	430	425	40.0

APPENDIX C
680 SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Biological	Frozen	Glass or aluminum foil	6 months	40 days

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
DFTPP 10ng on column Clock starts at injection	DFTPP 10ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification
Samples and the capping standard must be analyzed within 12 hours of the start of the clock	Samples and the capping standard must be analyzed within 12 hours of the start of the clock
Capping standard	Capping standard

DFTPP CRITERIA

m/z	Ion Abundance Criteria
127	40-60% of mass 198
197	<1.0% 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 mass443
442	>40% of mass 198
443	17-23% of mass 442

ANALYTICAL BATCH
SEE SL SOP AN02.

CALIBRATION ACCEPTANCE CRITERIA

Initial Calibration	Continuing Calibration
RRFavg <= 20% RSD	Percent difference <= 20% difference from initial calibration

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 20ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
Initial Calibration-minimum of five calibration standards	After Tune Check and when calibration verification standard fails acceptance criteria.	%RSD <= 20%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Performance Criteria	Evaluate mid level calibration standard each clock	-Mass abundance ratios of all calibration congeners within acceptance range (see Appendix B) -Baseline separation of PCB congener #87 from congeners #154 and #77 -Signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241 -decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples and at the end of the analytical sequence	%Difference <= 20%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate

QC Item	Frequency	Acceptance Criteria	Corrective Action
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be within 30% of the previous CCV and within 50% of the initial calibration Areas in samples should be evaluated for gross error . Consult superior	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within Section 5 QAP limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SL SOP AN02 and SL QAP Table 13.1
Lab Control Standard (LCS) - QAP subset	See AN02	All spiked targets within the accuracy criteria in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SL SOP AN02 and SL QAP Table 13.1
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See AN02	All targets within the accuracy and precision criteria in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SL SOP AN02 and SL QAP Table 13.1

QC Item	Frequency	Acceptance Criteria	Corrective Action
Initial Demonstration of Capability (IDOC)	Per analyst	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	See CA90	Evaluate according to SL SOP CA90	Evaluate according to SL SOP CA90

B-6 Chlorinated Herbicides

Approval Signature: R. Wayne Hobbins

Title : Corporate QA Manager Date : 1/14/99

CHLORINATED HERBICIDES (615 and 8151A)

1.0 SCOPE AND APPLICATION

1.1 This procedure can be used to determine the concentration of various chlorinated herbicides in water, groundwater, leachate, soil, sediment, and waste samples extracts. Table 1 lists the target compounds and the retention times for each target compound.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria are given in Section 5 of the current revisions of the Savannah Laboratories' quality assurance plans.

2.0 SUMMARY OF METHOD

2.1 A known volume or weight of sample is extracted in accordance with SL SOP EX45. The extracted chlorinated herbicide methyl derivatives are analyzed by gas chromatography (GC) configured with dual capillary columns and dual electron capture (EC) detectors. This configuration allows for simultaneous detection and confirmation of the herbicides. Identification of the target compounds in samples is done by comparing the retention times of the peaks with standards analyzed under the same GC conditions.

GC/MS confirmation may be employed if the target compound concentration is sufficiently high or if the extract is concentrated to an appropriate final volume. The esterified extract must be used for the GC/MS confirmation-do not use the 8270 extract.

2.2 This SOP is based on the guidance in SW-846 Method 8000B and Method 8151A and EPA Method 615.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and eye protection(lab glasses or face shields), must be worn. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

3.4 Diethyl ether is a flammable solvent and it must be used in a well-ventilated hood or extraction area. The solvent vapors will tend to accumulate along the floor. High concentrations of diethyl ether can cause drowsiness, dizziness, and headache.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. The glassware must be scrupulously cleaned (SL SOP AN60: Glassware Cleaning Procedures). All of the materials and reagents must be demonstrated to be free from contaminants by the analysis of reagent blanks (method blanks). Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences. The extraction procedure described in SL SOP EX45 has several steps that are designed to eliminate or minimize interferences due to sample matrix.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Aqueous samples are routinely collected in 1-L amber glass containers equipped with Teflon-lined caps. No preservative is required. The sample is iced at the time of collection and stored at 4C (less than 6C with no frozen samples) in the lab. Sample must be extracted within seven (7) days of collection and the extract must be analyzed within forty (40) days of extraction.

TCLP leachate samples are stored in the same manner as aqueous field samples. The extraction must be performed within seven(7) days of the leaching procedure and the extract analyzed within forty(40) days of extraction

5.2 Soils, sediments, sludges, and wastes are collected in glass containers equipped with Teflon-lined caps. The routine container is 500-mL glass. Larger or smaller containers may be supplied. No preservative is required. The samples are iced at the time of collection and stored at 4C (less than 6C with no frozen samples) in the lab. The hold time for herbicides in solid and non-aqueous matrices is 14 days from the date of collection. The extract must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph equipped with dual electron capture detectors and automatic liquid samplers.

6.2 Recommended Columns:

J&W DB-5 fused silica column, 30M x 0.53mm ID, 1.5um film
J&W DB-608 fused silica column, 30M x 0.53mm ID, 0.83um film

6.3 Data system compatible with the GC and capable of detecting and storing chromatographic data. Nelson 2600 or equivalent

6.4 Autosampler vials, septa, and caps

6.5 Microsyringes- 10-uL, 25-uL, 50-uL, 100-uL

7.0 REAGENTS

Hexane: Pesticide grade or equivalent

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all neat compounds or stock solutions. All preparation steps must be in accordance with SL SOP AN41:*Standard Material Traceability*.

Preparation of the Calibration Standards

If the stock standard is prepared using the free acid form, no correction is required for the concentration. The concentration of the standard is based on the weight of free acids added per unit volume prior to derivitization. A stock standard prepared from neat herbicide acids has to be derivitivized prior to analysis. If a herbicide standard is purchased as a neat methyl ester, the concentration of the standard must be corrected to the free acid concentration. See SL SOP AN43: Standard Preparation for guidance on the preparation of standards.

The calibration standards must be corrected to the weight of the acid. This will eliminate the need to correct the final concentration of the sample. The correction factors are given in Table 2.

The stock standards are prepared in hexane from either the stock standard mixes purchased from vendors or from the individual stock standards prepared in-house. An example of the preparation of the calibration standard is given in Table 3

9.0 SAMPLE PREPARATION

Sample preparation steps are given in SL SOP EX45.

10.0 PROCEDURE

10.1 GC Conditions

The columns and analytical conditions listed in this section are given for guidance. The lab must document the actual columns and conditions used for each analysis in the instrument maintenance, data system, or on the sample log.

10.1.1 Recommended Columns:

J&W DB-5 fused silica column, 30M x 0.53mm ID, 1.5um film
J&W DB-608 fused silica column, 30M x 0.53mm ID, 0.83um film

The columns are connected to a single injection port with either a glass Tee-splitter or a glass y-splitter. This configuration allows for the simultaneous analysis and confirmation of the target compounds.

Carrier gas flow: He at approximately 7mL/min (per column)
Make-up gas flow: N2 at approximately 25mL/min (per detector)

10.1.2 Suggested GC temperature programs

APP 9 temperature program:
Initial temperature: 170 C
Initial hold time: 4 minutes
Program rate: 8 C / minute
Final temperature: 280 C
Final hold: 2 minutes
TOTAL TIME: 20 minutes

8151 Full List temperature program
Initial temperature: 100 C
Initial hold time: 2 minutes
Program rate: 8 C / minute
Final temperature : 280 C
Final hold: 2 minutes
TOTAL TIME: 27 minutes

10.1.3 Heated zone temperatures

Injector: 240-260 C
Detector: 300C

10.1.4 Injection volume: 2-4uL (1-2-uL per column)

10.2 Initial Calibration
The external standard calibration technique is routinely employed for the determination of the concentration of herbicides. The lab also has the option of using internal standard calibration. Pentachloronitrobenzene (PCNB) may be a suitable compound to use as an internal standard

10.2.1 Prepare and inject the calibration standards using the guidelines listed in Section 8 of this SOP. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in Section 10.3.

Note that the following offers two (2) options for calibration and quantitation – average RF or regression curve. Only one need be chosen per analyte.

When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

10.2.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed?

>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?

>Are the peaks symmetrical? Is there tailing or fronting?

>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.2.3 Average RF/CF Option:

Calculate the response factor of each calibration standard, the average response factor and the relative standard deviation using the following equations:

Response Factor-external standard

$$X_{RF} = \frac{mg/mL}{area} = \frac{mg}{mL\ area}$$

Note that the inverse relationship can also be used to evaluate the response of the detector. Dividing the area of the standard by the concentration is referred to as the calibration factor.

Response factor-internal standard

$$RF = \frac{Ac}{Ais} \otimes \frac{Cis}{Cc}$$

where

Ac = area of the target compound
Ais = area of the internal standard
Cc = concentration or mass on-column of the target compound (ug/mL)
Cis = concentration or mass on-column of the internal standard (ug/mL)

Average Response Factor

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 + ..RFn}{n}$$

Relative Standard Deviation

$$\%\,RSD = \frac{standard\ deviation}{\overline{RF}} \otimes 100$$

and

$$Standard\ Deviation = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

Where

RF_i = response factor of the individual calibration level
RF = average response factor

Initial Calibration Criteria:

600-series: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear and the average response factor (or calibration factor) may be used for quantitation.

8000-series:If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear and the average response factor (or calibration factor) may be used for quantitation.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required since the lab has demonstrated that the lowest standard in the calibration curve (the equivalent of the RL) can be detected.

10.2.4 Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPs for other exceptions to using non-linear curve fitting.

10.3 Calibration Verification

Calibration is verified every 24 hours for 600-series methods and every 12 hours or 20 samples, whichever is more frequent, for 8000-series methods by the analysis of continuing calibration check standards. Note that the following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive for the compound(s) failing high are detected, these samples may be reported.

10.3.1 Analyze the mid-level standard(s). Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration:

$$\%Drift = \frac{result - expected}{expected} \times 100$$

Where
result = concentration or nanograms on-column of the calibration check standard quanted against the curve
expected = true concentration or nanograms on-column of the calibration check standard

$$\%Difference = \frac{RF_{init} - RF_{cont}}{RF_{init}} \times 100$$

Where
RF_{init} = average response factor from the initial calibration curve
Rf_{cont} = response factor from the continuing calibration standard

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10.3.2 Continuing Calibration Verification Criteria

If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

600-series: If the percent drift or percent difference is less than or equal to 10%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

8000-series: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor is used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

External Standard CCV: Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

10.4 Sample Analysis Sequence
The analytical sequences for the 600- and 8000-series methods are given in the SOP Summary after Section 15.

10.4.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks (hexane) may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.4.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. For the single point multicomponent products, the extract should be diluted until the area is no more than a factor of two above the area of the single point standard (see section 11.1 for quantitation of multi-peak target compounds).

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used (*F34 or *F42) or qualification in a case narrative provided to the client. For TCLP analyses, every effort should be made to achieve the regulatory level without substantial instrument overload.

For clients who demand we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve (i.e., a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client). Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SL SOP AN66: Determination of Retention Time Windows for Gas Chromatographic Analyses. If internal standard calibration is used, the determination of absolute retention time windows is not required. Relative retention times, as described in Section 11.4, are used to identify the target compounds.

11.0 DATA ANALYSIS/CALCULATIONS

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times), the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration, and the integration of the peaks. Manual integration must be documented in accordance with SL SOP AN65. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data.

The judgement and experience of the analyst and his/her colleagues is an important part of the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks correctly integrated?
- Are there co-eluting peaks or matrix interferences?

11.1 Qualitative analysis

Identification of the target compounds is based on retention time. The analyst should scan the sample chromatogram for the target compounds on the primary analytical column. The analyst should use the retention time (RT) window calculated around the CCV as guidance for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds to help gauge possible shifts in the RT of the target compounds. See SL SOP AN66.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peaks areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed above (10.3.2).

11.1.1 The surrogate should be evaluated first to check for shifts in retention times and to evaluate the surrogate recovery.

The extract contains the surrogate Dichloroacetic acid (DCAA). The recovery criteria are given in Section 5 of the SL QAPs.

Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Generally, it is expedient to calculate the recovery on one chromatogram. If this recovery is acceptable, the other channel is not evaluated for recovery. If interferences are suspected, evaluate recovery on the other channel. Note that given this nature, an extract is considered acceptable if one of the 2 potentially calculated recoveries is acceptable.

NOTE: If the recovery of the surrogate is above the upper control limit and no target compounds are detected in the sample, results may be reported. Refer to section 13 of the current QA Plan regarding this issue.

11.1.2 Label and calculate the concentration of each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences and the overall shapes of the peaks.

If the concentration is below the detection limit, the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.3.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range, the analysis at a dilution is not necessary.

11.1.3 If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds to help gauge possible shifts in the RT of the target compounds.

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

If the relative percent difference is less than 40%, the presence of the target compound is confirmed and the lower concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A large relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. If interference is detected, flag the result to note the disparity between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution.

The following table summarizes the general guidance for the evaluating of chromatographic data. The table assumes that the calibration criteria have been met and that the sample has acceptable associated surrogate and lab spike recoveries.

PEAK INFORMATION	ACTION	REPORT*
No peaks found on primary or secondary column		Report < RL
Peak found within RTW on primary column	Peak is tentatively identified as the target	If concentration < RL, report < RL If concentration > RL, evaluate confirmation column.
Peak found within RTW on confirmation column	Peak is confirmed as the target	If concentration < RL, report <RL If concentration >RL, calculate %RPD -if %RPD <40%, report lower concentration of primary and confirmation analyses. -if %RPD >40%, flag result to note the disparity Case narrative or note to PM may be required for complex matrices.

*RL may be the SL Reporting Limit in Table 5 of the CQAP or may be defined by the client QAP or contract.

The analyst must clearly show how the reported sample results were determined.

11.1.4 Identification "Tools"

Analysis by GC/MS (scan or SIM) may be used to confirm the presence of chlorinated herbicides (see SL SOP SM06: *Guidelines for SIM Analysis by GC/MS.)* The herbicide extract must be used to perform the GC/MS confirmation-unesterified extracts (e.g., the GC/MS extract) are not suitable for herbicide analysis or confirmation.

11.1.4.1 Relative Retention Time

The retention time of a surrogate compound provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions.. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification.

11.1.4.2 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the sinitial identification. The analyst is cautioned to use this approach with discretion and with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience. The extract must be fortified with herbicides as methyl esters-uniesterified herbicides are not amenable to GC analysis.

11.2 Calculations-External Standard

Aqueous/Liquid Samples

If the regression curve option is used, the sample concentration is calculated:

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{V}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

If the response factor option is chosen for quantitation:

$$Concentration(ug/L) = \frac{ug/mL}{response} \otimes response \otimes \frac{F \otimes DF}{V}$$

where
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

Soils/Solids Samples

Regression Curve

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
W = weight of sample extracted (kg)
solids = (percent solids)/100

If the response factor option is chosen for quantitation:

$$Concentration(ug/kg, dw) = \frac{ug/mL}{response} \otimes response \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)
W = weight of sample extracted (kg)
solids = (percent solids)/100

11.3 Internal Standard Calibration

Aqueous Samples

$$Concentration(ug/L) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{V} \otimes DF$$

where

Ac = area of the target compounds
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
V = volume of sample extracted (l)
DF =dilution factor

Soil Samples

$$Concentration(ug/kg, dw) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{W \otimes solids} \otimes DF$$

where

Ac = area of the target compounds
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
W =weight of sample extracted (kg)
Solids = (percent solids)/100
DF =dilution factor

12. 0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch is discussed in SL SOP AN02: *Analytical Batching*, and these criteria are summarized in the SOP Summary included in this SOP. Calculation of QC is also given in AN02.

12.2 The **method detection limit (MDL)** is defined as the concentration of the an analyte that can be measured with a 99% confidence that the result is greater than zero. See SL SOP CA90: *Procedure for Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items are included in this revision. See Section 10 of the current SL QAP.

14.0 TROUBLESHOOTING

No items are included in this revision.

15.0 REFERENCES

1. *Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

2. *Code of Federal Regulations, Title 40, Part 136*; U.S. Government Printing Office: Washington, DC, July 1, 1988.

3. Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*, current revision.

METHOD SUMMARY

HOLD/STORAGE

Parameter	Aqueous	Soils/Solids/Wastes
Routine Container	1-L amber glass fitted with Teflon-lined cap	500-mL or smaller glass fitted with Teflon-lined cap
Preservative	None	None
Hold time	7 days from date of collection; 40 days from date of extraction	14 days from date of collection; 40 days from date of extraction
Storage	4C (less than 6C with no frozen samples) from collection to analysis	4C (less than 6C with no frozen samples) from collection to analysis

EXTRACTION

Aquoues-500mL of sample; separatory funnel extraction at pH >12 with diethyl ether followed extraction at pH<2 with diethyl ether; esterify and dilute to 10mL final volume with hexane

Soils- acidify 30g of sample mixed with acidified sodium sulfate; sonication or Soxhlet extraction with 1:1methylene chloride/acetone; concentrate and hydrolyze with KOH, treat same as aqueous sample from this point forward.

SEQUENCE

615

Initial Calibration Standards
Client samples analyzed until 24 hour clock expires
Calibration Verification standard – mid-level concentration
Client samples analyzed until 24 hour clock expires

8151

Initial Calibration Standards
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration

The sequence continues until all samples are analyzed or until the CCV standard fails to meet the acceptance criteria.

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration- 5 point minimum with lowest point at RL	Prior to sample analysis or when CCV fails	1) RSD of each target <= 20%(8151) or 10% RSD (615); OR 2) plot regression curve (CC>=0.99) (see Section 10.2 for 8000-series "grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification(CCV)	After every twenty sample analyses (or 12 hours) and at the end of the sequence	8151: Percent difference or drift <= 15% 615: Percent difference or drift <= 10% (see Section 10.3 for 8000-series "grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Method blank	Per batch	All targets reported less than RL in Table 5 of the SL CQAP	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Lab control sample (LCS)- Subset of targets in SL CQAP	Per batch	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix spike(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogate	All samples, method blanks, and QC	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Reporting limit(RL) standard-lowest level calibration standard	Daily-required for Fla DEP	Detected with reasonable sensitivity	-Reanalyze RL standard -Remake and reanalyze RL standard -Perform instrument or column maintenance, recalibrate, and reanalyze associated samples
Initial Demonstration of Capability (IDOC)	Initially and when new analysts trained	Evaluate in accordance with method criteria	Repeat test for analytes that fail criteria
Method Detection Limit (MDL)	See SL SOP CA90	Evaluate in accordance with SL SOP CA90	Evaluate in accordance with SL SOP CA90
Retention time window determination	See SOP AN66	See SOP AN66	See SOP AN66

TABLE 1-"full list"		
Compound	RT COL 1	RT COL 2
Dalapon	2.90	3.35
DCAA (surrogate)	13.18	13.41
MCPP	13.56	14.08
Dicamba	13.66	13.63
MCPA	14.30	14.44
Dichlorprop	14.94	15.36
2,4-D	16.10	15.89
2,4,5-TP (Silvex)	18.27	19.22
2,4,5-T	20.49	20.33
Dinoseb	21.00	23.06
2,4-DB	22.81	23.36

COL1 = DB608 COL2 = DB-5

TABLE 1A-"short list"		
Compound	RT COL 1	RT COL 2
DCAA (surrogate)	5.38	5.75
2,4-D	7.65	7.61
2,4,5-TP (Silvex)	8.80	9.31
2,4,5-T	9.68	9.74

COL1 = DB608 COL2 = DB-5

TABLE 2

HERBICIDE MOLECULAR WEIGHTS AND CORRECTION FACTORS

Herbicide acid	MWacid	MWester/ether	Correction factor
2,4-D	221.04	235.07	0.940
Dalapon	142.97	157.00	0.911
2,4-DB	249.09	263.12	0.947
Dicamba	221.04	235.07	0.940
Dichloroprop	235.07	249.09	0.944
Dinsoeb	240.22	254.24	0.945
MCPA	200.62	214.65	0.935
MCPP	214.65	228.67	0.939
2,4,5-TP(Silvex)	269.51	283.54	0.951
2,4,5-T	255.48	269.51	0.948
DCAA	205.04	219.07	0.936
Picloram	241.48	255.51	0.945
Pentachlorophenol	266.35	280.37	0.950

Example Calculation

$$CF(2,4-D) = \frac{Wacid}{Wester} = \frac{221.04}{235.07} = 0.94$$

If the standard is expressed as mass of ester per volume, convert the concentration to he acid form by multiplying by the correction factor (CF).

Table 3-Example Standard Preparation "Recipes"

"Short List" Herbicide Intermediate Standard

STOCK STANDARD	VENDOR/ PART NO.	CONC. (ug/mL)	mL of stock	fvol (mL)	conc (ug/mL)
Herbicides	Ultra HBM-815M	100	1.0	10	10
DCAA	Ultra PPS-161	100	1.0		10

Solvent is hexane

"Short" list Calibration Standards

STOCK STANDARD	CONC. (ug/mL)	1*	2*	3*	4*	5*	6*	7*
"short" list herbicide intermediate std	10	50	100	200	250	500	750	1000

* microliters of intermediate standard to 10mL of hexane. Seven standards are prepared and analyzed but only 5 are used. MCPP and MCPA are difficult to calibrate at low concentrations.

"Short" list Calibration Standards

Target Compounds	1*	2*	3*	4*	5*	6*	7*
DCAA, 2,4,-D, 2,4,5-TP (Silvex), 2,4,5-T	0.05	0.10	0.20	0.25	0.50	0.75	1.0

* ug/mL

"Full" list Calibration Standards

STOCK STANDARD	CONC. (ug/mL)	1*	2*	3*	4*	5*	6*	7*
Ultra HBM-8150M		5	10	12.5	20	50	67	100
Dicamba, 2,4,5-TP (Silvex), 2,4,5-T	10							
2,4-D, 2,4-DB, Dichlorprop	100							
Dinoseb	50							
MCPA, MCPP	10000							

* microliters to 10mL hexane

Table 3-Example Standard Preparation "Recipes"

"Full" list Calibration Standards

STOCK STANDARD	CONC. (ug/mL)	1*	2*	3*	4*	5*	6*	7*
Ultra HBM-8150M								
Dicamba, 2,4,5-TP (Silvex), 2,4,5-T	10	0.005	0.010	0.0125	0.020	0.050	0.067	0.10
2,4-D, 2,4-DB, Dichlorprop	100	0.050	0.10	0.125	0.20	0.50	0.67	1.0
Dinoseb	50	0.025	0.050	0.0625	0.10	0.25	0.33	0.50
MCPA, MCPP	10000	5.0	10	12.5	20	50	67	100

*ug/mL

B-7 Continuous Liquid-Liquid Extraction

Approval Signature: _____
R. Wayne Robbins
Title: Corporate QA Manager Date: 7/13/99

CONTINUOUS LIQUID-LIQUID EXTRACTION

1.0 SCOPE AND APPLICATION

This SOP describes the general procedures for the extraction of semivolatile organic compounds (SVOC) from aqueous samples using continuous liquid-liquid extraction. This procedure is applicable to a wide range of aqueous matrices including monitoring wells, industrial wastewater, saline waters, and TCLP or EP leachates.

2.0 SUMMARY OF METHOD

2.1 A known volume of sample is adjusted to a specific pH if required, transferred to a continuous liquid-liquid extractor, and extracted using the solvent and conditions specified in Table 2. The extract is concentrated to an appropriate final volume using either the Zymark nitrogen blow-down concentrator (SL-SOP EX50) or the Kuderna Danish concentration procedure (SL-SOP EX51).

2.2 This SOP is based on the guidance in SW-846 Method 3520C and the extraction sections of the EPA 600-series methods.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedure that you do not understand or that will put you or others in potentially hazardous situations.

3.2 This SOP contains procedures that are designed to reduce the exposure of lab personnel to solvent vapors and to minimize the amount of solvent introduced into the lab air. All solvent transfer steps should be performed quickly and under a hood, if possible. Use the minimum amount of solvent to get the job done. Do not allow open containers of solvent or extracts to evaporate into the lab.

3.3 A lab coat or apron should be worn to protect clothing and skin from acids, bases, and solvents. Gloves must be worn to protect hands and eye protection (goggles or safety glasses) must be worn at all times in the lab. An analyst must not work alone in an isolated area.

3.4 A solvent such as methylene chloride can cause a burning sensation if it contacts the skin. The rapid evaporation of the solvent causes a rapid heat loss in the skin similar to frost bite. If solvent contracts the skin, wash the exposed skin in cold water to reduce the solvent evaporation.

3.5 The analyst should become familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in the extraction lab.

4.0 INTERFERENCES

4.1 Improperly cleaned glassware can contribute interferences to the sample extract. Material that is left inside the extractor after cleaning may show up in the next extraction. The glassware must be properly cleaned according to SL SOP AN60. After cleaning, inspect the glassware for the presence of water, especially in the small tubing of the extractor. This water can block the solvent return tube and prevent efficient extraction of the sample. A thorough acetone rinsing will help to eliminate or minimize this problem.

4.2 Sample with large amounts of sediments or particulate may clog the solvent return line. A layer of glass wool, placed in the bottom of the extractor, may be helpful when sediments or particulate are present.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Aqueous samples are routinely collected in 1-L amber glass containers equipped with Teflon-lined caps. No preservative is required and an orange dot is affixed to the container to indicate this. The sample is stored at 4°C± 2°C in the lab. The sample must be extracted within seven (7) days of collection and the extract must be analyzed within forty (40) days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Continuous liquid-liquid extractor with compatible condenser, extractor body, and receiving flask.

6.2 Heating mantle and adjustable support. The heating mantle must be connected to a rheostat to control the temperature.

6.3 Rheostat or variable transformer

6.4 Teflon stoppers to fit the receiving flask

6.5 Filter paper-grade 414, 18.5cm diameter (VWR 28320-143)

6.6 Glass funnels

6.7 Pyrex glass wool - purified by Soxhlet extraction with methylene chloride.

6.8 Disposable volumetric pipettes - 1.0-mL and 10-mL

6.9 Boiling stones-Soxhlet extracted with methylene chloride

6.10 Wide range pH paper

7.0 REAGENTS

7.1 Reagent water - lab generated deionized water

7.2 Methylene chloride - residue grade or better

7.3 Acetone - residue grade or better

7.4 Sodium hydroxide (NaOH) - reagent grade

7.5 Sodium hydroxide solution (10N): Dissolve 400g of NaOH pellets into about 500mL of reagent water contained in a 2-L beaker on a magnetic stirrer. Add the NaOH in small portions, with constant stirring, to minimize the time it takes to dissolve the pellets. A good deal of heat will be generated as the NaOH dissolves. After all 400g has been added, carefully dilute to 1000mL with reagent water. Mix the solution thoroughly and transfer to a storage container. Do not store sodium hydroxide solution in volumetric glassware or in containers with ground glass joints.

7.6 Sulfuric acid (H_2SO_4) - concentrated reagent grade

7.7 Sulfuric acid solution (1:1 v/v): Slowly and carefully add 550mL of concentrated H_2SO_4 to 500mL of reagent water contained in a 2-L beaker on a magnetic stirrer. Add the acid in small portions with constant stirring to reduce the heat evolved when the acid and water are combined. Cool and transfer the solution to a labeled storage container.

7.9 Sodium sulfate-anhydrous, granular, purified by heating for 4 hours at 400C in a shallow tray. Store in glass containers

8.0 STANDARDS

The preparation of the surrogate and matrix spiking solutions are described in SL SOP EX70: *Preparation of Semivolatile Surrogates and Matrix Spiking Solutions.* Table 1 is a summary of the spiking solutions used for the various analytical extraction methods. All preparation steps must be tracked in accordance with SL SOP AN41:*Standard Material Traceability.*

The tables included in SOP EX70 describe "recipes" that can be used to prepare the spiking solutions. The lab can prepare larger or smaller volumes if desired. The recipe used by the lab may be prepared as a "controlled posting" to prompt the analyst in the preparation of the spiking solutions. The controlled posting can be posted in the lab or placed in the extraction lab tracking log.

9.0 SAMPLE PREPARATION

The samples and QC items must be clearly and unambiguously labeled during each step of the extraction and analysis process. Labeling tape should be used to identify the glassware associated with each sample. Solvents will erase grease pens and "Sharpies". Use of different colored labeling tape will aid the analyst in keeping up with the extracts during each step of the extraction and concentration.

9.1 Sample Evaluation

9.1.1 Remove the samples to be extracted from the storage refrigerator. Allow the samples to equilibrate to room temperature while the appropriate glassware is gathered and prepared for the extraction. Fill out as much of the extraction log as possible prior to initiating the extraction.

9.1.2 Inspect the samples. Determine if the samples have multiple layers such as sediment or an oil layer. Consult with the supervisor or technical manager if the sample matrix is unusual or is difficult to categorize or handle.

9.1.3 Mark the level of the sample on the outside of the container. After the sample has been added to the extractor body, fill the empty container with water to the mark. Pour the water into a graduated cylinder and determine the volume. Record the volume in the extraction log to the nearest 5mL..

NOTE: If a reduced volume of sample must be extracted in order to provide sufficient sample volume to meet state or other program QC requirements, the sample is thoroughly mixed and an aliquot is measured with a graduated cylinder and poured into the extractor body. The following volumes are utilized for TCLP samples for BNA and pesticides:

TCLP PARAMETER	Volume of Sample Extracted(mL)	Final Volume (mL)
BNA by GC/MS (8270)	200	1.0
Pesticides by GC (8081)	20	10

9.2 Glassware Preparation

Process glassware and samples (Sections 9.2 through 9.4) in batches not greater than 20 field samples and QC in order to minimize the evaporation of methylene chloride into the lab.

9.2.1 Prepare the continuous liquid-liquid extraction apparatus by rinsing the extractor body, receiving flask, and condenser with acetone. Pay particular attention to the solvent return line for the presence of water drops or solids.

9.2.2 Set the extractor body on the holder. Add methylene chloride (50-100ml) to the extractor body in order to fill the drain tube and ensure that a continuous flow of solvent from the extractor body to the receiving flask is maintained.

NOTE: Inspect the solvent layer when the sample is added. Large quantities of sediment may plug the tube and extraction will not occur. The newest extractor bodies have been re-designed to reduce the amount of solvent necessary to ensure a continuous flow of solvent. Use the amount of solvent-50mL or 100mL- necessary to allow the extractor to function properly.

9.2.3 In a fume hood, add 100mL to 175mL of methylene chloride to the receiving flask and add a few boiling stones to the flask. Add the same volume to each flask in the batch. Leave the receiving flask with the methylene chloride under the hood until ready to attach it to the extractor or cover tightly with aluminum foil.

9.3 Attach the receiving flask to the extractor body and secure the heating mantle around the flask.

9.4 As soon as possible after the methylene chloride is added to the extractor, gently pour the entire sample into the extractor body, trying not to let the sample leak into the sidearm. The extractor body will be approximately ½ to 2/3 full and the methylene chloride in the solvent return should be about 3/4 of the way to the receiving flask.

NOTE: The lab has the option of using an aliquot of sample if the final volume of the extract is adjusted to compensate for the reduced volume of sample. In some cases, the volume of sample must be reduced in order to have sufficient sample to extract matrix spikes as required by a state or other program quality assurance plan or regulation. In this case, the volume of surrogate and matrix spiking solution added to the sample should also be reduced proportionately .

9.5 Add 1.0L aliquots of reagent water to each of two separate extractors to serve as the method blank and lab control sample (LCS).

EXTRACTION BATCH: An extraction bath consists of up to twenty (20) field samples and the following QC items: method blank, LCS, MS, and MSD. If insufficient sample is available to perform the MS and MSD, the LCS is extracted in duplicate(LCSD) and the extraction log is stamped: INSUFFICIENT SAMPLE AVAILABLE TO PERFORM MS/MSD.

9.6 Addition of Surrogates and Matrix Spikes
The surrogate and matrix spiking solutions are set up so that 1.0ml of the solution will be added . Be sure to use the correct solutions for the extraction of concern. The addition of the surrogates and matrix spikes should be witnessed by another analyst to ensure that the proper volume of the spiking solution is added to each sample.

9.6.1 Add 1.0ml of the surrogate spiking solution to each sample and QC item.

NOTE: If the volume of sample extracted must be reduced to provide sufficient sample to meet state or other program requirements, the volume of surrogate spiking solution must be reduced proportionately.

9.6.2 Add 1.0ml of the matrix spiking solution to each LCS, MS, and MSD.

NOTE: If the volume of sample extracted must be reduced to provide sufficient sample to meet state or other program requirements, the volume of matrix spiking solution must be reduced proportionately.

9.6.3 Adjust the pH of the samples to the range specified in Table 2. The pH should be adjusted by adding small aliquots of 10N sulfuric acid or 10N sodium hydroxide directly to the sample extraction body. Stir the sample with a glass rod or Teflon rod. Check the pH after each addition with wide range paper. Do not dip the paper into the sample. Use a glass rod or pipette to remove a small aliquot of sample and touch the liquid to the pH paper.

NOTE: Sulfuric acid will cause the pH of the sample to decrease; sodium hydroxide will cause the pH of the sample to increase.

9.7 Slowly add reagent water to the extractor body until the level of the liquid causes the methylene chloride to just flow over to the receiving flask.

9.8 Place the condenser securely on the extractor body. Make sure that water is flowing through the condenser and turn the heating mantle on. Observe the extraction for the first hour or so to ensure that the solvent is being boiled, condensed, and returned to the receiving flask. Extract the samples for the time listed in Table 2. The batch is labeled with the start time and the scheduled stop time. I f a single pH extraction is performed, go to 9.11; if a two-pH extraction must be performed, continue at 9.9.

9.9 If a dual pH extraction is required, turn the heating mantle "off" to stop the extraction at the appropriate time and allow the extraction vessel to cool. Adjust the pH of the sample by adding small aliquots of 10N sulfuric acid or 10N sodium hydroxide. Stir the sample with a glass rod or pipette and check the pH after each addition . Do not dip the pH paper into the sample but use a glass rod or pipette to remove a small aliquot of the sample and touch the liquid to the pH paper.

NOTE: Sulfuric acid will cause the pH of the sample to decrease; sodium hydroxide will cause the pH of the sample to increase.

9.10 Turn the heating mantle "on" and extract the sample at the second pH for the time listed in Table 2. The batch is labeled with the start time and the scheduled stop time

9.11 After the samples have extracted for the required time, turn the heating mantle "off" and allow the extraction vessel to cool to room temperature. It is important to allow the receiving flask to cool before it is removed from the extractor.

9.12 Remove the receiving flask from the extractor body and cover it with a Teflon stopper. The extract is stored under near a hood until the filtration and concentration step is performed.

9.13 Working under a hood, place a piece of filter paper into a glass funnel and add a small amount of purified sodium sulfate. Filter the extract through the glass funnel and collect the extract directly into a labeled Zymark tube. Discard the filter paper and sodium sulfate into a waste container which is kept under the hood until removed from the laboratory. The extract is now ready for concentration using the Zymark instrument(SL SOP EX50) and should be kept under a hood tightly covered with aluminum foil until the concentration step. The extract may also be concentrated using the Kuderna-Danish extract evaporation(SL SOP EX51).

NOTE: Perform the extract filtration and drying steps in batches to minimize the loss of solvent into the lab. Cover the remaining containers tightly with aluminum foil and leave under or near a hood.

After the extracts have been dried and filtered, dispose of the filter paper and sodium sulfate by putting them in a chlorinated solvent waste container that is kept under the hood.

NOTE: If the volume of sample extracted has been reduced to provide sufficient sample to meet state or other program QC requirements, the final volume of the extract must be reduced proportionately.

9.14 Rinse the ground-glass joint of the condenser with acetone and wrap in aluminum foil before placing the condenser in the rack. Collect the acetone rinsate in a separate container for disposal.

9.15 After the receiving flask has been removed from the extractor body, the methylene chloride remaining in the extractor body must be properly disposed. The extractor should be handled with minimum agitation to minimize the amount of solvent allowed to evaporate into the lab and stored in the chlorinated solvent waste container. Place the receiving flask side-arm over the chlorinated waste container. Add tap water to the extractor body to displace the methylene chloride into the waste container

The liquid remaining in the extractor body should be poured carefully down the sink and flushed continuously with water.

10.0 PROCEDURES

No items in this revision.

11.0 DATA ANALYSIS/CALCULATIONS

No items in this revision.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Table 1 -Surrogate and Matrix Spiking Solutions

METHODS	TYPES OF COMPOUNDS	SURROGATE SPIKING SOLUTION	MATRIX SPIKING SOLUTION
604	Phenols by GC-FID	SG ACID	604
8041	Phenols by GC-FID	SG ACID	8040
606,8061	Phthalate esters by GC-FID	SG BASE	606/8060
607,8070	Nitroaromatics by GC-FID	8141	target compounds
608	Chlorinated pesticides and PCBs	PEST	608
8081,8082	Chlorinated pesticides and PCBs	PEST	8080
8081,8082	PCBs	PEST	PCB
8081, 8082	Chlorinated pesticides-TCLP	PEST	TCLP-PEST
609,8090	Nitrosamines by GC-FID	SG BASE	target compounds
610	PAH by GC-FID	SG BASE	610
8100	PAH by GC-FID	SG BASE	8100
611,8110	Halogenated ethers by GC/EC	PEST	target compunds
612,8120	Chlorinated hydrocarbons	PEST	target compunds
614,614.1,622, 622.1	Phosphorous and nitrogen containing pesticides	8141	target compunds
8141	Phosphorous and nitrogen containing pesticides	8141	8141
617	Chlorinated pesticides	PEST	target compunds
619/620	Phosphorous and nitrogen containing pesticides	8141	target compunds
627	Chlorinated pesticides	PEST	target compunds
625	BNA	625/8270	625
8270-TCL	BNA	625/8270	8270
8270-K001	BNA	625/8270	K001(8270)
8270-TCLP, AP9	BNA	625/8270	TCLP-8270
8015-EXT	Petroleum products	SG BASE	Diesel-8015-mod Mineral spirits-8015-mod
613/8280	Chlorinated Dioxins & Furans	INT A and INT B	Octa and TH
625-OCPSF	BNA	62518270	625
FL-PRO	Petroleum products	FL-PRO	FL-PRO
8310	PAHs by LC	8310	8310

Table 2 -Extraction Conditions

METHODS	EXTRACTION CONDITIONS	EXTRACTION TIME(hr.)	SOLVENT	FINAL SOLVENT	FINAL VOLUME(mL)
604,8041	Single pH < 2	>12	MeCl2	MeCl2	1.0
606,8061	Single pH as received or at pH<2	>12	Mecl2	MeCl2	1.0
607,8070	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
608,8080,8081, 8082	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	10
609,8090	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
610,8100	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
611,8110	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
612,8120	Single pH as received or at pH<2	>12	MeCl2	Hexane	10
614,614.1,622, 622.1,8141	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	10
617	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	10
619	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	5.0 or 10
627	Single pH at neutral pH (5-9). pH range is critical for good recovery of some target compounds	>12	MeCl2	Hexane	10
8270-TCL,K001	Single pH <2	>12	MeCl2	MeCl2	1.0
625, 8270-TCLP	Dual pH at pH<2 followed by pH>12	18/18	MeCl2	MeCl2	1.0
8270-AP9	Dual pH at pH<2 followed by pH>12	>12/>12	MeCl2	MeCl2	1.0
8015-EXT	Single pH as received or at pH<2	>12	MeCl2	MeCl2	1.0
6131/8280	Single pH at neutral pH (5-9)	>12	MeCl2	Dodecane	0.2
625-OCPSF	Dual pH at pH>12 followed by pH<2. Extracts are kept separate.	18/18	MeCl2	MeCl2	1.0 Acid 1.0 Base
FL-PRO	Received at pH<2. Adjust to pH<2 if not received at ph<2.	>12	MeCl2	MeCl2/CS2	2.0
8310	Single pH at neutral pH (5-9)	>12	MeCl2	Acetonitrile	1.0

STL Savannah

CHANGE -IN-PROGRESS ATTACHMENT

SOP Document No: EX40:02.19.98:1
SOP Description: Ultrasonic Extraction

Approval Signature: R. Wayne Robbins
R. Wayne Robbins
Title: STL-SL QA Manager Date: Nov 4, 1999

The following revisions or additions have been made to the referenced SOP.

The following changes are made to clarify the procedures for high level samples. The default procedure will be to use the same extraction procedure described in this SOP for soils/solids that require a smaller (greater than or equal to 1g) aliquot because of high levels of target or non-target contaminants.

2.0 SUMMARY OF METHOD

2.1 A known weight of a sample (approximately 30g wet weight) is combined with anhydrous, purified sodium sulfate to form a free flowing, sandy mixture. A solvent, usually 1:1 acetone/methylene chloride, is added to the dried sample and the sample is extracted using an ultrasonic disrupter for three (3) minutes. The solvent is decanted and the extraction is repeated two more times. The extract is filtered and concentrated to an appropriate final volume. Table 1 defines the extraction conditions.

NOTE: The lab has the option of extracting a sample aliquot equal to 30g on a dry weight basis.

NOTE: If the sample has high levels of contaminants (target or non-target), a smaller aliquot of the sample may be weighed and extracted according to this procedure. The minimum weight of sample is 1g. Wastes and pure products may be prepared using the procedures in STL SL SOP EX42: *Waste Dilution Extraction*.

9.2.2 Weigh 30.0 - 30.5g of the homogenized sample into a pre-cleaned, labeled 500-mL extraction bottle. Record the weight for all samples in this batch.

NOTE: The lab has the option of weighing an aliquot of sample equal to 30g on a dry weight basis.

NOTE: If the sample has high levels of contaminants (target or non-target), a smaller aliquot of the sample may be weighed and extracted according to this procedure. The minimum weight of sample is 1g. Wastes and pure products may be prepared using the procedures in STL SL SOP EX42: *Waste Dilution Extraction*.

EX40:02.19.98:1

Approval Signature:	R. Wayne Robbins
R. Wayne Robbins	
Title: Corporate QA Manager	Date: 02/20/98

ULTRASONIC EXTRACTION

1.0 SCOPE AND APPLICATION

This SOP describes the process for extracting semivolatile organic compounds (SVOC) from solid matrices by ultrasonic (sonication) extraction. This procedure is applicable to soils, sediments, sludges, and wastes.

2.0 SUMMARY OF METHOD

2.1 A known weight of a sample (approximately 30g wet weight) is combined with anhydrous, purified sodium sulfate to form a free flowing, sandy mixture. A solvent, usually 1:1 acetone/methylene chloride, is added to the dried sample and the sample is extracted using an ultrasonic disrupter for three (3) minutes. The solvent is decanted and the extraction is repeated two more times. The extract is filtered and concentrated to an appropriate final volume.
Table 1 defines the extraction conditions.

NOTE: The lab has the option of extracting a sample aliquot equal to 30g on a dry weight basis.

2.2 This procedure is based on SW-846 Method 3550B.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 This SOP contains procedures that are designed to reduce the exposure of lab personnel to solvent vapors and to minimize the amount of solvent introduced into the lab air. All solvent transfer steps should be performed quickly and under a hood, if possible. Use the minimum amount of solvent to get the job done. Do not allow open containers of solvent or extracts to evaporate into the lab.

3.3 A lab coat or apron should be worn to protect clothing and skin from acids, bases, and solvents. Gloves must be worn to protect hands and eye protection (safety glasses, goggles) must be worn at all times in the lab. An analyst should not work alone in an isolated area.

3.4 A solvent such as methylene chloride can cause a burning sensation when it contacts the skin. The rapid evaporation of the solvent causes a rapid heat loss in the skin which is similar to frost bite. If this occurs, was the exposed skin in cold water to reduce the solvent evaporation.

3.5 The analyst should become familiar with the Material Safety Data Sheets (MSDS) for each reagent used in the extraction lab.

4.0 INTERFERENCES

Improperly cleaned glassware or equipment can contribute interferences to the sample extract. A thorough acetone rinsing will help to eliminate or minimize this problem.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Soils, sediments, sludges, and wastes are collected in glass containers equipped with Teflon-lined caps. The normal container is 500-mL glass. Larger or smaller containers may be supplied. The sample is not preserved and should have an orange dot affixed to indicate this. The samples are stored at 4C ± 2C in the lab. The hold time for SVOC in solid matrices is 14 days from the date of collection. The extract must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Ultrasonic disrupter: Tekmar Model or equivalent with ¾ inch horn-type titanium-tipped sonication probe. The sonicator should be capable of operating in the pulse mode at full power.

6.2 Sonabox - the sonicator should be placed in the sonabox to reduce noise. The sonabox must be placed under a fume hood.

6.3 Pre-cleaned 500ml extraction bottles (P/N SAV-145). Discard the bottles after use.

6.4 Large glass funnels

6.5 Filter paper-18.5cm

6.6 Pyrex glass wool - purchased purified or extracted with methylene chloride for a minimum of four (4) hours.

6.7 Stainless steel spatulas

6.8 Small cups or beakers for optional pH measurement.

6.9 wide range pH paper

6.10 Disposable volumetric pipets

6.11 Top-loading balance - capable of weighing to 0.1g. The balance calibration must be verified according to SL SOP AN10 each time the balance is used.

7.0 REAGENTS

7.1 Sodium sulfate, powdered and granular, anhydrous - purified by heating at 400C for four (hours) in a shallow tray.

7.2 Methylene chloride - residue grade or better

7.3 Acetone - residue grade or better

7.4 1:1 acetone/methylene chloride: Mix equal volumes of acetone and methylene chloride in an appropriate glass container.

8.0 STANDARDS

The preparation of the surrogate and matrix spiking solutions are described in SL SOP EX70: *Preparation of Semivolatile Surrogates and Matrix Spiking Solutions*. Table 2 is a summary of the spiking solutions used for the various analytical extraction methods. All preparation steps must be tracked in accordance with SL SOP AN41:*Standard Material Traceability*.

The tables included in SOP EX70 describe "recipes" that can be used to prepare the spiking solutions. The lab can prepare larger or smaller volumes if desired. The recipe used by the lab may be prepared as a "controlled posting" to prompt the analyst in the preparation of the spiking solutions. The controlled posting can be posted in the lab or placed in the extraction lab tracking log.

9.0 SAMPLE PREPARATION

9.1 Extraction Glassware Set-up

9.1.1 Remove samples to be extracted from the storage refrigerator and allow the samples to come to room temperature while the extraction glassware is being prepared.

9.1.2 Collect the appropriate glassware and rinse with acetone prior to use. Use the minimum amount of solvent to rinse the glassware.

9.1.3 Fit a filter pad into a large funnel and rinse the filter paper/funnel with acetone.

9.2 Sample Extraction

Record as much information on the extraction log as possible before beginning the extraction to minimize the number of entries required during the extraction. The extraction steps from weighing to the sonication should be carried out quickly to minimize the loss of SVOC through evaporation.

9.2.1 Open the sample container and inspect the sample. Remove any materials such as rocks, sticks, leaves, etc. Using a stainless steel spatula , thoroughly homogenize the sample, mixing in any water that may be present on top of the sediment or soil. Use a separate spatula for each sample to prevent cross-contamination of the samples.

NOTE: If it is difficult to homogenize the sample or if the sample matrix is difficult to characterize, contact the supervisor before preceding with the extraction. A careful inspection of the sample at this point can save time and effort later on in the analysis.

9.2.2 Weigh 30.0 - 30.5g of the homogenized sample into a pre-cleaned, labeled 500-mL extraction bottle. Record the weight for all samples in this batch.

NOTE: The lab has the option of weighing an aliquot of sample equal to 30g on a dry weight basis.

Weight 30g of powdered, purified sodium sulfate into each of two separate extraction vessels to serve as the method blank and lab control spike (LCS).

Weigh two additional 30g portions of a sample in the batch selected as the matrix spike (MS) and matrix spike duplicate(MSD). The MS/MSD may be specified by the client or may be chosen from the samples in the batch.

NOTE: Each extraction batch of twenty or fewer samples will have the following QC items: method blank,

LCS, LCSD, MS, and MSD. If there is not enough sample to perform the MS/MSD, the extraction log must be stamped "INSUFFIECIENT SAMPLE WAS AVAILABLE TO PERFROM THE MS/MSD" and the LCS extracted in duplicate(LCSD).

9.2.3 Add approximately 60g of powdered, purified sodium sulfate to each sample and MS/MSD and stir with a glass rod or stainless steel spatula to form a sandy, free-flowing mixture. The sodium sulfate combines with the water in the sample to "dry" the sample (remove the water). Additional sodium sulfate may be required if the sample is very wet.

NOTE: The next steps should be performed immediately after the sodium sulfate is added to minimize the loss of SVOC due to evaporation

9.2.4 Add 1.0mL of the appropriate surrogate spiking solution (Table 1) to the method blank, LCS, MS, MSD and each sample in the batch.

9.2.5 Add 1.0ml of the appropriate matrix spiking solution (Table 1) to the LCS, MS, and MSD. Place the extraction bottle under the hood near the sonicator

9.2.6 Working under a hood, immediately add 1:1 acetone/methylene chloride to the cover the sample approximately one inch above the solids. Stir the sample to break up any lumps that may have formed.

9.2.7 Check the "tune" of the sonicator using the procedure tested in SL SOP AN15: Tekmar Sonic Dismembrator Tuning Procedure.

9.2.8 Place the tip of the sonicator horn in the center of the beaker about ½ inch below the surface of the solvent but above the solid portion. More solvent may be added to bring the level to approximately one inch above the solid layer.

9.2.9 Sonicate for three (3) minutes with output control knob set at 10, mode switch to pulse, and percent duty cycle set at 50% If the sonication is properly performed, the solids and solvent will vigorously mix each time the sonicator pulses.

9.2.10 Decant the extract through the filter funnel and collect the extract in a beaker or directly in a labeled, "elongated" Zymark tube. The elongated tubes hold approximately 225mL of solvent; therefore, it is desireable to limit the volume of the three extracts to 225mL. If the sample is very wet and requires a large amount of sodium sulfate and a larger volume of solvent, the extracts are collected in separate Zyamrk tubes and combined prior to analysis to analysis.

9.2.11 Repeat steps 9.2.6 through 9.2.10 two more times with fresh portions of methylene chloride and collect the extract in the Zymark tube. Cover the Zymark tube with aluminum foil and leave it under the hood until the concentration step.

NOTE: Clean the sonicator horn between samples by rinsing with acetone. "Sonicate" an extraction bottle of acetone for 3 minutes with output control at 5 to further clean the horn (sodium sulfate may be added to the beaker to prevent cracking).

9.2.13 Dispose of extraction bottle and sample residue by placing in a chlorinated solvent waste container which is kept under a hood until removed from the laboratory.

10.0 ANALYTICAL PROCEDURE

No items in this revision.

11.0 CALCULATIONS

No items in this revision.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Table 1 -Surrogate and Matrix Spiking Solutions

METHODS	TYPES OF COMPOUNDS	SURROGATE SPIKING SOLUTION	MATRIX SPIKING SOLUTION
8040/8041	Phenols by GC-FID	SG ACID	8040
8060,8061	Phthalate esters by GC-FID	SG BASE	606/8060
8081, 8082	Chlorinated pesticides and PCBs	PEST	8080
8082	PCBs	PEST	PCB
8081	Chlorinated pesticides-TCLP	PEST	TCLP-PEST
8100	PAH by GC-FID	SG BASE	8100
8140,8141	Phosphorous and nitrogen containing pesticides	8141	8141
8270-TCL	BNA	625/8270	8270
8270-K001	BNA	625/8270	K001(8270)
8015-EXT	Petroleum products	SG BASE	Diesel-8015-mod Mineral spirits-8015-mod

Table 2 -Extraction Conditions

METHODS	TYPES OF COMPOUNDS	EXTRACTION CONDITIONS	SOLVENT	FINAL SOLVENT	FINAL VOLUME(mL)
8040,8041	Phenols by GC-FID	As received	1:1 Ace/MeCl2	MeCl2	1.0
8060,8061	Phthalate esters by GC-FID	As received	1:1 Ace/MeCl2	MeCl2	1.0
8081, 8082	Chlorinated pesticides and PCBs	As received	1:1 Ace/Hexane	Hexane	10mL
8100	PAH by GC-FID	As received	1:1 Ace/MeCl2	MeCl2	1.0mL
8140,8141	Phosphorous and nitrogen containing pesticides	As received	1:1 Ace/Hexane	Hexane	10mL
8270-TCL,K001	BNA	As received	1:1 Ace/MeCl2	MeCl2	1.0mL
8015-EXT	Petroleum products	As received	1:1 Ace/MeCl2	MeCl2	1.0mL

B-8 GEL Permeation Chromotography

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GEL PERMEATION CHROMATOGRAPHY

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Approved by:

R. Wayne Robbins — 25 July 2000 (Date)

Title: Technical Manager, QA

1.0 SCOPE AND APPLICATION

This SOP provides guidance for the use of gel permeation chromatography for semivolatile organic compound (SVOC-base/neutral, acids, pesticides, and PCBs). GPC is a size exclusion cleanup procedure using organic solvent and hydrophobic gels to separate large molecules such as fats and oils from the sample extract. GPC does not remove non-targets that have the same molecular size as the target compounds (e.g., petroleum-based products).

2.0 SUMMARY

The column is packed with the required amount of pre-swelled absorbent (Biobeads) and is flushed with solvent for an extended period according to the GPC manufacturer's instructions. The column is calibrated, loaded with the filtered sample extract, and eluted with solvent to separate the target compounds from high molecular weight material. The GPC-cleaned sample extract is exchanged to a suitable solvent if required and concentrated to an appropriate final volume in preparation for chromatographic analysis.

NOTE: One half of the sample extract is lost to waste when the sample is loaded on to the GPC. This loss of extract is accounted for by reducing the final volume of the extract to one half the normal final volume or by including a dilution factor of 2 in the target compound calculation.

This SOP is based on the guidance in S-846 Method 3640A and USEPA *CLP Statement of Work for Organic Analysis, Multi-Media, Multi Concentration, OMO4.2, May 1999.*

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 All solvent transfer steps should be performed quickly and under a hood, if possible. Use the minimum amount of solvent to get the job done. Do not allow open containers of solvent or extracts to evaporate into the lab.

3.3 A lab coat or apron should be worn to protect clothing and skin from acids, bases, and solvents. Gloves must be worn to protect hands and eye protection (safety glasses, goggles) must be worn at all times in the lab. An analyst should not work alone in an isolated area.

3.4 A solvent such as methylene chloride can cause a burning sensation when it contacts the skin. The rapid evaporation of the solvent causes a rapid heat loss in the skin which is similar to frost bite. If this occurs, wash the exposed skin in cold water to reduce the solvent evaporation.

3.5 The analyst should become familiar with the Material Safety Data Sheets (MSDS) for each reagent used in the extraction lab.

4.0 INTERFERENCES

4.1 The extract solvent must be methylene chloride. The presence of high concentrations of other solvents will affect the elution of the target compounds and may damage the GPC column.

4.2 The extract should contain 0.50g or less of residue per 5mL of extract (or the volume of extract loaded into the GPC). A procedure for screening the extract for residue is given in Section 9. If the residue is greater than 0.50g/5mL (0.1g/mL), the extract should be divided into several aliquots each containing less than 0.1g/mLand each aliquots subjected to GPC cleanup. The eluants of each aliquot are combined and evaporated.

4.3 The extract should have a viscosity less than the viscosity of a 1:1 mixture of glycerol and water. If the viscosity is greater, the extract should be diluted and divided into several aliquots for GPC cleanup and the eluants combined and evaporated.

5.0 SAMPLE COLLECTION, HANDLING, AND STORAGE

See the analytical method for the hold time of the extracts. In general, the extract must be subjected to the GPC cleanup and analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gel-permeation chromatography system-automated or manual, including a chromatographic column compatible with the GPC system, a UV detector, and a strip chart recorder or other recorder

6.2 0.45 micron filters

6.3 Hamilton syringes- 10 ml

7.0 REAGENTS

All reagents must be tracked in accordance with STL SOP AN44:*Reagent Traceability*.

7.1 Methylene chloride- pesticide quality or equivalent

7.2 Glycerol-reagent grade

7.3 Reagent water

7.4 1:1 Water/glycerol mixture-combine equal volumes (e.g., 5.0mL each) of glycerol and water and store in container that will minimize evaporation.

8.0 STANDARDS

All standards must be tracked in accordance with STL Sop AN41:*Standard Material Traceability*.

8.1 GPC calibration solution- prepare a calibration solution in methylene chloride containing the following analytes :

COMPOUND	CONCENTRATION (mg/mL)
Corn oil	25
Bis(2-ethylhexyl)phthalate	1.0
Methoxylchlor	0.20
Perylene	0.020
Sulfur	0.080

8.2 Pesticides GPC Check Solution-prepare 10mL of a standard containing the subset compounds at the routine spiking level in methylene chloride.

8.3 PCB GPC Check Solution-prepare 10mL of a standard containing AR1016 and AR1260 at the routine spiking level in methylene chloride.

8.4 BNA GPC Check Solution-prepare 10mL of a standard containing the subset compounds at the routine spiking level in methylene chloride.

8.5 Corn oil screening solution-add 5.0g of corn oil to a 100-mL volumetric flask and dilute to volume with methylene chloride. The theoretical concentration of the solution is 50mg/mL or 5mg/100uL.

9.0 SAMPLE PREPARATION

9.1 All sample extracts must be filtered through a 0.45um filter prior to introduction into the GPC.

9.2 Compare the viscosity of each extract (adjusted to a final volume of 10mL) to the viscosity of a 1:1 mixture of water and glycerol. If the viscosity of the sample extract is greater than the water/glycerol mixture, dilute the extract in to several 10-mL aliquots and perform GPC cleanup on each aliquot. Combine the eluants from each aliquot into a single extract and concentrate to the appropriate final volume.

9.3 Screening the Extract
Screen the extract to determine the weight of dissolved residue by evaporating a 100 µL aliquot to dryness and weighing the residue. The weight of dissolved residue loaded on the GPC column cannot exceed 0.500 g. Residues exceeding 0.500 g will very likely result in incomplete extract cleanup and contamination of the GPC switching valve (which results in cross-contamination of sample extracts).

9.3.1 Transfer 100 µL of the filtered extract to a tared aluminum weighing dish.

9.3.2 A suggested evaporation technique is to use a heat lamp. Set up a 250 watt heat lamp in a hood so that it is 8 + 0.5 cm from a surface covered with a clean sheet of aluminum foil. Surface temperature should be 80-100 C (check temperature by placing a thermometer on the foil and under the lamp). Place the weighing dish under the lamp using tongs. Allow it to stay under the lamp for 1 min. Transfer the weighing dish to an analytical balance or a micro balance and weigh to the nearest 0.1 mg. If the residue weight is less than 10 mg/100 µL, then further weighings are not necessary. If the residue weight is greater than 10 mg/100 µL, then determine if constant weight has been achieved by placing the weighing dish and residue back under the heat lamp for 2 or more additional 0.5 min. intervals. Reweigh after each interval. Constant weight is achieved when three weights agree within +10%.

9.3.3 Repeat the above residue analysis on a blank and a spike. Add 100 µL of the same methylene chloride used for the sample extraction to a weighing dish and determine residue as above. Add 100 µL of a corn oil spike (5 g/100 mL) to another weighing dish and repeat the residue determination. The corn oil spike should agree within 25 % of the theoretical concentration or residue weight.

9.3.4 A residue weight of 10 mg/100 µL of extract represents 500 mg in 5 mL of extract. Any sample extracts that exceed the 10 mg/100 µL residue weight must be diluted so that the 5 mL loaded on the GPC column does not exceed 0.500 g. When making the dilution, keep in mind the minimum volume of extract required to load the GPC unit.

10.0 PROCEDURE

10.1 Column Calibration

10.1.1 Transfer 10 mls of the calibration solution into a sampling vial.

10.1.2 Inject calibration solution and obtain a UV trace showing a discrete peak for each component.

10.1.3 Evaluate the UV chromatogram
-Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
-Corn oil and phthalate peaks must exhibit >85% resolution.
-Phthalate and methoxychlor peaks must exhibit > 85% resolution.
-Methoxychlor and perylene peaks must exhibit >85% resolution.

10.1.4 Establish collection windows
Using the information from the UV chromatogram establish the fraction collection window.

10.1.4.1 Measure the distance from the injection point to the desired collection point (1 cm= 1 minute). This will be the collect start time.

10.1.4.2 Measure the distance from the collect start time to the desired end time (1cm=1 minute). This is the peak fraction window or the time period in which the sample will be collected for analysis.

10.1.4.3 Measure the distance from the injection point to the desired end collection time (1cm= 1 minute) This will be the collect end time. *the difference between the collect end time and the collect start time should equal the value of the peak fraction window.

Semi-volatile MS- initiate collect before bis (2-ethylhexyl) phthalate and end collection after elution of perylene.

8080/608 Pesticides/ PCB's- initiate collect before methoxychlor and end collection after elution of perylene.

10.1.5 Calibration Check- Organochlorine Pesticides and PCB's

10.1.5.1 Before GPC cleanup of pesticide and PCB extracts a calibration check (prepared and analyzed by GC) must be performed. The GPC column is approved for use if the acceptance criteria are met.

-Load 10 mls of Pesticide solution onto injection loop.
-Load 10 mls of Aroclor mixture onto injection loop.
-Concentrate the collected GPC fraction, exchange to Hexane, and adjust the final volume to 10 mls, give to instrument group for assay.

Technical acceptance criteria for calibration check:
The recovery of each of the single component analytes must be >85%
The Aroclor patterns must be the same as those from the Aroclor 1016 and Aroclor 1260 standards in the initial calibration sequence.

If acceptance criteria are not met, check to ensure GPC and fraction collector are programmed correctly according to UV trace chromatogram, load another GPC calibration check and/or recalibrate the GPC column if necessary.

10.2 Extract Clean-Up

10.2.1 Analyze a GPC blank by loading 5 mL of methylene chloride into the GPC. Concentrate the methylene chloride that passes through the system during the collect cycle using a Kuderna-Danish (KD) evaporator. Analyze the concentrate by whatever detectors will be used for the analysis of future samples. Exchange the solvent, if necessary. If the blank exceeds the estimated quantitation limit of the analytes, pump additional methylene chloride through the system for 1-2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping, if necessary.

10.2.2 Adjust the extract volume to 10.0 mL. The solvent extract must be primarily methylene chloride. All other solvents, e.g. 1:1 methylene chloride/acetone, must be concentrated to 1 mL (or as low as possible if a precipitate forms) and diluted to 10.0 mL with methylene chloride. Thoroughly mix the extract before proceeding.

10.2.3 Filter the extract through a 0.45 micron filter disc by attaching a syringe filter assembly containing the filter disc to a 10 mL syringe. Draw the sample extract through the filter assembly and into the 10 mL syringe. Disconnect the filter assembly before transferring the sample extract into a small glass container, e.g. a 15 mL culture tube with a Teflon lined screw cap. Alternatively, draw the extract into the syringe without the filter assembly. Attach the filter assembly and force the extract through the filter and into the glass container. The latter is the preferred technique for viscous extracts or extracts with a lot of solids. Particulate larger than 5 microns may scratch the valve, which may result in a system leak and cross-contamination of sample extracts in the sample loops. Repair of the damaged valve is quite expensive.

NOTE: Viscosity of a sample extract should not exceed the viscosity of 1:1 water/glycerol. Dilute samples that exceed this viscosity.

10.2.1 Adjust the extract volume (if necessary) to 10mL. The extract solvent must be methylene chloride.

10.2.2 Filter the extract through a .45 micron filter disc.
Attach the filter disk to a 10 ml syringe.
Draw the sample into the 10 ml syringe, remove needle and replace with filter disk.
Force the extract through the filter into a 15 ml culture tube.

10.3 GPC Set-up and Operation

10.3.1 Program GPC system. (Refer to Manufacturer's instructions for set-up and programming information).

10.3.2 Program Fraction Collector (Refer to Manufacturer's instructions for set-up and programming information).

10.3.3 Prepare collection tubes in accordance with SOP AN60. There must be one labeled collection tube for each sample in analytical batch.

10.3.4 Load samples, begin program.

10.3.5 Check periodically to ensure proper operation and ample supply of methylene chloride is available for syringe flushes and fraction collection.

10.4 Concentrate extracts in accordance with Extraction Zymark SOP EX50.

11.0 CALCULATIONS-

See analytical procedures for the quantitation of target compounds. Note that one half of the extract is lost in loading the GPC. This loss of sample, must be compensated for in the calculation (multiply by 2) or in the final volume of the extract (evaporate to one half the routine final volume)

12.0 QUALITY CONTROL/QUALITY ASSURANCE-see SOP Summary

13.0 PREVENTIVE MAINTENANCE-see manufacturer's operation manual

14.0 TROUBLE-SHOOTING-see manufacturer's operation manual

15.0 REFERENCES

15.1 W-846 Method 3640A "*Gel-Permeation Cleanup*"

15.2 *SEPA CLP Statement of Work for Organic Analysis, Multi-Media, Multi Concentration, OMO4.2, May 1999.*

QC ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
GPC Calibration- (to establish dump/collect times)	Initially; when columns changed; when calibration verification fails	GPC Check solutions meet criteria -Peaks must be observed and should be symmetrical for all compounds in the calibration solution. -Corn oil and phthalate peaks must exhibit >85% resolution. -Phthalate and methoxychlor peaks must exhibit > 85% resolution. -Methoxychlor and perylene peaks must exhibit >85% resolution. -GPC Check solutions must pass acceptance criterion	-Adjust the column flow parameters to optimize separations -Repeat the GPC cleanup of check solutions -Recalibrate the GPC column -Check system for leaks and verify the volume collected -Inspect the GPC column for channeling and discoloration-clean column according to manufacturer's manual. -Prepare new column and calibrate
GPC Check Solutions -BNA Check Solution -Pesticide Check Solution -Aroclor Check Solution	When dump/collect times established	Target compounds >85% recovery	-Repeat the GPC cleanup of check solutions -Recalibrate the GPC column -Check system for leaks and verify the volume collected -Inspect the GPC column for channeling and discoloration-clean column according to manufacturer's manual. -Prepare new column and calibrate
GPC Calibration Verification (to verify dump/collect times)	At least once every seven days when samples are processed	-Retention times of GPC Calibration Solution within 5% of initial calibration	-Check system for leaks and verify the volume collected -Inspect the GPC column for channeling -Recalibrate the GPC column
GPC eluant volume	Monitor each sample	Within 25% of calibration volume	-Check system for leaks and verify the volume collected -Inspect the GPC column for channeling -Recalibrate the GPC column
Temperature	Monitor daily	<=72F (to prevent out gassing of methylene chloride)	-Adjust temperature and verify calibration if shifts are noted -Recalibrate the GPC column if shifts are outside acceptance criterion
Column back pressure	Monitor daily	Monitor	

B-9 Preparation of Organics in Biological Tissues

PREPARATION OF ORGANICS IN BIOLOGICAL TISSUES

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Approved by:

Title: STL-SL Technical Manager Date: Jan 28, 2000

1.0 SCOPE AND APPLICATION

This SOP describes the process for extracting semivolatile organic compounds (SVOC) from biological matrices by ultrasonic (sonication) extraction. Separate extraction procedures are used for BNAs (8270C), chlorinated pesticides (8081), PCBs as Aroclors (8082), PCB homologues (680), and chlorinated herbicides (8151).

2.0 SUMMARY OF METHOD

2.1 The tissue is thoroughly homogenized in a blender. A known weight of a tissue (approximately 10g) is combined with anhydrous, purified sodium sulfate to form a free flowing, sandy mixture. A solvent is added to the dried sample and the sample is extracted using an ultrasonic disrupter for three (3) minutes. The solvent is decanted and the extraction is repeated two more times. The extract is filtered and concentrated to an appropriate final volume. The extract is cleaned up using optional Gel Permeation Chromatography (GPC) or other analyte-specific cleanups

2.2 This procedure is based on SW-846 Method 3550B and EPA Region IV Method 0B 10.90 *"Extraction and Analysis of Organics in Biological Tissue."*

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 This SOP contains procedures that are designed to reduce the exposure of lab personnel to solvent vapors and to minimize the amount of solvent introduced into the lab air. All solvent transfer steps should be performed quickly and under a hood, if possible. Use the minimum amount of solvent to get the job done. Do not allow open containers of solvent or extracts to evaporate into the lab.

3.3 A lab coat or apron should be worn to protect clothing and skin from acids, bases, and solvents. Gloves must be worn to protect hands and eye protection (safety glasses, goggles) must be worn at all times in the lab. An analyst should not work alone in an isolated area.

3.4 A solvent such as methylene chloride can cause a burning sensation when it contacts the skin. The rapid evaporation of the solvent causes a rapid heat loss in the skin which is similar to frost bite. If this occurs, wash the exposed skin in cold water to reduce the solvent evaporation.

3.5 The analyst should become familiar with the Material Safety Data Sheets (MSDS) for each reagent used in the extraction lab.

4.0 INTERFERENCES

4.1 Improperly cleaned glassware or equipment can contribute interferences to the sample extract. A thorough acetone rinsing will help to eliminate or minimize this problem.

4.2 Biological tissues contain high levels of lipids that are not routinely encountered in environmental samples. Gel permeation chromatography must be used to reduce the lipid concentration of the extract.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Tissues are routinely collected and stored in glass containers or wrapped in aluminum foil. The tissue should be kept frozen until the time of preparation. A holding time of six months for the frozen fish fillets is recommended by Alabama Department of Environmental Management (ADEM) and will be used for all biological tissues.

6.0 APPARATUS AND MATERIALS

6.1 Ultrasonic disrupter: Tekmar Model or equivalent with ¾ inch horn-type titanium-tipped sonication probe. The sonicator should be capable of operating in the pulse mode at full power. The sonicator must have a minimum power of 300 watts.

6.2 Sonabox - the sonicator should be placed in the sonabox to reduce noise. The sonabox must be placed under a fume hood.

6.3 Pre-cleaned 500mL extraction bottles (P/N SAV-145). Discard the bottles after use.

6.4 Large glass funnels

6.5 Filter paper-18.5cm

6.6 Pyrex glass wool - purchased purified or extracted with methylene chloride for a minimum of four (4) hours.

6.7 Stainless steel spatulas

6.8 Disposable volumetric pipets

6.9 Top-loading balance - capable of weighing to 0.1g. The balance calibration must be verified according to STL-SL SOP AN10 each time the balance is used.

6.10 Stainless steel blender-heavy duty type capable of blending tissues.

6.11 Dry ice

7.0 REAGENTS

7.1 Sodium sulfate, powdered and granular, anhydrous - purified by heating at 400C for four hours in a shallow tray.

7.2 Methylene chloride - residue grade or better

7.3 Acetone - residue grade or better

7.4 1:1 acetone/methylene chloride: Mix equal volumes of acetone and methylene chloride in an appropriate glass container.

7.5 Hexane-residue grade or better

7.6 1:1 acetone/hexane: Mix equal volumes of acetone and hexane in an appropriate glass container.

Note: 1:1 acetone/hexane or 1:1 acetone methylene chloride may be used for the extraction of pesticides and PCBs in soils. If the 1:1 acetone/methylene chloride is used, a solvent exchange to hexane is required.

8.0 STANDARDS

The preparation of the surrogate and matrix spiking solutions are described in STL-SL SOP EX70: *Preparation of Semivolatile Surrogates and Matrix Spiking Solutions.* Table 2 is a summary of the spiking solutions used for the various analytical extraction methods. All preparation steps must be tracked in accordance with STL-SL SOP AN41:*Standard Material Traceability.*

The tables included in SOP EX70 describe "recipes" that can be used to prepare the spiking solutions. The lab can prepare larger or smaller volumes if desired. The recipe used by the lab may be prepared as a "controlled posting" to prompt the analyst in the preparation of the spiking solutions. The controlled posting can be posted in the lab or placed in the extraction lab tracking log.

9.0 SAMPLE PREPARATION

Organic compounds are generally not evenly distributed throughout biological tissue. There fore, the entire sample (whole or filet) should be ground into a homogenous consistency prior to extraction.

9.1 Sample Homogenization

9.1.1 Chop the sample into one inch or smaller chunks and place in the blender. Slightly frozen tissue is easier to work with at this point.

9.1.2 Add dry ice to the blender. Blend on appropriate power level until the tissue is thoroughly ground and homogenized. Inspect the tissue for the presence of "chunks" of tissue. Remove the well homogenized tissue and blend the remaining "chunks" until homogenized.

9.1.3 Mix the tissue thoroughly and separate into 10-g portions for the required analyses. If the extraction is not to take place soon after homogenization, the homogenized tissue or the individual 10-g portions can be frozen.

9.2 Extraction Glassware Set-up

9.2.1 Remove samples to be extracted from the storage freezer and allow the samples to come to room temperature while the extraction glassware is being prepared.

9.2.2 Collect the appropriate glassware and rinse with acetone prior to use. Use the minimum amount of solvent to rinse the glassware.

9.2.3 Fit a filter pad into a large funnel and rinse the filter paper/funnel with acetone.

9.3 Sample Extraction
Record as much information on the extraction log as possible before beginning the extraction to minimize the number of entries required during the extraction. The extraction steps from weighing to the sonication should be carried out quickly to minimize the loss of SVOC through evaporation.

9.3.1 Weigh 10.0 - 10.5g of the homogenized tissue into a pre-cleaned, labeled 500-mL extraction bottle. Record the weight for all samples in this batch.

Weigh 30g of powdered, purified sodium sulfate into each of two separate extraction vessels to serve as the method blank and lab control spike (LCS). If a "control tissue" has been obtained for QC, use the control tissue for the method blank and LCS, treating them in the same manner as the samples tissues.

Weigh two additional 10-g portions of a sample in the batch selected as the matrix spike (MS) and matrix spike duplicate (MSD). The MS/MSD may be specified by the client or may be chosen from the samples in the batch.

9.3.3 Add approximately 60g of powdered, purified sodium sulfate to each sample and MS/MSD and stir with a glass rod or stainless steel spatula to form a sandy, free-flowing mixture. The sodium sulfate combines with the water in the sample to "dry" the sample (remove the water). Additional sodium sulfate may be required if the sample is very wet.

NOTE: The next steps should be performed immediately after the sodium sulfate is added to minimize the loss of SVOC due to evaporation

9.3.4 Add 1.0mL of the appropriate surrogate spiking solution (Table 1) to the method blank, LCS, MS, MSD and each sample in the batch.

9.3.5 Add 1.0mL of the appropriate matrix spiking solution (Table 1) to the LCS, MS, and MSD. Place the extraction bottle under the hood near the sonicator.

9.3.6 Working under a hood, immediately add the extraction solvent to cover the sample approximately one inch above the solids. Stir the sample to break up any lumps that may have formed. See Table 2 for the appropriate extraction solvent.

9.3.7 Check the "tune" of the sonicator using the procedure tested in STL-SL SOP AN15: *Tekmar Sonic Dismembrator Tuning Procedure.*

9.3.8 Place the tip of the sonicator horn in the center of the beaker about ½ inch below the surface of the solvent but above the solid portion. More solvent may be added to bring the level to approximately one inch above the solid layer.

9.3.9 Sonicate for three (3) minutes with output control knob set at 10, mode switch to pulse, and percent duty cycle set at 50%. If the sonication is properly performed, the solids and solvent will vigorously mix each time the sonicator pulses.

9.3.10 Decant the extract through the filter funnel and collect the extract in a beaker or directly in a labeled, "elongated" Zymark tube. The elongated tubes hold approximately 225mL of solvent; therefore, it is desirable to limit the volume of the three extracts to 225mL. If the sample is very wet and requires a large amount of sodium sulfate and a larger volume of solvent, the extracts are collected in a larger beaker.

9.3.11 Repeat steps 9.2.6 through 9.2.10 two more times with fresh portions of solvent and collect the extract in the Zymark tube. Cover the Zymark tube with aluminum foil and leave it under the hood until the concentration step.

NOTE: Clean the sonicator horn between samples by rinsing with acetone. "Sonicate" a blank extraction bottle of acetone for 3 minutes with output control at 5 to further clean the horn (sodium sulfate may be added to the beaker to prevent cracking).

9.3.13 Dispose of extraction bottle and sample residue by placing in a chlorinated solvent waste container which is kept under a hood until removed from the laboratory.

9.4 Percent Lipid Determination

9.4.1 Transfer 1.0mL of the extract that will be subjected to the GPC cleanup to a tared aluminum weigh boat. This will not affect the final volume as only 5mL of the extract will be loaded onto the GPC-the remaining 4mL will lost in the transfer to the GPC.

9.4.2 Place the boat in a hood and allow the solvent to evaporate overnight. Handle the boat with tongs and protect from particulates.

9.4.3 Reweigh the boat and calculate the percent lipids as follows:

$$\%Lipids = \frac{(Wr - Wb)}{1mL \otimes \frac{Wsample}{10mL}} \otimes 100$$

where
Wr = weight of residue and aluminum weigh boat (g)
Wb = tare weight of the aluminum weigh boat (g)
Wsample = weight of sample extracted (g)

9.4 Sample Cleanup
All extracts should be subjected to GPC cleanup prior to analysis to reduce the lipid content of the extract.

9.4.1 Concentrate the extract to approximately 1mL or as low as practical..

9.4.2 Add methylene chloride to give a final volume of 10mL.

9.4.3 Perform GPC cleanup as required. See STL-SL SOP PS15 for cleanup procedures for PCBs as Aroclors or congeners.

9.4.4 Concentrate the extract to the final volume required by the analysis (Table 2). Additional cleanups may be required for the GC parameters.

9.4.5 For chlorinated herbicides, concentrate the extract to 10m and exchange to diethyl ether. Continue the herbicide extraction from Section 9.2.18 of STL-SL SOP EX45.

10.0 ANALYTICAL PROCEDURE

8270-BNA-STL SOP SM05
8081-Chlorinated Pesticides-SG45
8082-PCBs as Aroclors-SG45
680-PCB Homologues PS15 and SM07
8151-Chlorinated Herbicides-SG65

11.0 CALCULATIONS

See Analytical Procedure-calculation same as soils except reported "as is".

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

STL-SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with STL-SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

15.3 *"Extraction and Analysis of Organics in Biological Tissues"*, Method 0B 10/90, USEPA Environmental Services Division, Region IV, Analytical Support Branch, Athens, GA.

Table 1 -Surrogate and Matrix Spiking Solutions

METHODS	TYPES OF COMPOUNDS	SURROGATE SPIKING SOLUTION	MATRIX SPIKING SOLUTION
8081	Chlorinated pesticides	PEST	8080
8082	PCBs	PEST	PCB
8100	PAH by GC-FID	SG BASE	8100
8151	Chlorinated Herbicides	See EX45	See EX45
8270-TCL	BNA	625/8270	8270
608	PCB Homologues	See PS15 and SM07	See PS25 and SM07

Table 2 -Extraction Conditions

METHODS	TYPES OF COMPOUNDS	EXTRACTION CONDITIONS	SOLVENT	FINAL SOLVENT	FINAL VOLUME (mL)
8081, 8082	Chlorinated pesticides and PCBs	As received	1:1 Ace/Methylene Chloride	Hexane	5.0mL
8151	Chlorinated herbicides	As received	Diethyl ether	Hexane	5.0mL
8270-TCL	BNA	As received	1:1 Ace/MeCl2	MeCl2	0.50mL
680	PCB Homologues	As received	1:1 Ace/Chloride	Hexane	0.50mL

B-10 Total Organic Carbon-Shimadzu TOC Analyzer

BA09:05.01.98:1

Approval Signature:	R. Wayne Robbins
Title: Corporate QA Manager	Date: 5/1/98

TOTAL ORGANIC CARBON-SHIMADZU TOC ANALYZER

1.SCOPE AND APPLICATION

1.1 This method is applicable to the determination of total organic carbon (TOC) in ground water, surface water, drinking water, domestic and industrial wastewater, soils, sediments, and other solids.

1.2 The linear range for TOC aqueous samples is from 1mg/L to 500mg/L for a 250uL injection volume. Samples with TOC concentrations greater than 500mg/L require dilution before analysis. The linear range for soils and solids samples is from 500mg/kg to 235000mg/kg for a 100mg sample.

1.3 The reporting limit(RL), the method detection limit(MDL), and the accuracy and precision criteria are given in Table 5 of the Savannah Labs Comprehensive Quality Assurance Plan (1) or client-specified QAPP.

2.SUMMARY OF METHOD

2.1 Total organic carbon (TOC) is measured by treating a sample with hydrochloric acid (HCl) and purging the sample to eliminate the inorganic carbon present in the sample. The HCl converts any carbonates present in the sample to CO2, which is then purged out of the sample. Purging the sample also eliminates any volatile organic compounds from the sample matrix; therefore, any direct measurement of TOC is actually non-purgeable TOC. The acid treated sample is then combusted at 680C for liquids and 900C for soils. The carbon is converted to carbon dioxide and the carbon dioxide is measured by an infrared (IR) detector. The amount of carbon dioxide, expressed as carbon, is directly proportional to the carbon content of the sample.

2.2 It is important that the analyst know the type of carbon analysis to be determined so that the proper sample can be used for the analysis. The following "equation" shows the relationship between the various forms of carbon that can be measured with this procedure:

$$TC = TOC + TIC$$

where

TC = total carbon
TOC = total organic carbon
TIC = total inorganic carbon

Since TOC has two components, purgeable and non-purgeable, the "equation"can be expanded:

$$TC = npTOC + pTOC + TIC$$

where npTOC = non-purgeable TOC
pTOC = purgeable TOC

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Three of these forms of carbon can be measured directly- TC, npTOC, and TIC. The pTOC has to be determined by difference.

In most cases, the pTOC is very small compared to the npTOC and the npTOC is reported as the "total organic carbon".

2.3 This procedure is based on EPA Method 415.1(2), SW-846 Method 9060(3), and the SL COMPQAP(1).

3.SAFETY

3.1 The analyst must protect herself/himself from exposure to the sample matrix. Many of the samples analyzed for carbon content are potentially hazardous. The analyst must wear protective clothing (lab coat or apron), eye protection (eyeglasses or face shield), and disposable gloves when handling the samples.

3.2 The furnace on the carbon analyzer and on the soil analyzer must be cooled to room temperature before maintainence can be performed. The temperature of the analyzer furnace can reach 680C and the soil furnace can reach 900C. The analyst must be very careful to avoid touching these very hot surfaces.

3.3 Care must be taken when handling hydrochloric acid(HCl) and solutions of hydrochloric acid. The inorganic carbon reagent is 2N HCl and must be handled with caution. This acid will cause chemical burns and destroy unprotected clothing. The analyst must also use extreme caution when adding acid to soil samples. The acid should be added slowly to minimize potentially violent reactions that will splatter acid.

3.4 The carrier gas MUST remain on while the instrument is in operation. The analyst must ensure that there is sufficient carrier gas available in the tank to complete the run. Failure to have carrier gas running through the instrument while the combustion furnace is at the operating temperature may result in melting of the combustion tubes.

3.5 The Material Safety Data Sheets(MSDS) for each regent and standard used in this procedure are available for inspection and review by each analyst at each lab division. The MSDS contain information on the potential hazard that each reagent poses and the guidelines for safely handling the reagent. The MSDS also contains some first aid information and should be taken to the first aid center or hospital with any person that requires a higher level of medical attention after exposer to the reagent.

4.INTERFERENCES

4.1 Sample particulates that are larger than the syringe will not be included in the carbon determination. This condition may result in low results if the particulates contain measurable levels of carbon. The analyst should be alert to samples that may clog the sample syringe.

4.2 Liquid samples containing oil or are oily in nature will deactivate the catalyst. The catalyst will have to be regenerated or replaced prior to running other samples. It is highly recommended that oily

samples be analyzed using the soil module or that the CCV and CCB be analyzed immediately after these samples to evaluate the condition of the catalyst.

4.3 It is very important that the inorganic carbon be removed from the sample prior to the direct determination of the TOC (non-purgeable TOC). Failure to remove the inorganic component of the sample may result in artificially high TOC results.

5.SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Aqueous samples for total organic carbon are collected in 125-mL amber glass containers with zero headspace and preserved with HCl to pH < 2. A light blue dot is placed on the container to indicate that the sample has been preserved with HCl. The sample is iced at the time of collection and is stored at 4C +/- 2C until the time of analysis.

The pH of each aqueous sample for TOC must be determined and recorded when the sample arrives in the lab to ensure proper preservation.

5.1.1 Pour a small aliquot of the well-mixed sample into a small plastic cup.

5.1.2 Dip a piece of wide range pH paper into the sample in the cup. NEVER dip the pH paper into the sample container.

5.1.3 If the pH is less than 2 (<2), the preservative is correct and the sample can be stored in the cooler.

5.1.4 If the pH of the sample is greater than 2 (>2), adjust the pH to less than 2 (<2) with small aliquots of 1:1 HCl solution. Thoroughly mix the acid and sample and re-test another aliquot of the sample. Continue adding small aliquots of acid to the sample until the pH is less than 2. Record the pH of the sample along with any adjustments and/or comments on the TOC sample pH log.

5.2 Soil and sediment samples are collected in 125-ml amber glass containers with minimum headspace. The samples are iced at the time of collection and are maintained at 4C +/- 2C until the time of analysis.

5.3 The hold time for an aqueous sample preserved with HCL is 28 days from the date of collection. The maximum hold time for soil and sediment samples is 28 days from the date of collection.

6.APPARATUS AND MATERIALS

6.1 Shimadzu 5050A Carbon Analyzer

6.2 Volumetric pipets

6.3 Filters (0.45um) and disposable filters

6.4 Volumetric flasks

6.5 Disposable transfer pipets

6.6 Ceramic boats for soil analyses

6.7 Aluminium pans

6.8 Ultra pure compressed air, regulator and appropriate fittings. The compressed air must contain less than 1ppm of carbon dioxide. This gas is used as the carrier and sparge gas for the analysis of aqueous samples.

6.9 Oxygen, regulator, and appropriate fittings. The oxygen is used as the carrier gas in the analysis of soil samples.

7. REAGENTS

7.1 Reagent water- lab-generated deionized water

7.2 Hydrochloric acid (HCl)-concentrated, reagent grade or better. Record the acid lot number in the reagent prep log.

7.3 1:1 Hydrochloric acid solution (50% HCl)- Add approximately 400mL of reagent water to a 2.0-L beaker. Place the beaker on a magnetic stir plate located under a hood. Add a Teflon stir bar to the reagent water and turn the stir plate on. SLOWLY and CAREFULLY add 500mL of concentrated HCl to the beaker in small aliquots. Hydrochloric acid is extremely toxic and has a suffocating odor. After the entire 500-mL of HCl has been added, continue mixing and allow the solution to cool. After the solution has cooled, transfer it to a 1.0-L volumetric flask and dilute to volume with reagent water. This solution should be stored in a glass container. Prepare this solution every six months or as needed. Record the preparation in the reagent prep log.

7.4 2 N HCl Solution-Slowly add 42mL of concentrated HCl to about 150mL of reagent water contained in a 250-mL volumetric flask. Dilute to volume with reagent water. Transfer the reagent to a glass container. This reagent must be prepared under a hood. HCl has a suffocating odor and can cause severe chemical burns.

7.5 Vanadium pentoxide-reagent grade

7.6 Tungsten oxide-reagent grade

8.STANDARDS

8.1 Stock standards

8.1.1 Potassium hydrogen phthalate (KHP)-reagent grade or better.

8.1.2 Potassium hydrogen phthalate, 2000mg Carbon / liter

Dissolve 0.4256g of reagent grade KHP in approximately 800mL of reagent water contained in a 1.0-L volumetric flask. Dilute to volume with reagent water and transfer the stock standard to an amber glass container. Label the container with the standard identification, concentration, analyst's initials, date prepared, and the expiration date. Record the preparation in the standards prep log. Store the standard in the refrigerator

at 4C +/-2C. Prepare this standard monthly or as needed.

8.2 Calibration Standards-TOC

The KHP stock standard is used to prepare the calibration standards for the determination of TOC. The pH of each calibration standard is adjusted to less than 2 with 1:1 HCl. The following table gives the volumes of stock standard needed to prepare the given volume of the calibration standard:

Calibration Standard (mg/L)	mL of 2000mg/L Stock Standard	mL of Reagent Water	Final Volume
500	250	750	1000
200	100	900	1000
100	50	950	1000
50	25	975	1000
10	5.0	995	1000
5.0	2.5	997.5	1000
1.0	0.50	999.5	1000

The concentration of the calibration standard can be determined using the following equation:

$$(Cs)(Vs) = (Ccal)(Vcal)$$

$$Ccal = \frac{(Cs)(Vs)}{(Vcal)}$$

where

Cs = concentration of the stock standard (mg/L)
Vs = volume of stock standard needed to prepare the calibration standard(L)
Ccal = concentration of the calibration standard to be prepared (mg/L)
Vcal = volume of the calibration standard to prepare (L)

9. SAMPLE PREPARATION

9.1 There is essentially no sample preparation for aqueous samples. The samples are loaded on to the autosampler and the sampling sequence is initiated. The analyst should be alert for samples with high solids and particulates that may clog the syringe or may produce results that are lower than might expected.

NOTE: It may be difficult to establish correlation between the TOC, BOD, and COD for samples with high particulates. The syringe system used by the Shimadzu instrument may effectively filter out particulates that may be included in the determination of BOD and COD.

9.2 Soil samples for TOC analysis are dried to produce a free flowing, homogeneous sample. The dried sample is treated with concentrated HCl to convert the carbonates and bicarbonates to carbon dioxide, which evolves from the sample.

9.2.1 Weigh approximately 5g to 10g of the soil or sediment sample into an aluminum pan.

9.2.2 Place the pan in the oven and allow the moisture to evaporate over night at 105C.

NOTE: Heating the soil will drive off the volatile organics. The TOC measured from the soil is the non-purgeable or non-volatile component of the total organic carbon. Since the volatiles are usually very low compared to the non-volatile, the non-volatile component is reported as the TOC.

9.2.3 Remove the samples from the oven. Crush any lumps that may be present with a spatula or spoon and mix the sample to obtain a homogeneous sample.

9.2.4 Weigh out five (5) 100mg portions of the sample into the ceramic "boats". Record the weight of each sample to the nearest 0.1mg on the analysis log.

9.2.5 Add approximately 0.10mL of concentrated HCL to each ceramic "boat". Additional HCl can be added if the soil has a high carbonate content. This step should be performed in a hood.

9.2.6 Place the sample "boats" into the oven to allow the excess HCL to evolve. The sample is now ready for analysis.

10.PROCEDURE

10.1 Aqueous Samples-TOC

10.1.1 Turn the Shimadzu 5050A on and set the following parameters:

Analyzer Furnace: 680 C
Carrier Gas: 150mL/min (ultrapure compressed air)
Purge Gas: 50mL/min (ultrapure compressed air)
Injection Volume: 250uL (0.250mL)
Minimum Number of Replicates: 3
Maximum Number of Replicates: 5

Allow the instrument to equilibrate and the furnace to reach the set point before processing samples.

10.1.2 Set up the calibration standards on the autosampler. The lowest standards should be analyzed first. The analytical sequence is :

Range 1 Calibration Standards:
1.0mg/L
5.0mg/L
10mg/L

Range 2 Calibration Standards
1.0mg/L
10mg/L
50mg/L
100mg/L

Range 3 calibration Standards
1.0mg/L
100mg/L

200mg/L
500mg/L

Initial Calibration Verification(ICV/LCS)
Initial Calibration Blank (ICB)
10 client samples (in replicate)
Continuing Calibration Verification(CCV/LCSD)
Continuing Calibration Blank (CCB)
10 client samples (in replicate, including an MS/MSD per batch of twenty samples)
CCV/LCS
CCB

This sequence continues until there are no more samples to fill the slots. The CCV and CCB must "cap" the end of the analysis sequence. Note that the ICV and the CCV are from a separate source (ERA) and are evaluated for calibration verification and overall analytical control(accuracy and precision)

The initial calibration is performed each time samples are analyzed.

10.1.3 Record the sample identifications on the analysis log sheet to correspond to the position occupied by the samples on the autosampler tray.

10.1.4 Start the analyzer. The samples will be processed automatically.

10.1.5 After the sequence has ended, evaluate the QC and the sample results.

10.1.5.1 If the ICV or a CCV or the ICB or a CCB has failed, the samples analyzed between the failed CCV or CCB must be re-analyzed. The result for the ICV/CCV must be 80-120% of the true value. The results for the ICB/CCB must be less than the quantitation limit of 1mg/L.

10.1.5.2 If a sample contains a concentration of TOC greater than the linear range of the calibration curve, the sample must be diluted and re-analyzed.

10.2 Soil Samples-TOC The TOC analyzer is initially calibrated using a 1-point calibration curve on each analysis range. The curve is plotted from zero through the area corresponding to the standard for the specified range. The calibration levels are:

RANGE	mg KHP	mg Carbon
X1	2.1	1.0
X5	8.0	3.8
X30	50	23.5

These standards define the linear range of the instrument.

10.2.1 Turn the Shimadzu 5050A and soil analyzer module on and set the following parameters:

Soil Analyzer Furnace:	900 C
Carrier Gas:	50mL/min (oxygen)

Allow the instrument to equilibrate and the furnace to reach the set point before processing samples.

10.2.2 Before samples are processed, a 1.0-mL aliquot of 2N HCl is combusted in the furnace to clean and regenerate the catalyst.

10.2.2.1 Add 0.10mL of 2N HCl to the ceramic "boat"

10.2.2.2 Add sufficient ceramic "wool" to cover the acid.

10.2.2.3 Place the "boat" into the furnace and allow the sample to cycle through the combustion and cooling steps.

10.2.3 A method blank, consisting of the ceramic "boat", ceramic "wool" and metal catalyst (vanadium pentoxide or tungsten oxide), is then processed through the combustion and cooling cycle to evaluate the contribution of the "boat", ceramic "wool", and the metal catalyst to the overall TOC analysis. If the weight of a "blank sample" is assumed to be 100mg, the method blank should be less than 500mg/kg,dw.

10.2.4 After the method blank has been analyzed, the following QC Check Standards are analyzed: four 20-mg portions of dextrose and one 15-mg to 18-mg portion of the EPA Municipal Digested Sludge. The recovery of each of these check standards must be within the SL COMPQAP Table 5 criteria for recovery. The duplicate analyses of the dextrose standards ar e used to evaluate precision, which must be less than the Table 5 limit for %RPD

10.2.5 The client samples are prepared as described in Section 9.2. It is important that all of the inorganic carbon be converted to CO2 prior to the analysis of the soil for TOC.

10.2.6 One of the five prepared portions of soil is analyzed on the X5 range to determine the proper range for that particular sample.

10.2.6.1 If the result for the sample is within the X5 range, that is , if the result is quantifiable but less than the high standard (within the linear range), the X5 range is used and three of the other portions are analyzed on the X5 range. Each sample must be analyzed in quadruplicate.

10.2.6.2 If a peak is not quantified, a second 100mg portion of the sample is analyzed on the X1 range. The three remaining portions of sample are analyzed on the X1 range. Each sample must be analyzed in quadruplicate.

10.2.6.3 If the concentration of the initial analysis exceeds the linear range on the X5 range, a second portion of the sample is analyzed on the X30 range. If the result on the X30 range is within the linear range, the remaining portions are analyzed on the X30 scale. Each sample must be analyzed in quadruplicate.

If result is above the linear range on the X30 range, a smaller aliquot of sample should be weighed and analyzed.

10.2.7 Each soils sample must be analyzed in quadruplicate on the same range setting. The average result of the four replicates is reported unless a request is made for the result of each individual replicate.

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11. DATA ANALYSIS/CALCULATIONS

11.1 The concentrations of TOC in client and QC samples are taken from the linear regression curve.

11.1.1 The results for liquid samples are calculated as follows:

mg/L (in sample) = mg/L (from printout) x DF

where
DF = dilution factor

11.1.2 The Reporting Limit (RL) is calculated:

$RL(mg/L) = RL_{qap} \times DF$

where RL_{qap} = reporting limit from Table 5, SL COMPQAP
DF = dilution factor

The SL Quality Assurance Plan Reporting Limit assumes that DF=1.

11.1.3 The results for soil sample is calculated as follows:

mg/kg,dw = mg/kg(from curve)

Since the sample is dried prior to analysis, the result from the printout is given on a "dry weight basis". The weight of the soil is entered into the data system and the result is calculated as mg/kg, dw since the sample is dried prior to analysis.

11.1.4 The Reporting Limit (RL) is calculated:

$$mg/kg,dw = RL_{qap} \times \frac{100mg}{W}$$

where
RL_{qap} = reporting limit from Table 5, SL COMPQAP
W = weight of sample analyzed (mg)

The SL Quality Assurance Plan Reporting Limit and assumes that 100mg(0.100g) of sample is analyzed.

11.2 The concentration of TOC in the lab control and matrix spike samples is determined as in Section 11.1. The concentration is compared to the theoretical spike concentration and the percent recovery is calculated.

11.2.1 0.0mL of the 50mg/L matrix spiking solution is added to a 250mL aliquot of a liquid sample or to 10.0g of a soil sample(the soil sample is mixed with 250mL of reagent water)

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11.2.1.1 The theoretical concentration of an aqueous sample that has been spiked with the matrix spiking solution is

$$mg/L = \frac{0.5mL \times 2000mg/L}{100mL} = \frac{0.0005L \times 2000mg/L}{0.100\ L} = 10mg/L$$

11.2.1.2 The theoretical concentration of a soil sample that has been spiked with a 20mg portions of dextrose is:

$$mg/kg,dw = \frac{20mg \times 0.40mg\ C/mg}{100mg} = \frac{8mg\ C}{100mg} = 80000\ mg/kg$$

11.2.1.3 The percent recovery of the matrix spike in the matrix spike sample is calculated:

$$\%recovery\ (\%REC) = \frac{(Cms - Cs)}{Ts} \times 100$$

where

Cms = concentration of the spiked sample (MS or MSD)
Cs = concentration of the unspiked sample
Ts = theoretical concentration of the spike

11.2.1.4 The matrix spike samples are analyzed in duplicate. The relative percent recovery(%RPD), a measure of precision, is calculated for the MS/MSD pair as follows

$$\%RPD = \left| \frac{\%REC(MS) - \%REC(MSD)}{(\%REC(MS) + \%REC(MSD))/2} \right| \times 100$$

11.2.2 The lab control standard is purchased from ERA (Environmental Resource Associates). The standard comes in a vial that is diluted to a specified volume with reagent water. The results are compared to the values contained on the certificate of analysis. The lab control sample for soil samples is dextrose. Twenty milligrams of dextrose is weoghed into a ceramic boat and analyzed as a sample. An EPA sample, municipal digested sludge, is also analyzed as a control standard.

11.2.2.1 The recovery of the lab control standard is calculated as follows:

$$\%REC = \frac{Clcs}{Ct} \times 100$$

Where

Clcs = concentration of the LCS or LCSD
Ct = theoretical concentration of the LCS or LCSD

11.2.2.2 The lab control standards samples are analyzed in duplicate. The relative percent recovery(%RPD), a measure of precision, is calculated for the LCS/LCSD pair as follows

$$\%RPD = \left| \frac{\%REC(LCS) - \%REC(LSCD)}{(\%REC(LCS) + \%REC(LCSD)) / 2} \right| \times 100$$

12. QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items. The QC items for an analytical batch consist of a method blank, a lab control standard(LCS), a lab control standard duplicate(LCSD), a matrix spike(MS), and a matrix spike duplicate(if there is sufficient volume to perform the MS/MSD)

12.2 Evaluation of QC Data

12.2.1 The method blank is reagent water that is taken through all of the analytical procedures. The concentration of TOC in the method blank is a measure of the contamination due to the entire analytical process.

12.2.1.1 The concentration of TOC in the method blank must be less than 1.0mg/L for liquids and 500mg/kg for soils(Assuming a "soil" weight of 100mg(0.1000g).

12.2.1.2 If the concentration of TOC in the method blank is greater than 1.0mg/L for liquids and 500mg/kg for soils, contact the immediate supervisor to determine the cause of the contamination. If the cause of the contamination cannot be determined and corrected, the batch of samples processed with the method blank must be reanalyzed.

12.2.2 The lab control standard (LCS or LCSD) is a standard obtained or prepared from a separate source (a source different from the source of the calibration standards) and taken through all of the analytical procedures. The LCS for TOC is purchased from ERA. A certificate of analysis must accompany the lab control standard and the certificate must be kept as a record of the true (theoretical) concentration of the LCS.

12.2.2.1 The recoveries(%REC) of the LCS and LCSD must be between 80% and 120%. The relative percent difference(%RPD) between the LCS and LCSD must be less than 20%

12.2.2.2 If the recovery of TOC in the LCS or LCSD is outside of the 80-120% range, contact the immediate supervisor to determine the cause. If the %RPD of the LCS/LCSD is greater than 20%, contact the immediate supervisor to determine the cause. If the cause of the failure of either the %REC or %RPD cannot be determined and corrected, the batch of samples processed with the LCS/LCSD must be reanalyzed.

12.2.3 The matrix spike and matrix spike duplicate (MS/MSD) is prepared by adding a known concentration of a TOC standard to a known volume or weight of a client sample. The spiked samples are processed through the entire analytical procedure. The accuracy and precision results from the MS/MSD are a measure of the effect that the sample matrix has on the determination of TOC.

12.2.3.1 The recoveries(%REC) of the MS and MSD should be between 60% and 140%. The relative percent difference(%RPD) between the MS and MSD should be less than 40%.

12.2.2.2 If the recovery of TOC in the MS or MSD is outside of the 60-140% range, contact the immediate supervisor to determine the cause. If the %RPD of the LCS/LCSD is greater than 40%, contact the immediate supervisor to determine the cause. If the cause of the failure of either the %REC or %RPD cannot be determined and corrected and the LCS/LCSD are within the specified control limits, the cause can be attributed to matrix interference(s).

NOTE : FOR PROJECTS WHICH REQUIRE FLORIDA DEP QAS CRITERIA, MATRIX SPIKE RESULTS WILL BE UTILIZED FOR LABORATORY CONTROL. IF THE MATRIX SPIKES ARE OUT OF CONTROL, LABORATORY CONTROL STANDARD (LCS) AND METHOD CONTROL CRITERIA WILL BE UTILIZED FOR ULTIMATE CONTROL OF THE ANALYTICAL BATCH.

12.3 The method detection limit (MDL) is defined as the concentration of the an analyte that can be measured with a 99% confidence that the result is greater than zero. The MDL is determined by spiking 10 aliquots of reagent water or 10 aliquots of blank soil (a soil that has been analyzed previously and found to contain no TOC above the RL) with a known concentration of TOC. The spiked samples are taken through the analytical process. The results of the analysis of the spiked samples is used to calculate the MDL.

12.3.1 Prepare the spiked samples for the determination of the Method Detection Limit.

12.3.1.1 Spike ten(10) 100-mL aliquots of reagent water with 0.10mL of the TOC matrix spiking solution (2000mg/L) to determine the MDL for liquid samples. The concentration of TOC in the water sample is 1.0 mg/L.

12.3.1.2 Spike ten(10) aliquots of a blank soil (a soil that has been analyzed previously and found to contain no TOC above the RL) with 0.025mL of the TOC matrix spiking solution (2000mg/L) to determine the MDL for soil/solid samples. The concentration of the TOC in the soil sample is 1000 mg/kg.

12.3.2 Analyze (Section 10.) the MDL samples. Calculate the concentration of the samples (Section 11.). Enter the data on the LIMS sheets. A LIMS-based program will calculate the MDL. A report will also be generated and returned to each section as a record of the MDL

13. REFERENCES

(1) Savannah Laboratories *Comprehensive Quality Assurance Plan*, 1998

(2) *Method for Chemical Analysis of Water and Wastes;* U.S. EPA Office of Research and Development: Cincinnati, OHIO, March, 1983

(3) *Test Methods for the Evaluating Solids Wastes, Third Edition, SW-846;* US EPA Office of Solid Waste and Emergency Response: Washington, DC.

B-11 Total metals by EPA by ICP/AES (Method 6010B)

METHOD 6010B

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion. Samples which are not digested must either use an internal standard or be matrix matched with the standards. Refer to Chapter Three for the appropriate digestion procedures.

1.2 Table 1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the recommended analytical wavelengths and estimated instrumental detection limits for the elements in clean aqueous matrices. The instrument detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements and matrices other than those listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest (see Section 8.0) is demonstrated.

1.3 Users of the method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using the method for analysis.

1.4 Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g. Chapter Three). When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2 This method describes multielemental determinations by ICP-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 a 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 determined 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 should also be recognized and appropriate corrections made; tests for their presence are described in Section 8.5. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

3.0 INTERFERENCES

3.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

3.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

3.1.2 To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

3.1.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement

correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

3.1.4 When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument since the intensities will vary.

3.1.5 Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.

3.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences (Table 2) as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

3.1.7 Users of sequential instruments must verify the absence of spectral interference by scanning over a range of 0.5 nm centered on the wavelength of interest for several samples. The range for lead, for example, would be from 220.6 to 220.1 nm. This procedure must be repeated whenever a new matrix is to be analyzed and when a new calibration curve using different instrumental conditions is to be prepared. Samples that show an elevated background emission across the range may be background corrected by applying a correction factor equal to the emission adjacent to the line or at two points on either side of the line and interpolating between them. An alternate wavelength that does not exhibit a background shift or spectral overlap may also be used.

3.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

3.1.9 When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. If the correction factors or multivariate correction matrices tested on a daily basis are found to be within the 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at ± one reporting limit from zero, daily verification is not required. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

3.1.10 When interelement corrections are <u>not</u> used, verification of absence of interferences is required.

3.1.10.1 One method is to use a computer software routine for comparing the determinative data to limits files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte instrument detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

3.1.10.2 Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is ≥ 20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

3.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate

and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers. The test described in Section 8.5.1 will help determine if a physical interference is present.

3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

3.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

3.5 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

3.6 The dashes in Table 2 indicate that no measurable interferences were observed even at higher interferant concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

4.0 APPARATUS AND MATERIALS

4.1 Inductively coupled argon plasma emission spectrometer:

4.1.1 Computer-controlled emission spectrometer with background correction.

4.1.2 Radio-frequency generator compliant with FCC regulations.

4.1.3 Optional mass flow controller for argon nebulizer gas supply.

4.1.4 Optional peristaltic pump.

4.1.5 Optional Autosampler.

4.1.6 Argon gas supply - high purity.

4.2 Volumetric flasks of suitable precision and accuracy.

4.3 Volumetric pipets of suitable precision and accuracy.

5.0 REAGENTS

5.1 Reagent or trace metals grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration of the contamination is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.

5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an appropriately sized beaker.

5.1.3 Nitric acid (conc), HNO_3.

5.1.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 liter in an appropriately sized beaker.

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

5.3 Standard stock solutions may be purchased or prepared from ultra- high purity grade chemicals or metals (99.99% pure or greater). All salts must be dried for 1 hour at 105°C, unless otherwise specified.

Note: This section does not apply when analyzing samples that have been prepared by Method 3040.

CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling.

Typical stock solution preparation procedures follow. Concentrations are calculated based upon the weight of pure metal added, or with the use of the element fraction and the weight of the metal salt added.

For metals:

$$\text{Concentration (ppm)} = \frac{\text{weight (mg)}}{\text{volume (L)}}$$

For metal salts:

$$\text{Concentration (ppm)} = \frac{\text{weight (mg)} \times \text{mole fraction}}{\text{volume (L)}}$$

5.3.1 Aluminum solution, stock, 1 mL = 1000 µg Al: Dissolve 1.000 g of aluminum metal, weighed accurately to at least four significant figures, in an acid mixture of 4.0 mL of (1:1) HCl and 1.0 mL of concentrated HNO_3 in a beaker. Warm beaker slowly to effect solution. When dissolution is complete, transfer solution quantitatively to a 1-liter flask, add an additional 10.0 mL of (1:1) HCl and dilute to volume with reagent water.

NOTE: Weight of analyte is expressed to four significant figures for consistency with the weights below because rounding to two decimal places can contribute up to 4 % error for some of the compounds.

5.3.2 Antimony solution, stock, 1 mL = 1000 µg Sb: Dissolve 2.6673 g $K(SbO)C_4H_4O_6$ (element fraction Sb = 0.3749), weighed accurately to at least four significant figures, in water, add 10 mL (1:1) HCl, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.3 Arsenic solution, stock, 1 mL = 1000 µg As: Dissolve 1.3203 g of As_2O_3 (element fraction As = 0.7574), weighed accurately to at least four significant figures, in 100 mL of water containing 0.4 g NaOH. Acidify the solution with 2 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.4 Barium solution, stock, 1 mL = 1000 µg Ba: Dissolve 1.5163 g $BaCl_2$ (element fraction Ba = 0.6595), dried at 250°C for 2 hours, weighed accurately to at least four significant figures, in 10 mL water with 1 mL (1:1) HCl. Add 10.0 mL (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.5 Beryllium solution, stock, 1 mL = 1000 µg Be: Do not dry. Dissolve 19.6463 g $BeSO_4{\cdot}4H_2O$ (element fraction Be = 0.0509), weighed accurately to at least four significant figures, in water, add 10.0 mL concentrated HNO_3, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.6 Boron solution, stock, 1 mL = 1000 µg B: Do not dry. Dissolve 5.716 g anhydrous H_3BO_3 (B fraction = 0.1749), weighed accurately to at least four significant figures, in reagent water and dilute in a 1-L volumetric flask with reagent water. Transfer immediately after mixing in a clean polytetrafluoroethylene (PTFE) bottle to minimize any leaching of boron from the glass volumetric container. Use of a non-glass volumetric flask is recommended to avoid boron contamination from glassware.

5.3.7 Cadmium solution, stock, 1 mL = 1000 µg Cd: Dissolve 1.1423 g CdO (element fraction Cd = 0.8754), weighed accurately to at least four significant figures, in a

minimum amount of (1:1) HNO_3. Heat to increase rate of dissolution. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.8 Calcium solution, stock, 1 mL = 1000 µg Ca: Suspend 2.4969 g $CaCO_3$ (element Ca fraction = 0.4005), dried at 180°C for 1 hour before weighing, weighed accurately to at least four significant figures, in water and dissolve cautiously with a minimum amount of (1:1) HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.9 Chromium solution, stock, 1 mL = 1000 µg Cr: Dissolve 1.9231 g CrO_3 (element fraction Cr = 0.5200), weighed accurately to at least four significant figures, in water. When solution is complete, acidify with 10 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.10 Cobalt solution, stock, 1 mL = 1000 µg Co: Dissolve 1.00 g of cobalt metal, weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10.0 mL (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.11 Copper solution, stock, 1 mL = 1000 µg Cu: Dissolve 1.2564 g CuO (element fraction Cu = 0.7989), weighed accurately to at least four significant figures), in a minimum amount of (1:1) HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.12 Iron solution, stock, 1 mL = 1000 µg Fe: Dissolve 1.4298 g Fe_2O_3 (element fraction Fe = 0.6994), weighed accurately to at least four significant figures, in a warm mixture of 20 mL (1:1) HCl and 2 mL of concentrated HNO_3. Cool, add an additional 5.0 mL of concentrated HNO_3, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.13 Lead solution, stock, 1 mL = 1000 µg Pb: Dissolve 1.5985 g $Pb(NO_3)_2$ (element fraction Pb = 0.6256), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10 mL (1:1) HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.14 Lithium solution, stock, 1 mL = 1000 µg Li: Dissolve 5.3248 g lithium carbonate (element fraction Li = 0.1878), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.15 Magnesium solution, stock, 1 mL = 1000 µg Mg: Dissolve 1.6584 g MgO (element fraction Mg = 0.6030), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10.0 mL (1:1) concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.16 Manganese solution, stock, 1 mL = 1000 µg Mn: Dissolve 1.00 g of manganese metal, weighed accurately to at least four significant figures, in acid mixture (10 mL concentrated HCl and 1 mL concentrated HNO_3) and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.17 Mercury solution, stock, 1 mL = 1000 µg Hg: Do not dry, highly toxic element. Dissolve 1.354 g $HgCl_2$ (Hg fraction = 0.7388) in reagent water. Add 50.0 mL concentrated HNO_3 and dilute to volume in 1-L volumetric flask with reagent water.

5.3.18 Molybdenum solution, stock, 1 mL = 1000 µg Mo: Dissolve 1.7325 g $(NH_4)_6Mo_7O_{24}.4H_2O$ (element fraction Mo = 0.5772), weighed accurately to at least four significant figures, in water and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.19 Nickel solution, stock, 1 mL = 1000 µg Ni: Dissolve 1.00 g of nickel metal, weighed accurately to at least four significant figures, in 10.0 mL hot concentrated HNO_3, cool, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.20 Phosphate solution, stock, 1 mL = 1000 µg P: Dissolve 4.3937 g anhydrous KH_2PO_4 (element fraction P = 0.2276), weighed accurately to at least four significant figures, in water. Dilute to volume in a 1,000 mL volumetric flask with water.

5.3.21 Potassium solution, stock, 1 mL = 1000 µg K: Dissolve 1.9069 g KCl (element fraction K = 0.5244) dried at 110°C, weighed accurately to at least four significant figures, in water, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.22 Selenium solution, stock, 1 mL = 1000 µg Se: Do not dry. Dissolve 1.6332 g H_2SeO_3 (element fraction Se = 0.6123), weighed accurately to at least four significant figures, in water and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.23 Silica solution, stock, 1 mL = 1000 µg SiO_2: Do not dry. Dissolve 2.964 g NH_4SiF_6, weighed accurately to at least four significant figures, in 200 mL (1:20) HCl with heating at 85°C to effect dissolution. Let solution cool and dilute to volume in a 1-L volumetric flask with reagent water.

5.3.24 Silver solution, stock, 1 mL = 1000 µg Ag: Dissolve 1.5748 g $AgNO_3$ (element fraction Ag = 0.6350), weighed accurately to at least four significant figures, in water and 10 mL concentrated HNO_3. Dilute to volume in a 1,000 mL volumetric flask with water.

5.3.25 Sodium solution, stock, 1 mL = 1000 µg Na: Dissolve 2.5419 g NaCl (element fraction Na = 0.3934), weighed accurately to at least four significant figures, in water. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.26 Strontium solution, stock, 1 mL = 1000 µg Sr: Dissolve 2.4154 g of strontium nitrate ($Sr(NO_3)_2$) (element fraction Sr = 0.4140), weighed accurately to at least four significant figures, in a 1-liter flask containing 10 mL of concentrated HCl and 700 mL of water. Dilute to volume in a 1,000 mL volumetric flask with water.

5.3.27 Thallium solution, stock, 1 mL = 1000 µg Tl: Dissolve 1.3034 g $TlNO_3$ (element fraction Tl = 0.7672), weighed accurately to at least four significant figures, in water. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.28 Tin solution, stock, 1 mL = 1000 µg Sn: Dissolve 1.000 g Sn shot, weighed accurately to at least 4 significant figures, in 200 mL (1:1) HCl with heating to effect dissolution. Let solution cool and dilute with (1:1) HCl in a 1-L volumetric flask.

5.3.29 Vanadium solution, stock, 1 mL = 1000 µg V: Dissolve 2.2957 g NH_4VO_3 (element fraction V = 0.4356), weighed accurately to at least four significant figures, in a minimum amount of concentrated HNO_3. Heat to increase rate of dissolution. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.30 Zinc solution, stock, 1 mL = 1000 µg Zn: Dissolve 1.2447 g ZnO (element fraction Zn = 0.8034), weighed accurately to at least four significant figures, in a minimum amount of dilute HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.4 Mixed calibration standard solutions - Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks (see Table 3). Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for storage. Fresh mixed standards should be prepared, as needed, with the realization that concentration can change on aging. Some typical calibration standard combinations are listed in Table 3.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.

5.5 Two types of blanks are required for the analysis for samples prepared by any method other than 3040. The calibration blank is used in establishing the analytical curve, and the method blank is used to identify possible contamination resulting from varying amounts of the acids used in the sample processing.

5.5.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial and continuing calibration blank determinations (see Sections 7.3 and 7.4).

5.5.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

5.6 The Initial Calibration Verification (ICV) is prepared by the analyst by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument (see Section 8.6.1 for use).

5.7 The Continuing Calibration Verification (CCV)) should be prepared in the same acid matrix using the same standards used for calibration at a concentration near the mid-point of the calibration curve (see Section 8.6.1 for use).

5.8 The interference check solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest, particularly those with known interferences at 0.5 to 1 mg/L. In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material in Chapter Three, Inorganic Analytes, Sections 3.1 through 3.3.

7.0 PROCEDURE

7.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been prefiltered and acidified will not need acid digestion. Samples which are not digested must either use an internal standard or be matrix matched with the standards. Solubilization and digestion procedures are presented in Sample Preparation Methods (Chapter Three, Inorganic Analytes).

7.2 Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration). Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer.

7.2.1 Before using this procedure to analyze samples, there must be data available documenting initial demonstration of performance. The required data document the selection criteria of background correction points; analytical dynamic ranges, the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences. This data must be generated using the same instrument, operating conditions and calibration routine to be used for sample analysis. These documented data must be kept on file and be available for review by the data user or auditor.

7.2.2 Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. Because of differences among various makes and models of spectrometers, specific instrument operating conditions cannot be provided. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user. The analyst should follow the instructions provided by the instrument manufacturer unless other conditions provide similar or better performance for

a task. Operating conditions for aqueous solutions usually vary from 1100 to 1200 watts forward power, 14 to 18 mm viewing height, 15 to 19 liters/min argon coolant flow, 0.6 to 1.5 L/min argon nebulizer flow, 1 to 1.8 mL/min sample pumping rate with a 1 minute preflush time and measurement time near 1 second per wavelength peak for sequential instruments and 10 seconds per sample for simultaneous instruments. For an axial plasma, the conditions will usually vary from 1100-1500 watts forward power, 15-19 liters/min argon coolant flow, 0.6-1.5 L/min argon nebulizer flow, 1-1.8 mL/min sample pumping rate with a 1 minute preflush time and measurement time near 1 second per wavelength peak for sequential instruments and 10 seconds per sample for simultaneous instruments. Reproduction of the Cu/Mn intensity ratio at 324.754 nm and 257.610 nm respectively, by adjusting the argon aerosol flow has been recommended as a way to achieve repeatable interference correction factors.

7.2.3 The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but only when first setting up a new instrument or following a change in operating conditions. The following procedure is recommended or follow manufacturer's recommendations. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

7.2.3.1 Ignite the radial plasma and select an appropriate incident RF power. Allow the instrument to become thermally stable before beginning, about 30 to 60 minutes of operation. While aspirating a 1000 ug/L solution of yttrium, follow the instrument manufacturer's instructions and adjust the aerosol carrier gas flow rate through the nebulizer so a definitive blue emission region of the plasma extends approximately from 5 to 20 mm above the top of the load coil. Record the nebulizer gas flow rate or pressure setting for future reference. The yttrium solution can also be used for coarse optical alignment of the torch by observing the overlay of the blue light over the entrance slit to the optical system.

7.2.3.2 After establishing the nebulizer gas flow rate, determine the solution uptake rate of the nebulizer in mL/min by aspirating a known volume of calibration blank for a period of at least three minutes. Divide the volume aspirated by the time in minutes and record the uptake rate; set the peristaltic pump to deliver the rate in a steady even flow.

7.2.3.3 Profile the instrument to align it optically as it will be used during analysis. The following procedure can be used for both horizontal and vertical optimization in the radial mode, but is written for vertical. Aspirate a solution containing 10 ug/L of several selected elements. These elements can be As, Se, Tl or Pb as the least sensitive of the elements and most needing to be optimize or others representing analytical judgement (V, Cr, Cu, Li and Mn are also used with success). Collect intensity data at the wavelength peak for each analyte at 1 mm intervals from 14 to 18 mm above the load coil. (This region of the plasma is referred to as the analytical zone.) Repeat the process using the calibration blank. Determine the net signal to blank intensity ratio for each analyte for each viewing height setting. Choose the height for viewing the plasma that provides the best net intensity ratios for the elements analyzed or the highest intensity ratio for the least

sensitive element. For optimization in the axial mode, follow the instrument manufacturer's instructions.

7.2.3.4 The instrument operating condition finally selected as being optimum should provide the lowest reliable instrument detection limits and method detection limits.

7.2.3.5 If either the instrument operating conditions, such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma and viewing height should be re-optimized.

7.2.3.6 After completing the initial optimization of operating conditions, but before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction in particular are discussed in the section on interferences. Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within ± one reporting limit from zero. The upper control limit is the analyte instrument detection limit. Once established the entire routine must be periodically verified every six months. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verification of the routine should be kept on file. Special cases where continual verification is required are described elsewhere.

7.2.3.7 Before daily calibration and after the instrument warmup period, the nebulizer gas flow rate must be reset to the determined optimized flow. If a mass flow controller is being used, it should be set to the recorded optimized flow rate. In order to maintain valid spectral interelement correction routines the nebulizer gas flow rate should be the same (< 2% change) from day to day.

7.2.4 For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements.

7.2.5 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

7.2.5.1 Method detection limits must be established for all wavelengths utilized for each type of matrix commonly analyzed. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit. Refer to Chapter One for additional guidance on the performance of MDL studies.

7.2.5.2 Determination of limits using reagent water represent a best case situation and do not represent possible matrix effects of real world samples.

7.2.5.3 If additional confirmation is desired, reanalyze the seven replicate aliquots on two more non consecutive days and again calculate the method detection limit values for each day. An average of the three values for each analyte may provide for a more appropriate estimate. Successful analysis of samples with added analytes or using method of standard additions can give confidence in the method detection limit values determined in reagent water.

7.2.5.4 The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum for three, preferably five, different concentration standards across the range. One of these should be near the upper limit of the range. The ranges which may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file. The upper range limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Determined analyte concentrations that are above the upper range limit must be diluted and reanalyzed. The analyst should also be aware that if an interelement correction from an analyte above the linear range exists, a second analyte where the interelement correction has been applied may be inaccurately reported. New dynamic ranges should be determined whenever there is a significant change in instrument response. For those analytes that periodically approach the upper limit, the range should be checked every six months. For those analytes that are known interferences, and are present at above the linear range, the analyst should ensure that the interelement correction has not been inaccurately applied.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self absorption effects. These curves may be used if the instrument allows; however the effective range must be checked and the second order curve fit should have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These non-linear response curves should be revalidated and recalculated every six months. These curves are much more sensitive to changes in operating conditions than the linear lines and should be checked whenever there have been moderate equipment changes.

7.2.6 The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

7.3 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 5.4. Flush the system with the calibration blank (Section 5.5.1) between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve must consist of a minimum of a blank and a standard.

7.4 For all analytes and determinations, the laboratory must analyze an ICV (Section 5.6), a calibration blank (Section 5.5.1), and a continuing calibration verification (CCV) (Section 5.7) immediately following daily calibration. A calibration blank and either a calibration verification (CCV) or an ICV must be analyzed after every tenth sample and at the end of the sample run. Analysis of

the check standard and calibration verification must verify that the instrument is within ± 10% of calibration with relative standard deviation < 5% from replicate (minimum of two) integrations. If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV or check standard must be reanalyzed. The analysis data of the calibration blank, check standard, and ICV or CCV must be kept on file with the sample analysis data.

7.5 Rinse the system with the calibration blank solution (Section 5.5.1) before the analysis of each sample. The rinse time will be one minute. Each laboratory may establish a reduction in this rinse time through a suitable demonstration.

7.6 Calculations: If dilutions were performed, the appropriate factors must be applied to sample values. All results should be reported with up to three significant figures.

7.7 The MSA should be used if an interference is suspected or a new matrix is encountered. When the method of standard additions is used, standards are added at one or more levels to portions of a prepared sample. This technique compensates for enhancement or depression of an analyte signal by a matrix. It will not correct for additive interferences, such as contamination, interelement interferences, or baseline shifts. This technique is valid in the linear range when the interference effect is constant over the range, the added analyte responds the same as the endogenous analyte, and the signal is corrected for additive interferences. The simplest version of this technique is the single addition method. This procedure calls for two identical aliquots of the sample solution to be taken. To the first aliquot, a small volume of standard is added; while to the second aliquot, a volume of acid blank is added equal to the standard addition. The sample concentration is calculated by: multiplying the intensity value for the unfortified aliquot by the volume (Liters) and concentration (mg/L or mg/kg) of the standard addition to make the numerator; the difference in intensities for the fortified sample and unfortified sample is multiplied by the volume (Liters) of the sample aliquot for the denominator. The quotient is the sample concentration.

For more than one fortified portion of the prepared sample, linear regression analysis can be applied using a computer or calculator program to obtain the concentration of the sample solution.

NOTE: Refer to Method 7000 for a more detailed discussion of the MSA.

7.8 An alternative to using the method of standard additions is the internal standard technique. Add one or more elements not in the samples and verified not to cause an interelement spectral interference to the samples, standards and blanks; yttrium or scandium are often used. The concentration should be sufficient for optimum precision but not so high as to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation. This technique is very useful in overcoming matrix interferences especially in high solids matrices.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection. All quality control measures described in Chapter One should be followed.

8.2 Dilute and reanalyze samples that exceed the linear calibration range or use an alternate, less sensitive line for which quality control data is already established.

8.3 Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank is a volume of reagent water carried through the same preparation process as a sample (refer to Chapter One).

8.4 Analyze matrix spiked duplicate samples at a frequency of one per matrix batch. A matrix duplicate sample is a sample brought through the entire sample preparation and analytical process in duplicate.

8.4.1.1 The relative percent difference between spiked matrix duplicate determinations is to be calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (replicate).

(A control limit of ± 20% RPD or within the documented historical acceptance limits for each matrix shall be used for sample values greater than ten times the instrument detection limit.)

8.4.1.2 The spiked sample or spiked duplicate sample recovery is to be within ± 25% of the actual value or within the documented historical acceptance limits for each matrix.

8.5 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in Sections 8.5.1 and 8.5.2, will ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

8.5.1 Dilution Test: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an 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.

8.5.2 Post Digestion Spike Addition: 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 and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

8.6 Check the instrument standardization by analyzing appropriate QC samples as follows.

8.6.1 Verify calibration with the Continuing Calibration Verification (CCV) Standard immediately following daily calibration, after every ten samples, and at the end of an analytical run. Check calibration with an ICV following the initial calibration (Section 5.6). At the laboratory's discretion, an ICV may be used in lieu of the continuing calibration verifications. If used in this manner, the ICV should be at a concentration near the mid-point of the calibration curve. Use a calibration blank (Section 5.5.1) immediately following daily calibration, after every 10 samples and at the end of the analytical run.

8.6.1.1 The results of the ICV and CCVs are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.6.1.2 The results of the check standard are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.6.1.3 The results of the calibration blank are to agree within three times the IDL. If not, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

8.6.2 Verify the interelement and background correction factors at the beginning of each analytical run. Do this by analyzing the interference check sample (Section 5.8). Results should be within ± 20% of the true value.

9.0 METHOD PERFORMANCE

9.1 In an EPA round-robin Phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been spiked with various metal concentrates. Table 4 lists the true values, the mean reported values, and the mean percent relative standard deviations.

9.2 Performance data for aqueous solutions and solid samples from a multilaboratory study (9) are provided in Tables 5 and 6.

10.0 REFERENCES

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TABLE 1
RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Detection Element	Wavelength[a] (nm)	Estimated IDL[b] (µg/L)
Aluminum	308.215	30
Antimony	206.833	21
Arsenic	193.696	35
Barium	455.403	0.87
Beryllium	313.042	0.18
Boron	249.678x2	3.8
Cadmium	226.502	2.3
Calcium	317.933	6.7
Chromium	267.716	4.7
Cobalt	228.616	4.7
Copper	324.754	3.6
Iron	259.940	4.1
Lead	220.353	28
Lithium	670.784	2.8
Magnesium	279.079	20
Manganese	257.610	0.93
Mercury	194.227x2	17
Molybdenum	202.030	5.3
Nickel	231.604x2	10
Phosphorus	213.618	51
Potassium	766.491	See note c
Selenium	196.026	50
Silica (SiO_2)	251.611	17
Silver	328.068	4.7
Sodium	588.995	19
Strontium	407.771	0.28
Thallium	190.864	27
Tin	189.980x2	17
Titanium	334.941	5.0
Vanadium	292.402	5.0
Zinc	213.856x2	1.2

[a]The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted (e.g., in the case of an interference) if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see Section 3.1). In time, other elements may be added as more information becomes available and as required.

[b]The estimated instrumental detection limits shown are provided as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[c]Highly dependent on operating conditions and plasma position.

TABLE 2
POTENTIAL INTERFERENCES
ANALYTE CONCENTRATION EQUIVALENTS ARISING FROM
INTERFERENCE AT THE 100-mg/L LEVEL[c]

Analyte	Wavelength (nm)	Interferant[a,b] Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a] Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al -	1000 mg/L	Mg -	1000 mg/L
Ca -	1000 mg/L	Mn -	200 mg/L
Cr -	200 mg/L	Tl -	200 mg/L
Cu -	200 mg/L	V -	200 mg/L
Fe -	1000 mg/L		

[b] The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.

[c] Interferences will be affected by background choice and other interferences may be present.

TABLE 3
MIXED STANDARD SOLUTIONS

Solution	Elements
I	Be, Cd, Mn, Pb, Se and Zn
II	Ba, Co, Cu, Fe, and V
III	As, Mo
IV	Al, Ca, Cr, K, Na, Ni,Li, and Sr
V	Ag (see "NOTE" to Section 5.4), Mg, Sb, and Tl
VI	P

TABLE 4. ICP PRECISION AND ACCURACY DATA[a]

Element	Sample No. 1				Sample No. 2				Sample No. 3			
	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD[b] (%)	Accuracy[d] (%)	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD[b]	Accuracy[d] (%)	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD[b] (%)	Accuracy[d] (%)
Be	750	733	6.2	98	20	20	9.8	100	180	176	5.2	98
Mn	350	345	2.7	99	15	15	6.7	100	100	99	3.3	99
V	750	749	1.8	100	70	69	2.9	99	170	169	1.1	99
As	200	208	7.5	104	22	19	23	86	60	63	17	105
Cr	150	149	3.8	99	10	10	18	100	50	50	3.3	100
Cu	250	235	5.1	94	11	11	40	100	70	67	7.9	96
Fe	600	594	3.0	99	20	19	15	95	180	178	6.0	99
Al	700	696	5.6	99	60	62	33	103	160	161	13	101
Cd	50	48	12	96	2.5	2.9	16	116	14	13	16	93
Co	700	512	10	73	20	20	4.1	100	120	108	21	90
Ni	250	245	5.8	98	30	28	11	93	60	55	14	92
Pb	250	236	16	94	24	30	32	125	80	80	14	100
Zn	200	201	5.6	100	16	19	45	119	80	82	9.4	102
Se[c]	40	32	21.9	80	6	8.5	42	142	10	8.5	8.3	85

[a]Not all elements were analyzed by all laboratories.
[b]RSD = relative standard deviation.
[c]Results for Se are from two laboratories.
[d]Accuracy is expressed as the mean concentration divided by the true concentration times 100.

TABLE 5

ICP-AES PRECISION AND ACCURACY FOR AQUEOUS SOLUTIONS[a]

Element	Mean Conc. (mg/L)	N[b]	RSD[b] (%)	Accuracy[c] (%)
Al	14.8	8	6.3	100
Sb	15.1	8	7.7	102
As	14.7	7	6.4	99
Ba	3.66	7	3.1	99
Be	3.78	8	5.8	102
Cd	3.61	8	7.0	97
Ca	15.0	8	7.4	101
Cr	3.75	8	8.2	101
Co	3.52	8	5.9	95
Cu	3.58	8	5.6	97
Fe	14.8	8	5.9	100
Pb	14.4	7	5.9	97
Mg	14.1	8	6.5	96
Mn	3.70	8	4.3	100
Mo	3.70	8	6.9	100
Ni	3.70	7	5.7	100
K	14.1	8	6.6	95
Se	15.3	8	7.5	104
Ag	3.69	6	9.1	100
Na	14.0	8	4.2	95
Tl	15.1	7	8.5	102
V	3.51	8	6.6	95
Zn	3.57	8	8.3	96

[a]these performance values are independent of sample preparation because the labs analyzed portions of the same solutions

[b]N = Number of measurements for mean and relative standard deviation (RSD).

[c]Accuracy is expressed as a percentage of the nominal value for each analyte in acidified, multi-element solutions.

TABLE 6

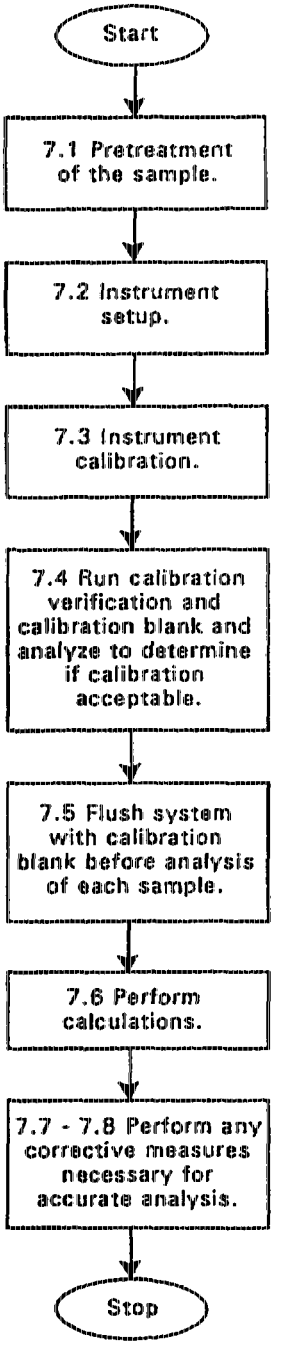
ICP-AES PRECISION AND BIAS FOR SOLID WASTE DIGESTS[a]

Element	Spiked Coal Fly Ash (NIST-SRM 1633a) Mean Conc. (mg/L)	N[b]	RSD[b] (%)	Bias[c] (%AAS)	Spiked Electroplating Sludge Mean Conc. (mg/L)	N[b]	RSD[b] (%)	Bias[c] (%AAS)
Al	330	8	16	104	127	8	13	110
Sb	3.4	6	73	96	5.3	7	24	120
As	21	8	83	270	5.2	7	8.6	87
Ba	133	8	8.7	101	1.6	8	20	58
Be	4.0	8	57	460	0.9	7	9.9	110
Cd	0.97	6	5.7	101	2.9	7	9.9	90
Ca	87	6	5.6	208	954	7	7.0	97
Cr	2.1	7	36	106	154	7	7.8	93
Co	1.2	6	21	94	1.0	7	11	85
Cu	1.9	6	9.7	118	156.	8	7.8	97
Fe	602	8	8.8	102	603	7	5.6	98
Pb	4.6	7	22	94	25	7	5.6	98
Mg	15	8	15	110	35	8	20	84
Mn	1.8	7	14	104	5.9	7	9.6	95
Mo	891	8	19	105	1.4	7	36	110
Ni	1.6	6	8.1	91	9.5	7	9.6	90
K	46	8	4.2	98	51	8	5.8	82
Se	6.4	5	16	73	8.7	7	13	101
Ag	1.4	3	17	140	0.75	7	19	270
Na	20	8	49	130	1380	8	9.8	95
Tl	6.7	4	22	260	5.0	7	20	180
V	1010	5	7.5	100	1.2	6	11	80
Zn	2.2	6	7.6	93	266	7	2.5	101

[a]These performance values are independent of sample preparation because the labs analyzed portions of the same digests.

[b]N = Number of measurements for mean and relative standard deviation (RSD).

[c]Bias for the ICP-AES data is expressed as a percentage of atomic absorption spectroscopy (AA) data for the same digests.


Start
7.1 Pretreatment of the sample.
7.2 Instrument setup.
7.3 Instrument calibration.
7.4 Run calibration verification and calibration blank and analyze to determine if calibration acceptable.
7.5 Flush system with calibration blank before analysis of each sample.
7.6 Perform calculations.
7.7 - 7.8 Perform any corrective measures necessary for accurate analysis.
Stop

B-12 Dioxin / Dibenzofuran

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SOP Release Form

Released to:

Name Joan Pioli 978-322-2863

Company Menzie-Cura and Associates

Location 1 Courthouse Lane
Chelmsford MA 01824

Released by:

Name Dan Zehr

Date 11/27/2000

Mgmt Approval

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SOP ID	Version	SOP ID	Version
DHR182	6		

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290

TLI SOP No.	DHR182 Version: 6	Effective Date:	March 25, 1998
Author:	Wojciech Krol	Date Written:	February 9, 1998
Authorization:	Management	Date Authorized:	2/16/98

I. **SCOPE AND APPLICATION:** This method provides procedures for the analysis of polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologues; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologues; PCDFs) from extracts of samples prepared according to SW 846 Method 8290.

II. **SAFETY CONSIDERATIONS:** The 2,3,7,8-TCDD isomer has been found to be acenegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in the 2,3,7,8 positions are known to have toxicities comparable to that of 2,3,7,8-TCDD. Extreme care must be exercised in all handling of extracts and standards in the application of this SOP. For additional safety information, see the TLI Safety and Health Manual and the appropriate MSDSs.

III. **EQUIPMENT:** The High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (GC/MS/DS) equipment utilized for this analysis includes the following:

A. Gas Chromatographs: Hewlett Packard 5890 or 5890 Series II (equipped for temperature programming and capillary columns)

B. High Resolution Mass Spectrometers: VG 70 Series, VG 70-SE Series, VG Autospec.

C. Data Systems: VG Analytical PDP11 with 11-250 software or VAX Alpha with Opus 3.2 software.

D. GC Injection Port - The GC injection port is designed for capillary columns. Typically, 2 µL injection volumes are used unless otherwise noted.

E. Gas Chromatograph/Mass Spectrometer(GC/MS) Interface - The GC/MS interface components can withstand 350°C. The interface has been designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. The GC column is fitted directly into the mass spectrometer ion source without being

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exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel™, or equivalent, ferrules are recommended.

F. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley).

G. Data System - A dedicated data system is employed to control the rapid selected-ion monitoring (SIM) process and to acquire the data. Quantitation data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantitation may be reported based upon computer generated peak areas or upon measured peak heights. The data system is set to acquire data as low as 10 ions in a single scan. Table 1. presents a listing of the ions which are typically monitored. The data system is set to switch to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system provides hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It also acquires mass spectral peak profiles and provides hard copies of peak profiles to demonstrate the required resolving power. Measurements of noise on the base line are performed using the hard copies of individual ion chromatograms provided by the data system.

NOTE: The detector ADC zero setting must be set to allow peak-to-peak measurement of the noise on the base line of every monitored channel and allow for good estimation of the instrument resolving power.

H. GC Columns

1. In order to have an isomer specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60 m DB-5 fused silica capillary column is used.

2. The 2,3,7,8-TCDF isomer must be confirmed on a 30 m DB-225 fused silica capillary column, when 2,3,7,8-TCDF is detected on the DB-5 column at a level greater than or equal to the target detection limit.

IV. STANDARDS:

A. Calibration Solutions Six nonane solutions containing 17 unlabeled PCDDs and PCDFs and 18 $^{13}C_{12}$ -labeled PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The analyte concentration ranges are homologue dependent, with the lowest values for the tetrachlorinated dioxin and

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furan (0.5 pg/μL) and the highest values for the octachlorinated congeners (2000 pg/μL) (Table 2.).

B. Recovery Standard Solution - This nonane solution contains two recovery standards, $^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD, at a nominal concentration of 100 pg/μL per compound. A 20 μL of this solution is spiked into each sample extract before the HRGC/HRMS analysis.

C. GC Column Performance/Retention Window Check Solution (RTCHK)- This solution contains the first and last eluting isomers for each homologous series from tetra- through heptachlorinated congeners. The solution also contains a series of closely eluting TCDD and TCDF isomers for the purpose of documenting the chromatographic resolution (Table 3).

D. Acceptance Criteria for Newly Prepared Standards - All components and concentrations of each calibration standard, recovery standard, internal standard, and matrix spike solutions are verified prior to use for the analysis of samples. Testing consists of back to back analysis of the "test" solution (the newly prepared solution) and a "control" solution (a known good solution). Control solutions are isolated from the production standards in a protected location. Both the control and test solutions are evaluated versus the current continuing calibration standard and versus each other. Each component of the test solution must be within 80 - 120% of the true concentration when calculated versus the control standard. The control standard must be within 80 - 120% of the true value when calculated versus the continuing calibration standard.

E. Standards are stored in 1/2 dram amber glass vials at room temperature.

V. SYSTEM PERFORMANCE CRITERIA

System performance criteria are presented below. It must be documented that all applicable system performance criteria specified in this Section are met before analysis for any sample is performed. Table 4 provides recommended GC conditions that can be used to satisfy the required criteria. During a typical 12-hour analysis sequence, the GC column performance and mass spectrometer resolving power checks must be performed at the beginning of the 12-hour period of operation. A routine calibration verification is required at the beginning and end of each 12-hour period during which samples are analyzed. A method blank or HRGC/HRMS solvent blank is required between a calibration run and the first sample run.

A. GC Column Performance

1. Inject the column performance check solution (Section IV. C.) and acquire selected-ion monitoring (SIM) data as described in Section III. E., III. F., and Table 4.

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2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers or between 2,3,7,8-TCDF and the peaks representing other TCDF isomers must be resolved with a valley of $\leq$ 25% (Fig. 1.), where:

   . Valley Percent = (x/y) x 100

   x = height of valley measured between 2,3,7,8-TCDD or 2,3,7,8-TCDF and the closest TCDD or TCDF eluting isomers, and

   y = the peak height of 2,3,7,8-TCDD or 2,3,7,8-TCDF.

3. The acquisition time windows must be set to allow observation of the first and last eluting isomer of each congener. All first and last eluters of a homologous series should be labeled and identified on chromatograms.

B. Mass Spectrometer Performance

1. The mass spectrometer must be operated in the electron ionization mode. It is recommended that the ionization potential be set to optimize sensitivity for the given column flow and source design. A static resolving power of at least 10,000 (10% valley definition) must be demonstrated at appropriate masses before any analyses are performed. Static resolving power checks must be performed at the beginning and at the end of each 12-hour period of operation. It is recommended, however, that a check of the static resolution be made and documented by using the peak matching unit before and after each analysis. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, use a lock-mass ion from the reference compound Perfluorokerosene (PFK) used for tuning the mass spectrometer and monitor and record the lock-mass ion channel during SIM acquisitions. The level of the reference compound (PFK) metered inside the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the selected lock-mass ion signal, regardless of the description number, does not exceed 10% of the full scale deflection for a given set of detector parameters. Under those

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conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

3. Using PFK molecular leak and an appropriate ion within the scan window, tune the instrument to meet the minimum required resolving power of 10,000 (10% valley).

   a) Documentation of the mass spectrometer resolving power is accomplished by recording the peak profile of the high-mass reference signal (m/z 416.9760) obtained during a peak matching experiment by using the low-mass PFK ion at m/z 330.9792 (or lower in mass) as a reference.

   b) The format of the peak profile representative allows manual determination of the peak resolution. The peak width (at 5% peak height) of the high-mass reference ion must not exceed 100 ppm (resolving power: 10,000). Peak width is determined by triangulation with no more than 10% allowance for sampling error. Instrumental ion transmission and resolution will be checked, adjusted and documented in case the resolution is below the minimum required 10,000 resolving power.

VI. CALIBRATION PROCEDURES:

A. Initial Calibration (ICAL)- Initial calibration of the instrument is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any continuing calibration does not meet the required criteria listed in Section VI. B..

1. All six calibration solutions listed in Table 2. must be used for the initial calibration.

2. Tune the instrument with PFK to achieve a static resolving power of at least 10,000 (10% valley) as described in section V. B..

3. Inject 2 µL of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section III. F. - G. Any further analysis must not be performed until it has been documented that the column performance criteria listed in section V. A. was met.

4. Using the same GC and MS conditions that produced acceptable results for the column performance check solution, analyze a 2 µL portion of each of the six calibration solutions with the following requirements.

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a) The ratio of integrated ion current for the ions appearing in Table 5 (homologous series quantitation ions) must be within the indicated control limits (set for each homologous series).

b) The ratio of integrated ion current for the ions belonging to the carbon-labeled internal, surrogate, alternate, and recovery standards must be within the control limits stipulated in Table 5.

NOTE: Ion ratios for all 17 native analytes and 18 carbon-labeled internal and recovery standards must be within the specified control limits simultaneously in one run for each of the six (6) calibration standard solutions. If the ion abundance ratios are outside the limits, corrective action must be taken and acceptable abundance ratios achieved before any samples may be analyzed.

c) For each SICP and for each GC signal representing the elution of a target analyte the signal-to-noise ratio (S/N) must be better than or equal to 10:1.

d) Referring to Table 6., calculate the 17 relative response factors (RRF) for unlabeled target analytes relative to their appropriate internal standards, according to the following formula:

$$RRF(n) = \frac{A_x \times Q_{is}}{Q_x \times A_{is}}$$

Where:

RRF(n)	=	Analyte RRF
A_x	=	sum of the integrated ion abundances of the quantitation ions (Table 1.) for unlabeled PCDDs/PCDFs,
A_{is}	=	sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled internal standards,
Q_{is}	=	quantity of the internal standard injected (pg),
Q_x	=	quantity of the unlabeled PCDD/PCDF analyte injected (pg).

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The RRF(n) is a dimensionless quantity; the units used to express Q_{is} and Q_x must be the same.

e) Calculate the average analyte $\overline{RRF}$ and their respective percent relative standard deviations (%RSD) for the six calibration solutions.

$$\overline{RRF}_{(n)} = \frac{1}{6}\sum_{j=1}^{6} RRF_{(n)j}$$

Where n represents a particular PCDD/PCDF (2,3,7,8 substituted congener), and j is the injection number (or calibration solution number).

f) The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

(1) For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section V. A. 4. e).

NOTE: The calibration solutions do not contain $^{13}C_{12}$-OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the $[M+6]^+$ ion of $^{13}C_{12}$-OCDF from the $[M+2]^+$ ion of OCDD (and $[M+4]^+$ from $^{13}C_{12}$-OCDF with $[M]^+$ of OCDD). Therefore, the RRF for OCDF is calculated relative to $^{13}C_{12}$-OCDD.

(2) For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (PeCDF - two, HxCDF - four, HxCDD - three, HpCDF - two) the mean RRF used for those homologous series will be the average of the mean RRFs calculated for all individual 2,3,7,8-substituted congeners.

NOTE: HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution pattern are assumed to be the same as the

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responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

g) Referring to Table 7. and 8. calculate relative response factors (RRF) and average relative response factors (RRF) for internal, surrogate, and alternate standards relative to their appropriate recovery standards, according to the following formula:

$$RRF_{(m)} = \frac{A_s \times Q_{rs}}{Q_s \times A_{rs}}$$

$$\overline{RRF}_{(m)} = \frac{1}{6}\sum_{j=1}^{6} RRF_{(m)j}$$

Where:

m	=	congener type,
j	=	injection number,
A_s	=	sum of the integrated ion abundances of the quantitation ions for a given standard,
A_{rs}	=	sum of the integrated ion abundances of the quantitation ions for the appropriate recovery standard,
Q_{rs}, Q_s	=	quantities of, respectively, the recovery standard (rs) and a particular standard injected (pg),
$RRF_{(m)}$	=	relative response factor of a particular standard relative to an appropriate recovery standard, as determined from one injection, and
$\overline{RRF}_{(m)}$	=	calculated mean relative response factor of a particular labeled standard relative to an appropriate recovery standard, as determined from the six initial calibration injections.

5. Acceptance Criteria for Initial Calibration - The criteria listed below for acceptable calibration must be met before any analysis is performed.

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a) The percent relative standard deviations for the mean response factors from the 17 unlabeled standards must not exceed ± 20 percent, and those for the labeled reference compounds must not exceed ± 30%.

b) The S/N ratio for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10.

c) The isotopic ratios (Table 5.) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section VI. A. 5. a) is met, the analyte specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the continuing calibration criteria (Section VI. B.) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

B. Continuing Calibration Check (CONCAL) - Continuing calibrations must be performed at the beginning of a 12 hour period after successful mass resolution and QC resolution performance checks. A continuing calibration is also required at the end of a 12 hour shift.

1. Inject 2 µL of the CONCAL solution HRCC-3 standard (Table 2). By using the same HRGC/HRMS conditions as described in Section V., and used to determine and document an acceptable calibration as provided in Section VI. A. 5..

2. Acceptance Criteria for Continuing Calibration - The following criteria must be met before further analysis is performed.

a) The measured RRFs for the unlabeled standards obtained during the continuing calibration runs must be within ± 20 percent of the mean values established during the initial calibration (Section VI. A. 4. e) and f)).

b) The measured RRFs for the labeled standards obtained during the routine calibration must be within ± 30 percent of the mean values established during the initial calibration (Section VI. A. 4. g)).

c) The ion-abundance ratios (Table 5.) must be within the allowed control limits.

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d) If either one of the criteria in Sections VI. B. 2. a) and b) is not satisfied, repeat one more time. If these criteria are still not satisfied, the entire continuing calibration process must be reviewed.

e) If the continuing calibration at the end of a 12-hour period fails no more than 25% RPD for the unlabeled compounds and 35% RPD for the labeled reference compounds, use the mean RRFs from the two daily calibration runs to compute analyte concentrations, instead of the RRFs obtained from the initial calibration.

NOTE: If RRFs for up to two labeled standards fail by more than 35%, the calibration is considered acceptable, as long as corresponding unlabeled analytes meet criteria.

f) Continuing calibrations analyzed at the end of the 12 hour period which fail by more than 25% RPD for the unlabeled compounds and 35% RPD for the labeled reference compounds must be documented according to the non-conformance SOP. The analyst must assess the effect of failure on overall data quality. The assessment will include evaluation of any spiked samples included in the 12 hour "clock", presence of reportable levels of the affected analytes in the samples, and sample data obtained during the period between beginning and ending calibrations. All samples which contain reportable levels of a "failed" analyte will be reanalyzed unless a passing spiked sample has been analyzed between the sample and the ending calibration standard.

VII. ANALYSIS

A. A valid analysis of column performance check, calibration and method or instrument blank must have been obtained prior to the analysis of any sample.

B. Remove the sample extract (blown to dryness) or blank from storage. Add 20 µL of recovery standard and mix thoroughly. Care must be taken to coat the walls of the vial several times to dissolve the sample material deposited on the walls during the concentration process.

NOTE: A final volume of 20 µL or more should be used whenever possible. A 10 µL final volume is difficult to handle, and injection of 2 µL out of 10 µL leaves little sample for confirmations and repeat injections, and for archiving.

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C. Inject a 2 µL aliquot of the extract into the GC, operated under the GC conditions that have produced acceptable CONCAL and RTCHK results.

D. Acquire SIM data using the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors.

NOTE: The acquisition period must at least encompass the PCDD/PCDF overall retention time window previously determined. Selected ion current profiles (SICP) for the lock-mass ions (one per mass descriptor) must also be recorded and included in the data package. These SICPs must be true representations of the evolution of the lock-mass ions amplitudes during the HRGC/HRMS run. The analyst may be required to monitor a PFK ion, not as a lock mass, but as a QC ion, in order to meet this requirement. It is recommended to examine the QC ion or lock-mass ion SICP for obvious basic sensitivity and stability changes of the instrument during the GC/MS run that could affect the measurements. Report any discrepancies in the case narrative.

E. Identification Criteria - For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

1. Retention Times

a) For 2,3,7,8-substituted congeners, which have an isotopically labeled internal or recovery standard present in the sample extract (this represents a total of 10 congeners including OCDD; Table 1.), the retention time (RRT; at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 1.), must be within -1 to +3 seconds of the isotopically labeled standard.

b) For 2,3,7,8-substituted compounds that do not have an isotopically labeled internal standard present in the sample extract (this represents a total of eight congeners; Table 1.), the retention time must fall within 0.005 retention time units of the relative retention times measured in the continuing calibration. Identification of OCDF is based on its retention time relative to $^{13}C_{12}$-OCDD as determined from the 12 hour continuing calibration results.

c) For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the RTCHK solution.

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d) The ion current responses for both ions used in quantitation (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach maximum simultaneously (± 2 seconds).

e) The ion current responses for both ions used for the labeled standards (e.g., for $^{13}C_{12}$-TCDD: m/z 331.9368 and m/z 333.9339) must reach maximum simultaneously (± 2 seconds).

2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned (Table 5.).

3. Signal-to-Noise (S/N) Ratio

All ion current intensities must be ≥ 2.5 times noise level for positive identification of a PCDD/PCDF compound or a group of coeluting isomers.

4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N ≥ 2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 1.) channel or if the PCDPE signal is less than 10% of the PCDF signal.

F. Quantitation

1. For gas chromatographic peaks that have met specified criteria calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times W \times \overline{RRF}_{(n)}}$$

Where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) in pg/g,

A_x = sum of the integrated ion abundances of the quantitation ions (Table 1.) for unlabeled PCDDs/PCDFs,

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A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled internal standards,

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

W = weight or volume, in grams or liters, of the sample (solid or liquid), and

$\overline{RRF}_{(n)}$ = calculated mean relative response factor for the analyte.

2. Calculate the percent recovery of the nine internal standards measured in the sample extract, using the formula:

$$\text{Internal Standard Recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times \overline{RRF}_{(m)}} \times 100$$

Where:

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled internal standard,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (Table 7.),

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

Q_{rs} = quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$\overline{RRF}_{(m)}$ = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 7) recovery standard.

3. Calculate the percent recovery of seven surrogate and alternate standards using expression VII. F. 2..

NOTE: For the air samples (PUF, M-0023A) calculate percent recovery of five surrogate standards relative to their appropriate internal standards.

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4. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compound exceeds the dynamic range of the instrument (i.e., saturation in a mass channel) then solvent will be added to the extract to bring the signal level into the instrument's dynamic range. Any dilutions must be pre-approved by the client.

5. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value must be specified in the report.

6. Sample Specific Estimated Detection Limit - The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, depending on the type of response produced during the analysis of a particular sample.

   a) Samples giving a response for at least one quantitation ion that is less than 2.5 times the background level.

      The area of the analyte is replaced by the noise level measured in a region of the chromatogram clear of genuine GC signals multiplied by an empirically determined factor. The detection limits represent the maximum possible concentration of a target analyte that could be present without being detected.

$$DL = \frac{2 \times 2.5 \times (F \times H) \times \underline{Q}_{is}}{A_{is} \times \overline{RRF}_{(n)} \times W}$$

Where:

DL	=	estimated detection limit for a target analyte, expressed in ng or pg.
2.5	=	minimum signal/ noise required for a GC signal to be accepted.
F	=	an empirical number that approximates the area to height ratio for a GC signal. (F= 3.7 for all dioxin/ furan analyses.)

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H	=	height of the noise.
A_{is}	=	integrated current of the characteristic ions of the corresponding internal standard.
Q_{is}	=	amount of internal standard added to the sample before extraction.
$\overline{RRF_{(n)}}$	=	mean analyte relative response factor from the initial calibration.
W	=	sample weight or volume.
2	=	used to account for the two signals present for every analyte.

b) Samples characterized by a response above the background level with a S/N of at least 2.5 for both quantitation ions.

When the response of a signal having the same retention time as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet ion ratio requirements (Table 5.) calculate the "Estimated Maximum Possible Concentration" (EMPC) according to the expression shown in Section VII. F. 1., except that A_x should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio.

VIII. QUALITY CONTROL REQUIREMENTS

A. GC column performance must be demonstrated initially and verified prior to analyzing any sample in a 12-hour period. The GC column performance check solution must be analyzed under the same chromatographic and mass spectrometric conditions used for other samples and standards.

B. Routine calibrations must be performed at the beginning of a 12-hour period after successful mass resolution and GC resolution performance checks and at the end of a 12-hour period following the analysis of samples.

C. The target detection limits will be the following:

1. TDLs for waters (sample size - 1L):

≤ 10 ppq	TCDD & TCDF
≤ 50 ppq	Penta - HeptaCDD & CDF
≤ 100 ppq	OCDD & OCDF

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2. TDLs for solids (sample size - 10g):

   ≤ 1 ppt TCDD & TCDF

   ≤ 5 ppt Penta - HeptaCDD & CDF

   ≤ 10 ppt OCDD & OCDF

3. For air (e.g., PUF, M-0023A) or Wipes (sample size - 1):

   ≤ 50 pg TCDD & TCDF

   ≤ 250 pg Penta - HeptaCDD & CDF

   ≤ 500 pg OCDD & OCDF

D. Method Blank

1. Percent recoveries of all labeled standards should be between 40 - 135%. Method blank with standard recoveries below 40% (but ≥ 25%) is acceptable as long as signal to noise for affected standards is ≥ 10:1.

2. Target detection limits must be met for each analyte. (VIII. C.)

3. No reportable analytes should be present in the Method Blank. Up to 3 analytes may be present at levels below 1/2 Target Detection Limit (TDL) as long as the compounds were not reported in the previous analysis.

4. For Method Blanks associated with high level samples, the analyte level in the blank must be < 5% of the quantity present in the samples.

E. LCS/ LCSD and MS/ MSD

1. Percent recoveries of all labeled standards should be between 40 - 135%. LCS/ LCSD or MS/ MSD with labeled standard recoveries below 40% (but ≥ 25%) are acceptable as long as signal to noise for affected standards is ≥ 10:1.

2. Percent recoveries of all analytes should be between 70 - 130%. For up to two analytes, recoveries may be as high as 145% or as low as 60%, as long as the associated relative percent differences (%RPDs) meet criteria.

3. Relative percent difference between LCS and LCSD or MS and MSD should be ≤ 20% for all analytes. For up to two analytes, %RPDs may be higher (up to 35%), as long as the associated percent recoveries meet criteria.

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F. Samples

1. Percent recoveries of all labeled standards should be between 40 - 135%. Samples with standard recoveries below 40% (but ≥ 25%) are acceptable as long as signal to noise for affected standards is ≥ 10:1. In the case of OCDD internal standards, the OCDF analyte must be below TDL also.

2. Specific detection limits must not exceed the target detection limits unless prohibited by a limited sample size or the need for dilution.

3. If the method blank contains reportable analytes but those analytes are not detected in the sample, the sample data may be reported.

4. Samples which fail acceptance criteria listed above (VIII. F. 1. and 2.) or associated with failing method blank or LCS/ LCSD (VIII. D. 1. - 4.; VIII. E. 1. - 3.) must be reextracted and reanalyzed. Exceptions may be made for sample matrices which require extreme additional cleanup procedures.

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TABLE 1: ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HRGC/HRMS FOR PCDDs AND PCDFs

Descriptor Number[b]	Accurate Mass[a]	Ion Type	Elemental Composition	Analyte
2	292.9825	LOCK	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF (S)
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF (S)
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD (S)
	330.9792	QC	C_7F_{13}	PFK
	331.9368	M	$13C_{12}H_4{}^{35}Cl_4O_2$	TCDD (S)
	333.9339	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD (S)
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF (S)
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF (S)
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD (S)
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD (S)
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF (S)

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TABLE 1 CONTINUED

Descriptor Number[b]	Accurate Mass[a]	Ion Type	Elemental Composition	Analyte
3 Continued	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF (S)
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	392.9760	LOCK	C_9F_{15}	PFK
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD (S)
	403.8529	M+4	$13C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD (S)
	445.7555	M+4	$C_{12}H_2{}^{25}Cl_6{}^{37}Cl_2O$	OCDPE
	430.9729	QC	C_9F_{13}	PFK
4	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	417.8253	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF (S)
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF (S)
	423.7766	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD (S)
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD (S)
	479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCDPE
	430.9729	LOCK	C_9F_{17}	PFK
	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7779	M+2	$^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD (S)
	471.7750	M+4	$^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD (S)

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TABLE 1 CONTINUED

Descriptor Number[b]	Accurate Mass[a]	Ion Type	Elemental Composition	Analyte
4 Continued	513.6775	M+4	$C_{12}{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE
	442.9728	QC	$C_{10}F_{17}$	PFK

a) The following nuclidic masses were used:

H = 1.007825	O = 15.994915
C = 12.000000	^{35}Cl = 34.968853
^{13}C = 13.003355	^{37}Cl = 36.965903
F = 18.9984	

S = Labeled Standard
QC = Ion Selected for Monitoring the Instrument Stability During the GC/MS Analysis

b) Descriptor 1 contains mono-, di- and trichlorinated dibenzodioxins and dibenzofurans that are not quantitated by Method 8290.

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TABLE 2: COMPOSITION OF THE INITIAL CALIBRATION SOLUTIONS

Compound	Concentrations (pg/µL) Sol. Number: 1	2	3	4	5	6
Unlabeled Analytes						
2,3,7,8-TCDD	0.5	1	10	50	100	200
2,3,7,8-TCDF	0.5	1	10	50	100	200
1,2,3,7,8-PeCDD	2.5	5	50	250	500	1000
1,2,3,7,8-PeCDF	2.5	5	50	250	500	1000
2,3,4,7,8-PeCDF	2.5	5	50	250	500	1000
1,2,3,4,7,8-HxCDD	2.5	5	50	250	500	1000
1,2,3,6,7,8-HxCDD	2.5	5	50	250	500	1000
1,2,3,7,8,9-HxCDD	2.5	5	50	250	500	1000
1,2,3,4,7,8-HxCDF	2.5	5	50	250	500	1000
1,2,3,6,7,8-HxCDF	2.5	5	50	250	500	1000
1,2,3,7,8,9-HxCDF	2.5	5	50	250	500	1000
2,3,4,6,7,8-HxCDF	2.5	5	50	250	500	1000
1,2,3,4,6,7,8-HpCDD	2.5	5	50	250	500	1000
1,2,3,4,6,7,8-HpCDF	2.5	5	50	250	500	1000
1,2,3,4,7,8,9-HpCDF	2.5	5	50	250	500	1000
OCDD	5	10	100	500	1000	2000
OCDF	5	10	100	500	1000	2000
Internal Standards						
$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100	100

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TABLE 2 CONTINUED

Compound	Concentrations (pg/µL) Sol. Number: 1	2	3	4	5	6
Internal Standards Continued						
$^{13}C_{12}$-OCDD	200	200	200	200	200	200
$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100	100
Surrogate Standards						
$^{37}Cl_4$-2,3,7,8-TCDD	0.5	1	10	50	100	200
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	100	100	100	100	100
Alternate Standard						
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100	100
Recovery Standards						
$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100	100

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TABLE 3: GC RETENTION TIME WINDOW DEFINING SOLUTION AND ISOMER SPECIFICITY TEST STANDARD (SECTION IV. C.)

DB-5 COLUMN GC RETENTION TIME WINDOW DEFINING SOLUTION

CDD/ CDF	FIRST ELUTED	LAST ELUTED
TCDF	1,3,6,8	1,2,8,9
TCDD	1,3,6,8	1,2,8,9
PeCDF	1,3,4,6,8	1,2,3,8,9
PeCDD	1,2,4,7,9	1,2,3,8,9
HxCDF	1,2,3,4,6,8	1,2,3,4,8,9
HxCDD	1,2,4,6,7,9	1,2,3,4,6,7
HpCDF	1,2,3,4,6,7,8	1,2,3,4,7,8,9
HpCDD	1,2,3,4,6,7,9	1,2,3,4,6,7,8

DB-5 COLUMN TCDD SPECIFICITY TEST STANDARD

1,2,3,7 + 1,2,3,8 - TCDD
2,3,7,8 - TCDD
1,2,3,9 - TCDD

DB-225 COLUMN TCDF ISOMER SPECIFICTY TEST STANDARD

2,3,4,7 - TCDF
2,3,7,8 - TCDF
1,2,3,9 - TCDF

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TABLE 4: GAS CHROMATOGRAPHY CONDITIONS

Column type	DB-5	DB-225
Length (m)	60	30
i.d. (mm)	0.25	0.25
Film Thickness (um)	0.25	0.25
Carrier Gas	Helium	Helium
Carrier Gas Flow (mL/min)	1-2	1-2
Injection Mode	⇐ splitless ⇒	
Valve Time (s)	60	60
Initial Temperature (°C)	150	130
Injection Port Temperature (°C)	250	250
Program Temperature	⇐ See Note ⇒	

Note: The GC temperature program is subject to change. Refer to work area guidelines for exact definitions of the current run conditions and descriptor name.

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TABLE 5. ION-ABUNDANCE RATIO ACCEPTABLE RANGES FOR PCDDs AND PCDFs

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6a	M/M+2	0.51	0.43	0.59
7b	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

a) Used only for ^{13}C-HxCDF
b) Used only for ^{13}C-HpCDF

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TABLE 6. UNLABELED ANALYTES QUANTITATION RELATIONSHIPS

Analyte	Standard Used During Quantitation
2,3,7,8-TCDD	$^{13}C_{12}$-2,3,7,8-TCDD
Other TCDDs	$^{13}C_{12}$-2,3,7,8-TCDD
1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,7,8-PeCDD
Other PeCDDs	$^{13}C_{12}$-1,2,3,7,8-PeCDD
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
Other HxCDDs	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD
Other HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD
OCDD	$^{13}C_{12}$-OCDD
2,3,7,8-TCDF	$^{13}C_{12}$-2,3,7,8-TCDF
Other TCDFs	$^{13}C_{12}$-2,3,7,8-TCDF
1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF
Other PeCDFs	$^{13}C_{12}$-1,2,3,7,8-PeCDF
1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF

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TABLE 6 CONTINUED

Analyte	Standard Used During Quantitation
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
Other HxCDFs	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
Other HpCDFs	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
OCDF	$^{13}C_{12}$-OCDD

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TABLE 7: INTERNAL STANDARDS QUANTITATION RELATIONSHIPS

Internal Standard	Standard Used During Percent Recovery Determination
$^{13}C_{12}$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-OCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-2,3,7,8-TCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD

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TABLE 8: SURROGATE/ALTERNATE STANDARDS QUANTITATION RELATIONSHIPS

Surrogate/Alternate/Cleanup Standard	Standard Used During Percent Recovery Determination
$^{37}Cl_4$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	13C_{12}-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD

NOTE : For air samples (PUF and M-0023A) recoveries of surrogate standards are quantitated relative to their appropriate internal standards.

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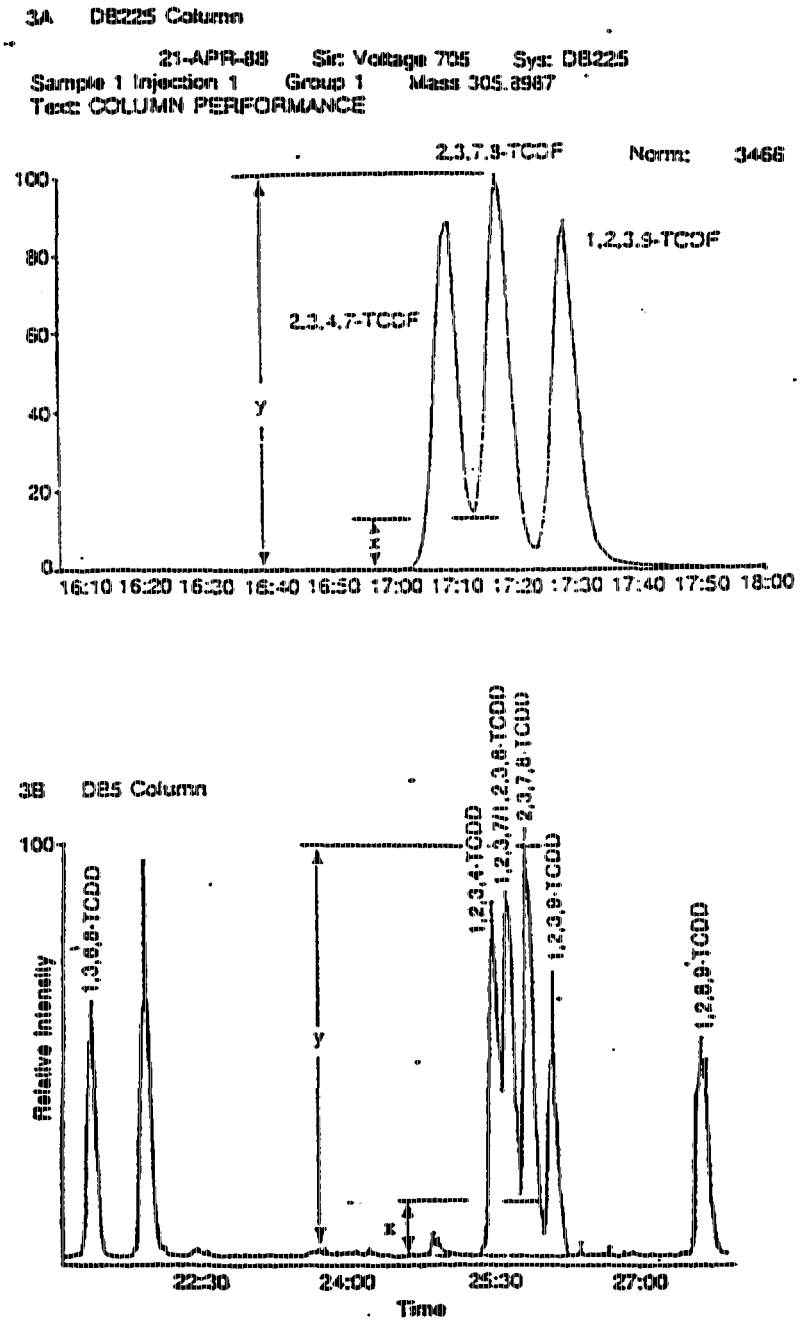

FIGURE 1. Valley between 2,3,7,8 - Tetra Dioxin and Furan Isomers and Other Closely Eluted Isomers.

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APPENDIX 1

Deviations from and improvements to US EPA Method 8290
POLYCHLORINATED DIBENZODIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs) BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/ HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/ HRMS)
as performed at Triangle Laboratories, Inc.

See Section	
• IV. A. and Table 2.	Triangle Laboratories uses 6 calibration points ranging from 0.5 pg/µL to 200 pg/µL for tetrachlorinated dioxin and furan. Concentrations of penta- through heptachlorinated analytes are 5 times higher than tetra congeners and octachlorinated analytes are 10 times higher (Compare with Table 5., Method 8290).
• IV. A.	Seven more labeled PCDDs/ PCDFs are used as surrogate and alternate standards to provide additional quality control information.
• IV. A.	The 1,2,3,6,7,8 - hexachlorodibenzofuran (HxCDF) is used as an internal standard instead of 1,2.3,4,7,8 - HxCDF. Triangle Laboratories uses 1,2,3,4,7,8 - HxCDF as a surrogate standard.
• IV. A.	Each carbon-labeled standard in the initial calibration solution has a concentration of 100 pg/µL except $^{13}C_{12}$ - OCDD, which is at 200 pg/µL. Concentrations of $^{13}C_{12}$ - labeled standards in sample fortification solutions are the same as in the initial calibration solution (See section 5.9., Method 8290).
• V.	The method blank does not need to be analyzed on each analysis clock that samples are analyzed. Once a valid analysis is provided for the method blank it may be replaced with HRGC/ HRMS solvent blank (See section 8.2., Method 8290).
• V. B. 3. a)	Documentation of the mass spectrometer resolving power is accomplished by recording the peak profile of m/z 416.9760 and 330.9792 (See section 8.2.2.3., Method 8290).
• VII. F. 6. a)	To calculate the sample specific Estimated Detection Limit, noise equivalent area is used instead of noise height (See section 7.9.5.1.1., Method 8290).
• VIII. D. - F. 1.	The Internal Standard recoveries in all QC samples and field samples are considered valid as long as the signal to noise ratio is greater than 10:1, the recovery is $\geq$ 25% and target detection limits for the analytes are met (See section 8.4., Method 8290).
• VIII. E. 2.	For up to two analytes in LCS/ LCSD and MS/ MSD, recoveries may be as high as 145% or as low as 60%, as long as the associated relative percent differences (%RPDs) meet criteria.

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APPENDIX 1 Continued

- VIII. E. 3. For up to two analytes in LCS/ LCSD and MS/ MSD, %RPDs may be higher (up to 35%), as long as the associated percent recoveries meet criteria.

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Appendix C

AMEC Bioassay Laboratory

Standard Operating Procedures

C-1 48-Hr Static Acute Test Using Daphnids

C-2 7-Day Static Renewal Test Using *Ceriodaphnia dubia*

C-3 48-Hr or 96-Hr Static Renewal Test Using *Pimephales promelas*

C-4 7-Day Static Renewal Tests Using *Pimephales promelas*

C-5 10-Day Static Freshwater Sediment Toxicity Tests with the Amphipod *Hyallela azteca*

C-6 10-Day Static Freshwater Sediment Toxicity Testa with the Midge Larvae *Chironomus tentans*

C-7 28-Day Bioaccumulation Analyses with the Asiatic Clam *Corbicula fulminea*

C-1 48-Hr Static Acute Test Using Daphnids

I. PURPOSE

This method measures the acute toxicity of whole effluents to the cladoceran *Ceriodaphnia dubia* or *Daphnia pulex* in a 48-hour static test. Test results are based on survival.

II. EQUIPMENT/SUPPLIES

- Environmental chamber maintained at 25 ± 1°C and cold room at 4°C
- Thermometer, pH meter, dissolved oxygen meter, conductivity meter
- Test chambers: four 30-mL disposable polystyrene cups per concentration and control
- Numbered "cerio boards" for holding test chambers
- Dissection microscope with substage lighting
- Clear covers to prevent contamination of test chambers
- 400-mL beakers
- 100-mL graduated cylinders
- 1 and 10-mL disposable pipettes
- Daphnid neonates, <24 hours old
- Yeast/Trout chow/Chlorophyll (YTC) and *Selenastrum* suspension to feed animals
- Lake Arrowhead and Evian Water or 20 percent dilute mineral water (8:2)
- $CuCl_2$ for reference toxicant testing
- Safety equipment - lab coats, eye protection, gloves and respirator as required

III. PROCEDURE

A. *Pre-test Set up*

1. Test animals are purchased from a reputable dealer and cultured in the laboratory.

2. Twenty four hours prior to test initiation, gravid females are isolated in 30-mL polystyrene cups containing laboratory control water. Isolated females are fed 200 µl each of *Selenastrum* and YCT.

3. On the day of the test initiation, neonates (<24 hrs. old) produced by the isolated females are collected and placed in a crystallizing dish containing control water and food. Only neonates originating from broods of 8 or more are used in tests.

4. Samples are checked in upon receipt. See procedural SOP for details on routine sample check-in procedures. Samples are immediately placed in a refrigerator at 4°C until the test is initiated. All test must be initiated within 36 hours of sample collection. Check with laboratory surpervisor for project-specific differences.

B. Test Initiation

1. Record sample description with matrix, color, odor, and particulate information on the data sheet.

2. Prepare dilutions according to test specific concentrations. Concentrations are directed by permit language. Bioassay which require a 0.5 dilution are serially diluted. All other concentrations must be measured with graduated cylinders and pipettes. Warm to 25 ± 1°C.

3. Measure physical and chemical measurements (pH, dissolved oxygen, temperature and conductivity) and record on the data sheet.

4. Distribute dilutions to test chambers.

5. Using a 1-mL disposable transfer pipet with the tip cut off to produce a wide bore, remove neonates from the crystallizing dish individually and randomly distribute 5 to each test chamber.

C. Daily Monitoring

1. Measure and record the temperature.

2. Count and record the number of surviving organisms. Remove dead organisms from test cahmbers.

D. Test Termination

1. Test is terminated 48 ± 2 hours after initiation.

2. Count and record the number of surviving organisms.

3. Composite each concentration, measure and record physical and chemical parameters of each concentration.

IV. TEST ACCEPTABILITY CRITERIA

Control survival at test termination must be 90 percent or greater for test results to be considered acceptable.

V. HEALTH AND SAFETY

Health and safety precautions and applicable regulations should be considered at all times. Gloves should always be worn when handling effluents.

VI. PERSONNEL

Only qualified technicians who have been properly trained and can demonstrate competency with these techniques are approved to conduct this test.

VII. QUALITY ASSURANCE REQUIREMENTS

Quality assurance practices encompass all aspects of testing including the collection, handling, and preparation of test organisms, samples, and dilution waters. Proper record keeping is required, and concentrated efforts are made for complete documentation on a real-time basis.

VIII. REFERENCE

"Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms." EPA/600/4-90/027F, August 1993.

C-2 7-Day Static Renewal Test Using *Ceriodaphnia dubia*

7 - DAY STATIC RENEWAL TEST USING *CERIODAPHNIA DUBIA*

I. PURPOSE

This method estimates the chronic toxicity of whole effluents and receiving water to the cladoceran *Ceriodaphnia dubia*. Test results are based on survival and reproduction.

II. EQUIPMENT/SUPPLIES

- Environmental chamber maintained at 25 ± 1°C with timer-controlled ambient lighting and a cold room at 4°C
- Thermometer, pH meter, dissolved oxygen meter, and conductivity meter for routine physical and chemical measurements
- $CuCl_2$ for reference toxicant testing
- *Ceriodaphnia dubia* cultures
- Balance - analytical, capable of weighing accurately to 0.01 mg
- Test chambers - ten 30-mL disposable plastic test chambers per concentration and control
- Numbered "cerio boards" for holding test chambers
- Clear covers to prevent contamination of test chambers
- 1–L and 400-mL beakers for test solution preparation
- Graduated cylinders
- 10-mL and 1-mL disposable pipets
- Yeast/Trout chow/Chlorophyll (YTC) and *Selenastrum* suspension to feed animals
- Dissecting microscope with substage lighting
- Safety equipment - lab coats, eye protection, gloves, and respirators as required

III. PROCEDURE

A. Pre-test Set up

1. Test animals are purchased from a reputable dealer and cultured in the laboratory.

2. Twenty four hours prior to test initiation, gravid females are isolated in 30-mL polystyrene cups containing laboratory control water. Isolated females are fed 200 µl each of *Selenastrum* suspension and vitamin-enriched YTC.

Approved By:

Laboratory Manager (Date) QA Supervisor (Date)

3. On the day of the test initiation, neonates (<24 hour old) produced by the isolated females are collected and placed in a crystallizing dish containing control water and food. Only neonates originating from broods of 8 or more are used in tests.

B. Test Initiation

1. Samples are checked in upon receipt. See procedural SOP for details on routine sample check-in procedures. Routinely, tests must be initiated within 36 hours of sample collection. Check with laboratory supervisor for client-specific differences.

2. Record a brief sample description with matrix, color, odor, and particulate information on the data sheet.

3. Measure residual chlorine concentration, hardness, alkalinity, and any adjustments made to pH of the control, receiving, and sample waters and record on the data sheet.

4. Prepare dilutions according to test specific concentrations. Concentrations are directed by permit language. Bioassays which require a 0.5 dilution series are serially diluted. All other concentrations must be measured with graduated cylinders and pipettes.

5. Measure and record physical and chemical parameters in the control, middle, and high concentrations. Water quality measurements include pH, dissolved oxygen, conductivity, and temperature.

6. Warm each dilution to 25±1°C.

7. Obtain a numbered "cerio board" and place the appropriate number of cups on the board (10 cups/concentration and control). Each vertical row will represent one concentration.

8. Distribute 200 µl each of *Selenastrum* suspension and vitamin-enriched YTC to each test chamber.

9. Distribute approximately 15 mL of the appropriate test solution into each of the ten test containers.

10. Into each test container place one neonate, hereafter referred to as the "Alpha organism."

11. Place the test "cerio board" in the 25°C environmental chamber and cover.

C Daily Monitoring

1. Each successive day of the test prepare new test solutions, warm to 25±1°C, and distribute to new test containers. Distribute 200 µL of each food to each test container.

2. View test containers under a dissecting scope. Count and record the number of neonates and the survival status of the Alpha organism. Remove the Alpha organism with a wide bore transfer pipette, and place in the corresponding cup containing renewed solution.

3. Composite and save the test solution from the control, middle, and high concentrations for final water quality measurements.

4. Measure and record chemical and physical parameters (pH, DO, conductivity, and temperature) of the composite samples.

D. Test Termination

1. View each test container under the dissecting scope. Count and record the number of neonates and the survival status of the Alpha organism.

2. If control survival is greater than or equal to 80 percent and 60 percent of surviving females are in their third brood with an average of 15 or greater juveniles per female, the test is terminated. If the test is terminated on day 6, the animals are maintained until day 7 for the purpose of evaluating final survival status of each organism.

3. Composite and save the test solution from the control, middle, and high concentrations for final water quality measurements.

4. Measure and record chemical and physical parameters (pH, DO, conductivity, and temperature) of the composite samples.

IV. TEST ACCEPTABILITY CRITERIA

This is not a time-dependent test although extension or shortening of more than one day may indicate problems and should prompt close examination of test procedures and animal condition. The acceptability criteria for test termination are the following:

- 80% survival in the controls (including males)
- 60% of surviving females reaching at least three broods
- An average of 15 or greater offspring per surviving female

In addition, a valid reference toxicant test with a logical dose response curve should be obtained, using animals from the same batch.

V. HEALTH AND SAFETY

Health and safety precautions and applicable regulations should be considered at all times. Gloves should always be worn when handling effluents.

VI. PERSONNEL

Only qualified technicians who have been properly trained and can demonstrate competency with these techniques are permitted to conduct this test.

VII. QUALITY ASSURANCE REQUIREMENTS

Quality assurance practices encompass all aspects of testing including the collection, handling, and preparation of test organisms, samples, and dilution waters. Proper record keeping is required and concentrated efforts are made for complete documentation on a real-time basis.

VIII. REFERENCE

"Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms." EPA/600/4-89/001, March 1989.

C-3 48-Hr or 96-Hr Static Renewal Test Using *Pimephales promelas*

I. PURPOSE

This method estimates the acute toxicity of whole effluents and receiving water to the fathead minnow *Pimephales promelas* in a 48-hour static or 96-hour static-renewal test. Results are based on survival.

II. EQUIPMENT/SUPPLIES

- Environmental chamber maintained at either 20±1°C or 25±1°C and cold room at 4°C
- Thermometer, pH meter, dissolved oxygen meter, and conductivity meter
- 10,000 *ug*/L $CuCl_2$ for reference toxicant testing
- Fathead minnow larvae 1-14 days old
- Brine shrimp (*Artemia* cysts) for feeding
- Test chambers - two to four 1L test chambers are required for each concentration and control
- Clear covers to prevent contamination of test chambers
- 2-liter beakers, graduated cylinders, and pipettes for test solution preparation
- Deionized and Perrier water for 8:2 water preparation
- Safety equipment - lab coats, eye protection, gloves, and respirators as required

III. PROCEDURE

A. Pre-test Set up

1. Laboratory control water is prepared 24 hours (minimum) to 14 days (maximum) prior to testing. AMEC uses 20% dilute mineral water (8:2 water). See procedural SOP for specifications on preparation of 8:2 water.

2. Set up *Artemia* cysts for hatching 24 hours prior to testing. See procedural SOP on specifics on the preparation and harvesting of Artemia.

3. Fish larvae are purchased from a reputable dealer and shipped via overnight delivery to the bioassay laboratory. The fish must be 1-14 days old upon test initiation. The larvae are acclimated to testing temperature (25 ± 1°C or 20 ± 1°C) upon receipt and fed *Artemia* nauplii immediately prior to shipping and during holding.

B. Test Initiation

1. Samples are checked in upon receipt. See procedural SOP for details on routine sample check-in procedures. Tests are initiated within 36 hours of sample collection. Check with laboratory

supervisor for project-specific differences.

2. Record sample description with matrix, color, odor, and particulate information.

3. Measure and record chlorine content, hardness, alkalinity, and any adjustments made to the control, receiving, and sample waters.

4. Prepare dilutions according to test specific concentrations. Concentrations are directed by permit language. Bioassays which require a 0.5 dilution series are serially diluted. All other concentrations must be measured with graduated cylinders and pipettes.

5. Warm each dilution to 25±1°C or 20±1°C

6. Measure and record water quality parameters in each test chamber. Water quality parameters include pH, DO, conductivity, and temperature.

7. Distribute 500 mL of each test solution into each of two properly labeled test containers.

8. Randomly distribute 10 fathead larvae to each test chamber and place the test containers in the 25°C or the 20°C environmental chamber.

C. Daily Monitoring

1. Animals are fed prior to the 48 hour test solution renewal period (96 static-renewal test). Animals are not fed during the test period (48 hour static test).

2. Measure and record water quality parameters for each test chamber. Count and record number of surviving fish.

RENEWAL (96-HOUR STATIC-RENEWAL TEST ONLY)

1. The 96-hour test requires test solution renewal at 48 hours. Prepare test solutions as instructed above.

2. Measure and record water quality parameters for each test chamber. Count and record number of surviving fish.

3. To renew the tests, siphon test solution leaving 15-20 percent of the original volume. Excess food and debris should be cleaned from the bottom using a disposable transfer pipette.

4. Gently pour the new test solution down the side of each test chamber to avoid subjecting the organisms to excessive turbulence.

D. Test Termination

1. Count the number of surviving fish and record on the data sheet.

2. Measure final water quality parameters in each test chamber.

3. Dispose of all test materials in accordance with local, state, and federal regulations.

IV. TEST ACCEPTABILITY CRITERIA

Control survival at test termination must be 90 percent or greater for test results to be considered acceptable.

V. HEALTH AND SAFETY

Health and safety precautions and applicable regulations should be considered at all times. Gloves should always be worn when handling effluents.

VI. PERSONNEL

Only qualified technicians who have been properly trained and can demonstrate competency with these techniques are permitted to conduct this test.

VII. QUALITY ASSURANCE REQUIREMENTS

Quality assurance practices encompass all aspects of testing including the collection, handling, and preparation of test organisms, samples, and dilution waters. Proper record keeping is required, and concentrated efforts are made for complete documentation on a real-time basis.

VIII. REFERENCE

"Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms." EPA/600/4-90/027F, August 1993.

C-4 7-Day Static Renewal Tests Using *Pimephales promelas*

7 - DAY STATIC RENEWAL TEST USING *PIMEPHALES PROMELAS*

I. PURPOSE

This method estimates the chronic toxicity of whole effluents and receiving water to the fathead minnow *Pimephales promelas* in a seven day static-renewal test. Test results are based on the survival and growth (final mass) of the larvae.

II. EQUIPMENT/SUPPLIES

- Environmental chamber maintained at 25±1°C and cold room at 4°C
- Thermometer, pH meter, dissolved oxygen meter, and conductivity meter for routine physical and chemical measurements
- $CuCl_2$ for reference toxicant testing
- Fathead minnows, <24 hours old
- Brine shrimp (*Artemia* cysts)
- Balance - analytical, capable of weighing accurately to 0.01 mg
- Test chambers - four 400-mL disposable plastic test chambers per concentration and control
- Clear covers to prevent contamination of test chambers
- Light table for counting the number of surviving larvae
- 2-L and 1-L beakers for solution preparation
- Graduated cylinders
- 1- and 10-mL pipets
- Siphons for test renewal
- Deionized and Perrier water for 8:2 water preparation
- Safety equipment - lab coats, eye protection, gloves, and respirators as required

III. PROCEDURE

A. Pre-test Set up

1. Laboratory control water is prepared 24 hours (minimum) to 14 days (maximum) prior to testing. AMEC uses 20% dilute mineral water (8:2 water). See procedural SOP for specifications on preparation of 8:2 water.

Approved By:

Laboratory Manager (Date) QA Supervisor (Date)

2. Set up *Artemia* cysts for hatching 24 hours prior to testing. See procedural SOP or specifics on the preparation and harvesting of Artemia.

3. Fish larvae are purchased from a reputable dealer and shipped via overnight delivery to the bioassay laboratory. The larvae are acclimated to testing temperature (25 ± 1°C) upon receipt and fed *Artemia* nauplii during transport and holding. Immediately prior to testing, place ten larvae at random in individual 30-mL polystyrene cups filled with control water.

B. Test Initiation

1. Samples are checked in upon receipt. See procedural SOP for details on routine sample check-in procedures. Tests must be initiated within 36 hours of sample collection. Check with laboratory supervisor for project-specific differences.

2. Record sample description with matrix, color, odor, and particulate information on the data sheet.

3. Measure chlorine content, hardness, alkalinity, and any adjustments made to the control, receiving, and sample waters and record on the data sheet.

4. Prepare 1.0 L of each test concentration and control water. Prepare dilutions according to test specific concentrations. Concentrations are directed by permit language. Bioassays which require a 0.5 dilution series are serially diluted. All other concentrations must be measured with graduated cylinders and pipettes.

5. Warm each dilution to 25±1°C.

6. Measure and record physical and chemical parameters in the control, middle, and high concentrations. Parameters are pH, dissolved oxygen, conductivity, and temperature.

7. Distribute 250 mL of the test solutions into each of four properly labeled test containers. Add fish from step A.3 above.

9. Place the test containers in the 25°C environmental chamber.

10. Feed animals in each test container. See procedural SOP for specifications on feeding.

C. Daily Monitoring

1. Animals are fed three times per day. See procedural SOPs for feeding specifics.

2. Follow steps 4-6 under section III-B.

3. To renew the tests, siphon test solution leaving 15-20% of the original volume. Save a composite sample from the control, middle, and high concentrations. Excess food, dead animals and debris should be cleaned from the bottom using a disposable transfer pipet.

4. Count and record the number of surviving fish.

5. Gently pour the new test solution down the side of each test chamber to avoid subjecting the larvae to excessive turbulence.

6. Measure chemical and physical parameters (pH, DO, conductivity, and temperature) of the composite samples. Record in the appropriate column on the data sheets.

D. Test Termination

1. Fish are not fed on termination day.

2. Count and record the number of surviving fish.

3. Each test container is drained through a 35-µm mesh screen to collect surviving larvae. A composite sample is saved from the control, middle, and high concentrations for measurement of chemical and physical parameters. The fish are rinsed with deionized water, placed into a tared weighing pan with forceps, and dried at 100°C for a minimum of two hours or 60°C for 24 hours. See procedural SOP for specifications on weighing procedures.

IV. TEST ACCEPTABILITY CRITERIA

Control survival at test termination must be 80 percent or greater and the final mass per test animal must meet or exceed 0.25 mg for test results to be considered acceptable.

V. HEALTH AND SAFETY

Health and safety precautions and applicable regulations should be considered at all times. Gloves should always be worn when handling effluents.

VI. PERSONNEL

Only qualified technicians who have been properly trained and can demonstrate competency with these techniques are permitted to conduct this test.

VII. QUALITY ASSURANCE REQUIREMENTS

Quality assurance practices encompass all aspects of testing including the collection, handling, and preparation of test organisms, samples, and dilution waters. Proper record keeping is required and concentrated efforts are made for complete documentation on a real-time basis.

VIII. REFERENCE

"Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms." EPA/600/4-89/001, March 1989.

C-5 10-Day Static Freshwater Sediment Toxicity Tests with the Amphipod *Hyallela azteca*

10-DAY STATIC FRESHWATER SEDIMENT TOXICITY TESTS WITH THE AMPHIPOD (*HYALELLA AZTECA*)

I. PURPOSE

This method is designed to measure the acute toxicity of freshwater sediments to the amphipod *Hyalella Azteca* using survival and growth as the endpoints.

II. EQUIPMENT

- Proven source of clean freshwater (e.g.,Culligan water)
- Environmental chamber maintained at 20 ± 1°C
- Cold room maintained at 4°C to hold sediment samples prior to test initiation
- Thermometer, pH meter, dissolved oxygen (D.O.) meter, conductivity meter, dissecting light microscope, and analytical balance
- 1-L glass jars with lids (or equivalent) to serve as test exposure chambers (5 jars per test site, reference, and control); holes in lids to permit aeration
- 0.5-mm and 1.0-mm nitex mesh screens, transfer pipets, and glass holding bowls
- Aeration source, 1.0-mL pipets, and airline tubing
- Test, control, and reference sediments
- 30-mL plastic cups for holding animals; freshwater squirt bottle
- Safety equipment - lab coats, eye protection, gloves, and respirator as required

III. TEST PROCEDURE

A. Pre-test Set-up

1. Test animals are purchased from a reliable, experienced dealer and shipped to the lab via next day delivery service. Organisms are held in plastic bowls and allowed to acclimate for at least six hours to laboratory conditions before test initiation.

2. During this holding period animals are acclimated to lab conditions by replacing culture water with laboratory water by dripping laboratory test water into holding bowl. During acclimation temperature changes should not exceed 2°C per 24 hr period.

3. Samples are checked in upon receipt. See procedural SOP for details on sediment sample check-in procedures. Sediment samples are sieved through a 0.5-mm screen to filter out any resident organisms and debris. The sieved sediment is placed in a clean, labeled, plastic bag, and stored at 4°C until tests are initiated.

24-Hrs Prior to Test Initiation

4. Set up a randomization key using TOXCALC program.

Approved By:

Laboratory Manager (Date) QA Supervisor (Date)

5. Label test chambers according to the randomization sheet and add 2 cm of the appropriate sediment to the labeled jars.

6. Prepare an additional test chamber for each test site. This surrogate will be used for water quality measurements during the test period.

7. Fill jars to the 900 mL level with laboratory test water.

8. Place jars in the 20°C environmental chamber and let sit overnight to stabilize. Adjust light cycle in chamber to 16L/8D hour light cycle.

B. Test Initiation - Day 0

1. Measure and record water quality readings (D.O., conductivity, temperature, and pH) from the surrogate jars. Remove a subsample for ammonia analysis from each site and preserve with acid.

2. Place 10 animals into a 30-mL cup containing laboratory water.

3. A second count is made to verify that there are 10 animals in each cup.

4. Randomly distribute two cups with 20 test animals to each chamber by gently rinsing the contents of each cup into a test chamber with a laboratory water squirt bottle.

5. Aerate test chambers gently with 1-mL glass pipets placed in the water column with special care taken to prevent disturbing the sediment.

6. All test chambers are inspected within two hours of initiation to ensure that no amphipods are trapped in the surface tension of the water. Any floating animals found are replaced new animals.

C. Reference Toxicant Testing

1. A 96 hour reference toxicant is run concurrently with sediment tests to determine the quality of the test organisms. Potassium chloride is used a the reference toxicant. The testing concentrations for the reference toxicant are Control, 125,250, 500, 1000, and 2000 mg/L.

2. The LC_{50} for survival must be within two standard deviations of the control chart mean.

D. Daily Monitoring and Feeding Regime - Days 1 to 10

1. Measure and record pH, D.O., salinity, and temperature daily in surrogate chambers.

2. Observe all chambers and correct obvious problems (e.g., aeration rate) as required.

3. Test chambers are feed on Day 0, Day 2, and Day 6 with 1 mL of a 2g TetraMin per 100 mLs lab water mixture per test chamber.

E. **Test Termination - Day 10.**

1. Measure and record water quality (D.O., pH, salinity, and temperature) in surrogate test chambers. Remove a subsample for ammonia analysis from each site and preserve with acid.

2. Gently suspend sediment in the test chambers and pour onto 0.5-mm cylindrical screen. Rinse away remaining sediment using laboratory water within 2°C of the test temperature.

3. Count and record the number of alive, dead, or missing animals.

4. Collect and rinse surviving amphipods with deionized water. Using forceps, gently place on an appropriately labeled and tared aluminum pan.

5. Dry at 60°C for 24 hours.

6. Remove from oven. Weigh to the nearest 0.00001g and record on the data sheet.

IV. TEST ACCEPTABILITY CRITERIA

Mean control sediment survival at test termination must be 80 percent or greater. There is no acceptability criterion for the weight endpoint as it is not specified in the protocol.

V. HEALTH AND SAFETY

Health and safety precautions and applicable regulations should be considered at all times. Gloves must always be worn when handling test sediments.

VI. PERSONNEL

Only qualified technicians who have been properly trained and can demonstrate competency with these techniques are permitted to conduct this test.

VII. QUALITY ASSURANCE REQUIREMENTS

Quality assurance practices encompass all aspects of testing including the collection, handling, and preparation of test organisms, samples, and dilution waters. Proper record keeping is required and concentrated efforts are made for complete documentation on a real-time basis.

VIII. REFERENCES

ASTM Standards on Aquatic Toxicology and Hazard Evaluation. ASTM publication Code Number: 03-547093-16, September 1993. pp. 294-306.

"Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates." EPA 600/R-94/024, June 1994.

"Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S.- Testing Manual (Draft)." Environmental Protection Agency/Corps of Engineers. EPA-823-B-94-002, June 1994.

C-6 10-Day Static Freshwater Sediment Toxicity Testa with the Midge Larvae *Chironomus tentans*

STANDARD OPERATING PROCEDURE

10-DAY STATIC FRESHWATER SEDIMENT TOXICITY TESTS WITH THE MIDGE LARVAE (*CHIRONOMUS TENTANS*)

I. PURPOSE

This method is designed to measure the toxicity of freshwater sediments to the midge larvae *Chironomus tentans* using survival and growth as endpoints.

II. EQUIPMENT

- Proven source of clean freshwater water (e.g.,8:2 lab water)
- Environmental chamber maintained at 20 ± 1°C
- Cold room maintained at 4°C to hold sediment samples prior to test initiation
- Thermometer, pH meter, dissolved oxygen (D.O.) meter, conductivity meter, dissecting light microscope, and analytical balance
- 1-L glass jars with lids (or equivalent) to serve as test exposure chambers (5 jars per test site, reference, and control); holes in lids to permit aeration
- 0.5-mm and 1.0-mm nitex mesh screens, transfer pipets, and glass holding bowls
- Aeration source, 1.0-mL pipets, and airline tubing
- Test, control, and reference sediments
- 30-mL plastic cups for holding animals; freshwater squirt bottle
- Safety equipment - lab coats, eye protection, gloves, and respirator as required

III. TEST PROCEDURE

A. Pre-test Set-up

1. Test animals are purchased from a reliable, experienced dealer and shipped to the lab via next day delivery service. Organisms are held in plastic bowls and allowed to acclimate for at least six hours to laboratory conditions before test initiation.

2. During this holding period animals are acclimated to lab conditions by replacing culture water with laboratory water by dripping laboratory test water into holding bowl. During acclimation temperature changes should not exceed 2°C per 24 hr period.

3. Samples are checked in upon receipt. See procedural SOP for details on sediment sample check-in procedures. Sediment samples are sieved through a 0.5-mm screen to filter out any resident organisms and debris. The sieved sediment is placed in a clean, labeled, plastic bag, and stored at 4°C until tests are initiated.

Approved By:

Laboratory Manager (Date) QA Supervisor (Date)

24-Hrs Prior to Test Initiation

4. Set up a randomization key using TOXCALC program.

5. Label test chambers according to the randomization sheet and add 2 cm of the appropriate sediment to the labeled jars.

6. Prepare an additional test chamber for each test site. This surrogate will be used for water quality measurements during the test period.

7. Fill jars to the 900 mL level with laboratory test water.

8. Place jars in the 20°C environmental chamber and let sit overnight to stabilize. Adjust light cycle in chamber to 16L/8D hour light cycle.

B. Test Initiation - Day 0

1. Measure and record water quality readings (D.O., conductivity, temperature, and pH) from the surrogate jars. Remove a subsample for ammonia analysis from each site and preserve with acid.

2. Place 10 animals into a 30-mL cup containing laboratory water.

3. A second count is made to verify that there are 10 animals in each cup.

4. Randomly distribute 10 test animals to each chamber by gently rinsing the contents of each cup into a test chamber with a laboratory water squirt bottle.

5. Aerate test chambers with 1-mL glass pipets placed in the water column with special care taken to prevent disturbing the sediment.

C. Daily Monitoring and Feeding Regime - Days 1 to 10

1. Measure and record pH, D.O., salinity, and temperature daily in surrogate chambers.

2. Observe all chambers and correct obvious problems as required.

3. Test chambers are feed on Day 0 and every second day there after with 1 mL of a 2g TetraMin per 100 mLs lab water mixture per test chamber.

D. Reference Toxicant Testing

1. A 96 hour reference toxicant is run concurrently with sediment tests to determine the quality of the test organisms. Potassium chloride is used as the reference toxicant. The testing concentrations for the reference toxicant are Control, 1250, 2500, 5000, 7500, and 10000 mg/L.

2. The LC_{50} for survival must be within two standard deviations of the control chart mean for the laboratory.

E. **Test Termination - Day 10.**

1. Measure and record water quality (D.O., pH, salinity, and temperature) in surrogate test chambers. Remove a subsample for ammonia analysis from each site and preserve with acid.

2. Gently suspend sediment in the test chambers and pour onto 0.5-mm cylindrical screen. Rinse away remaining sediment using laboratory water within 2°C of the test temperature.

3. Count and record the number of alive, dead, or missing animals.

4. The surviving animals are rinsed with deionized water, placed onto tared weighing pan with forceps, and dried at 60 °C to a constant weight (≥24 hours). Report total weight to the nearest 0.1 mg.

IV. TEST ACCEPTABILITY CRITERIA

Mean control sediment survival at test termination must be 70 percent or greater. Average weight of *C. tentans* in the control sediment must be at least 0.6 mg at test termination.

V. HEALTH AND SAFETY

Health and safety precautions and applicable regulations should be considered at all times. Gloves must always be worn when handling test sediments.

VI. PERSONNEL

Only qualified technicians who have been properly trained and can demonstrate competency with these techniques are permitted to conduct this test.

VII. QUALITY ASSURANCE REQUIREMENTS

Quality assurance practices encompass all aspects of testing including the collection, handling, and preparation of test organisms, samples, and dilution waters. Proper record keeping is required and concentrated efforts are made for complete documentation on a real-time basis.

VIII. REFERENCES

ASTM Standards on Aquatic Toxicology and Hazard Evaluation. ASTM publication Code Number: 03-547093-16, September 1993. pp. 294-309.

"Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates." EPA 600/R-94/024, June 1994.

"Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S.- Testing Manual (Draft)." Environmental Protection Agency/Corps of Engineers. EPA-823-B-94-002, June 1994.

C-7 28-Day Bioaccumulation Analyses with the Asiatic Clam *Corbicula fulminea*

I. PURPOSE

This method exposes the Asiatic clam *Corbicula fluminea* to sediment for 28 days followed by a 48-hr depuration period. Animal tissue is analytically assayed to determine bioaccumulation of site-specific constituents of concern.

II. EQUIPMENT

- Proven source of clean freshwater water (e.g.,Culligan water)
- Flow-through or static-renewal system capable of providing a renewed supply of freshwater maintained at a temperature of 20 ± 1°C
- Coldroom for storing sediment samples capable of maintaining 4°C
- Thermometer, pH meter, dissolved oxygen (D.O.) meter, conductivity meter, and an analytical balance
- 38-L glass aquaria
- 1.0-mm nitex sieve
- Test, control, and reference sediments
- Clean, food-grade plastic bags to hold tissue samples after test termination
- Freezer capable of maintaining frozen tissue samples at approximately -20°C
- Safety equipment - lab coats, eye protection, gloves and respirator as required

III. PROCEDURE

A. Pre-test Set-up

1. Test animals are purchased from a reliable, experienced dealer and shipped to the lab via next day delivery service.

2. Test animals are acclimated to test conditions in the laboratory for 1-2 days prior to test initiation.

Approved By:

Laboratory Manager (Date) QA Supervisor (Date)

3. Sediment samples are checked in upon receipt. See procedural SOP for details on sediment sample check-in procedures. Sediment samples are sieved through a 0.5 mm screen to filter out any resident organisms or debris. The sieved sediment is placed in a clean, labeled plastic bags and stored at 4°C until test initiation.

4. Test exposures are initiated within the appropriate holding time.

24-Hr Prior to Test Initiation

5. Label test chambers, following with the addition of test, reference, or control sediment to a final sediment depth of at minimum 5 cm in each replicate (5).

6. Place aquaria in the designated testing area.

7. Set-up static-renewal or flow-through equipment to renew water in aquaria.

8. Allow test chambers to equilibrate overnight prior to introducing test animals.

B. Test Initiation - Day 0

1. Measure and record water quality readings (DO, conductivity, temperature, and pH) from each replicate tank. Remove a subsample for ammonia analysis from each site and preserve with acid.

2. Adjust water renewal rate as required to maintain the dissolved oxygen above 5.0 mg/L.

3. Randomly distribute 20-30 clams to each replicate tank. Number of animals per replicate should correlate to tissue volume requirements.

C Daily Monitoring - Days 1-28

1. Measure and record pH, DO, conductivity, and temperature daily.

2. Monitor all replicates for mortality and remove dead organisms; record mortality on the water quality data sheets.

3. Adjust water renewal volume and rate as required to maintain DO levels over 5.0 mg/L.

4. Feed ground Tetramin food every third day to each test chamber.

D. Depuration - Days 28-30

1. Label 38-L aquaria with the same identification numbers as the test sites and place 5-8 cm of sieved control sediment into the aquaria.

2. Transfer the contents of each replicate to a 1.0 mm screen and wash away the testing material with test temperature freshwater.

3. Count and record surviving control test animals.

4. Place surviving test organisms into corresponding aquaria from step 1.

5. Allow test animals to depurate for a period of 24-48 hours.

E. Test Termination - Day 30

1. Prepare a randomization key using EXCEL program.

2. Label plastic bags with random number, client name, and date.

3. Remove surviving test organisms and place in the appropriately labeled bag.

4. Freeze samples at -20°C overnight; deliver frozen samples to chemistry laboratory for analysis.

IV. TEST ACCEPTABILITY CRITERIA

Mean control survival at test termination should be at least 70 percent. This level of survival verifies that the test organisms used were healthy.

V. HEALTH AND SAFETY

Health and safety precautions and applicable regulations should be considered at all times. Gloves must always be worn when handling test sediment.

VI. PERSONNEL

Only qualified technicians who have been properly trained and can demonstrate competency with these techniques are permitted to conduct this test.

VII. QUALITY ASSURANCE REQUIREMENTS

Quality assurance practices encompass all aspects of testing including the collection, handling, and preparation of test organisms, samples, and dilution waters. Proper record keeping is required and concentrated efforts are made for complete documentation on a real-time basis.

VIII. REFERENCES

Compilation of general guidance from:

Portland Harbor Sediment Management Plan, June, 1999.

"Evaluation of Dredged Material Proposed for Ocean Disposal." (Green Book Testing Manual), February 1991. Environmental Protection Agency/Corps of Engineers. 1991.

Appendix D

Pennington & Associates

Standard Operating Procedures

D-1 Laboratory Processing and Benthic Community Samples for Taxonomic Identification

STANDARD OPERATING PROCEDURE
LABORATORY PROCESSING OF BENTHIC COMMUNITY SAMPLES FOR TAXONOMIC IDENTIFICATION

1.0 GENERAL APPLICABILITY

This Standard Operating Procedure describes how benthic macroinvertebrates samples received by laboratory will be processed and identified. This SOP may be supplemented and modified by lab-specific protocols provided by Pennington & Associates, Inc.

2.0 PROCEDURE

The preservative will be removed from the sample to prevent exposure of volatile fumes to the sorter. This will be done by pouring the sample through a 250-um mesh screen. Large debris (rocks, leaves, twigs etc.) will be rinsed and discarded from the sample.

The procedure provided in USEPA 1998 will be followed. Subsampling will be done to obtain a minimum of 200 organisms from the sample. The sample will be divided into eight aliquots using a specially designed subsampler. Each aliquot will be placed into a separate container and examined until a minimum of 200 organisms is reached. An aliquot that has been started will be completed regardless of the number of organisms, to enable a total number to be calculated. Contents will be examined in gridded petri dishes using a dissecting microscope, and all organisms removed, counted and placed in labeled vials containing 70% ethyl alcohol. The remainder of the sample (i.e., the portion from which no subsamples have been taken) will be re-preserved with the preservative used by the lab (alcohol, formalin, etc.). Before preservation, one subsample will be kept separate, in case the identifiers need more material (e.g., some of the oligochaetes worms are fragments and more individuals are then needed to comprise the 200 specimen count).

2.1 Quality Control Checks

Quality control checks will be performed on 10 percent of the samples for each sorter.

If less than 10 percent of the total number of organisms is found in the QC check, no more samples will be checked. If greater than 10 percent of the total number of organisms is found in any sample, then another 10 percent will be checked. The results of the QC checks will be presented with other data.

2.2 Identification

After the organisms have been removed according to the procedures outlined above, all of the organisms will be identified. The dissecting microscope is used for all identifications except for chironomid larvae and oligochaetes, or when specific parts of an organism have to be checked (e.g., water mite palps, plecopteran lacinia). Chironomids and oligochaetes will be identified using compound microscopes. These organisms will be mounted under a coverslip with two coverslips per slide, and five oligochaetes under a coverslip with two coverslips per slide. Separate data sheets are used for the identification of oligochactes and chironomid larvae. These sheets are designed so that a specimen can be located with a minimum of effort.

The following taxonomic groups will be identified to the genera/species level:

Oligochaeta, Isopoda, Decapoda, Hydrachnida, Ephemeroptera, Odonata, Plecoptera, Hemiptera, Megaloptera, Neuroptera, Trichoptera, Coleoptera, Diptera (including Chironomidae), Gastropoda and Bivalvia.

3.0 DOCUMENTATION

3.1 Voucher Collection

A voucher collection will be provided. For each taxon encountered, one to three individuals are removed per taxon for the collection.

3.2 Data Analysis

All data will be entered into Excel and provided in the prescribed formnat (Excel, Lotus, ASCII). The data will be analyzed for taxa richness, abundance, percent dominant taxon/taxa, and community composition.

4.0 REFERENCES

USEPA. 1998. Lake and Reservoir Bioassessment and Biocriteria: Technical Guidance Document, EPA Office of Water, EPA 841-B-98-007

VOLUME 3C
SITE-SPECIFIC HEALTH AND SAFETY PLAN
For the
ECOLOGICAL RISK ASSESSMENT SUPPORT SAMPLING

SAUGET AREA 2
SITES O, P, Q, R, S
SAUGET & CAHOKIA, ILLINOIS

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3 SITE AIR SURVEILLANCE RECORD
THERMO 580B CALIBRATION AND OPERATION
E HOSPITAL ROUTE MAP AND EMERGENCY
F HEALTH AND SAFETY ORIENTATION FORM

LIST OF ACRONYMS AND ABBREVIATIONS

ABRTF	American Bottoms Regional Water Treatment Facility
BTEX	Benzene, toluene, ethylbenzene, xylenes
CERCLA	Comprehensive Environmental Responsibility Compensation and Liability Act
CPR	Cardiopulmonary resuscitation
DOT	Department of Transportation
HASP	Health and Safety Plan
HSM	Project Health and Safety Manager
IDLH	Immediately Dangerous to Life and Health
IEPA	Illinois Environmental Protection Agency
MSDS	Material Safety and Data Sheet
OSHA	Occupational Safety and Health Administration
PAH	Polynuclear hydrocarbon
PCB	Polychlorinated Biphenyl
PEL	Permissible Exposure Limit
PPE	Personal Protective Equipment
PM	Project Manager
SOPs	Standard Operating Procedures
USCG	United States Coast Guard
USEPA	United States Environmental Protection Agency

1.0 INTRODUCTION

The purpose of this document is to establish standard health and safety procedures for AMEC Earth and Environmental, Inc. (AMEC) employees and their subcontractors in performance of the planned field activities associated with conducting the ecological risk assessment at the Sauget Area 2 Sites O, P, Q, R, and S, located in Sauget and Cahokia, Illinois (Figure 1).

Site activities shall be performed in accordance with AMEC health and safety policies and procedures; applicable Occupational Safety and Health Administration (OSHA) Standards; 29 CFR Part 1910 and 1926; applicable Environmental Protection Agency (EPA) requirements; and consensus standards. Where the word "shall" is used, the provisions of this plan are mandatory.

The levels of personal protection and the procedures specified in this plan are based on the best information available from reference documents and site characterization data. Therefore, these recommendations represent the minimum health and safety requirements to be observed by all personnel engaged in this project. Unforeseeable site conditions or changes in scope-of-work may warrant a reassessment of protection levels and controls stated. All adjustments to the site-specific health and safety plan (HASP) must have prior approval by the AMEC Site Health and Safety Officer (SHSO), the AMEC Corporate Health and Safety Manager (HSM), the AMEC Project Manager, and other designated authorities.

All personnel involved in this project must read this document carefully. Any questions or concerns, which the reader feels are not adequately addressed should be directed to the AMEC SHSO and/or HSM. Personnel on-site shall be required to follow all appropriate health and safety procedures; be alert to the hazards associated with work on a hazardous waste site; be aware of the hazards associated with working over or near water; and exercise reasonable caution at all times. Personnel have the right to refuse to work under conditions which they feel are of a health or safety concern until the concern is appropriately resolved.

All site visitors must receive prior approval from the SHSO and may do so only for the purpose of observing site conditions or operations. Upon arrival, visitors will report to the SHSO where he/she will be logged in the field notebook, required to fill out a medical data sheet and undergo a safety and evacuation orientation (as necessary, given the nature of ongoing work activities).

Visitors will be expected to comply with relevant OSHA requirements such as medical surveillance, training, and respiratory protection as determined by the Site Health and Safety Officer.

2.0 SITE DESCRIPTION AND FEATURES

Sauget Area 2 is located in the City of East St. Louis and the Villages of Sauget and Cahokia in St. Clair County, Illinois. The Sauget Area 2 study area is east of the Mississippi River and south of the MacArthur bridge railroad tracks. The study area is west of Route 3 (Mississippi Avenue) and north of Cargill Road.

Site	Former Use	Municipality
Site O	Sewage Sludge Dewatering	Village of Sauget
Site P	Municipal and Industrial Waste Disposal	City of East St. Louis Village of Sauget
Site Q	Municipal and Industrial Waste Disposal	Village of Sauget Village of Cahokia
Site R	Industrial Waste Disposal	Village of Sauget
Site S	Chemical Reprocessing Waste Disposal	Village of Sauget

These sites are located in an area historically used for heavy industry, including chemical manufacturing, metal refining and power generation, and waste disposal. Currently the area is used for heavy industry, warehousing, bulk storage (coal, refined petroleum, lawn and garden products and grain), waste water treatment, hazardous waste treatment, waste recycling and truck terminals. Four commercial establishments are located at the north end of the study area. No residences are located within the study area. Residential areas closest to Sauget Area 2 are approximately 3,000 feet east of Site P and about 3,000 feet east of Site O. These residential areas are located, respectively, in East St. Louis and Cahokia.

2.1 SITE LOCATION AND PHYSICAL SETTING

Sauget Area 2 is located in the floodplain of the Mississippi River in an area known as American Bottoms. Topographically, the area consists primarily of flat bottom land although local topographic irregularities do occur. Generally, land surface in the American Bottoms slopes from north to south and from east to west toward the Mississippi River. Land surface elevation ranges from 400 to 410 feet above Mean Sea Level (MSL) with little topographic relief.

Sauget Area 2 consists of five former disposal areas, Sites O, P, Q, R and S, adjacent, or in close proximity, to the Mississippi River. These five disposal areas were given letter designations by the

Illinois Environmental Protection Agency (IEPA) in the 1980s. Two of these sites, Sites Q and R, are located on the wet side of the floodwall and levee which is operated and maintained by the US Corps of Engineers and the Metro East Sanitary District. The floodwall is designed to protect the City of East St. Louis and the Villages of Sauget and Cahokia from flooding. Sites O, P and S are located on the dry side of the floodwall and levee.

2.2 PRESENT AND PAST FACILITY OPERATIONS AND DISPOSAL PRACTICES

Each of the five sites in Sauget Area 2 is described below. Maximum chemical concentrations included in these site descriptions were included by USEPA in the AOC and are summarized in Table 1.

2.2.1 Site O

Site O, located on Mobile Avenue in Sauget, Illinois, occupies approximately 20 acres of land to the northeast of the American Bottoms Regional Wastewater Treatment Facility (ABRTF). An access road to the ABRTF runs through the middle of the site. In 1952, the Village of Sauget Waste Water Treatment Plant began operation at this location. In addition to providing treatment for the Village of Sauget, the plant treated effluent from the various Sauget industries.

During its operation the treatment plant received and treated industrial and municipal wastewater. Approximately 10 million gallons per day of wastewater was treated most of which was from area industries.

Four lagoons were constructed at the wastewater treatment plant in 1965 and placed in operation in 1966/1967. Between 1966/67 and approximately 1978, these lagoons were used to dispose of clarifier sludge from the wastewater treatment plant. They were designated as Site O during a site investigation conducted by IEPA in the 1980s. The lagoons were closed in 1980 by stabilizing the sludge with lime and covering it with approximately two feet of clay. Currently, the lagoons are covered with clay and are vegetated.

Parties that USEPA alleges discharged to the Sauget Wastewater Treatment Plant during the time period that the sludge lagoons were in operation included, at a minimum:

- Amax Zinc Corporation,
- American Zinc Company
- Cerro Copper Products Company
- Clayton Chemical Co.
- Darling Fertilizer
- Ethyl Corporation
- Ethyl Petroleum Additives, Inc.
- Midwest Rubber Reclaiming
- Mobil Oil Corporation
- Monsanto Company
- Rogers Cartage Company
- Wiese Planning and Engineering

Parties which own and/or operate, or previously owned and/or operated, portions of Site O include:

- Village of Sauget

The USEPA alleges that soil samples collected from Site O contain VOCs, SVOCs, PCBs, dioxin and metals at concentrations of up to:

VOCs (ppb)	
Benzene	30,769
Chlorobenzene	58,974
Ethylbenzene	166,667
4-Methyl-2-Pentanone	7,692
Toluene	29,487
Xylenes	615,385

PCBs (ppb)	
Arochor 1232	30,366
Arochor 1242	1,871,795

Dioxin (ppb)	
Tetrachlorodibenzo-p-dioxin	170

Metals (ppm)	

SVOCs (ppb)	
1,2-Dichlorobenzene	606,000
1,3-Dichlorobenzene	112,821
1,4-Dichlorobenzene	1,030,000
1,2,4-Trichlorobenzene	65,300
1,1,1-Trichloroethane	1,410
1,2,4-Trichlorophenol	26,923
Pentachlorophenol	1,620,000
Benzo(a)anthracene	121,795
Chrysene	282,051
Fluoranthene	74,000
Naphthalene	34,615
Phenanthrene	230,000
Pyrene	282,051
2-Methylnaphthalene	160,256
n-Nitrosodiphenylamine	50,000
Butyl Benzyl Phthalate	3,846,154

Cadmium	31
Copper	341
Mercury	6.3
Nickel	136
Zinc	1,398

The USEPA alleges that groundwater samples collected from Site O contain VOCs, SVOCs and metals at concentrations of up to:

VOCs (ppb)	
Benzene	190,000
2-Butanone	62,000
Chlorobenzene	180,000
trans-1,2-Dichloroethene	14,000
Methylene Chloride	52,000
4-Methyl-2-Pentanone	38,000
1,1,2,2-Tetrachoroethane	12,000
Tetrachloroethene	10,000
Toluene	15,000
Trichloroethene	83,000

SVOCs (ppb)	
4-Chloroaniline	780
1,2-Dichlorobenzene	11,000
1,4-Dichlorobenzene	15,000
4-Methylphenol	1,100
Phenol	1,100

Metals (ppb)	
Arsenic	113
Cadmium	11
Lead	6,350

2.2.2 Site P

Site P, which is bounded by the Illinois Central Gulf Railroad tracks, the Terminal Railroad Association tracks and Monsanto Avenue, occupies approximately 20 acres of land located in the City of East St. Louis and the Village of Sauget. It was operated by Sauget and Company as an IEPA-permitted landfill from 1973 to approximately 1984 accepting general wastes, including diatomaceous earth filter cake, from Edwin Cooper (now Ethyl Corporation) and non-chemical wastes from Monsanto. IEPA inspections documented the presence of drums labeled "Monsanto ACL-85, Chlorine Composition," drums labeled phosphorus pentasulfide from Monsanto and Monsanto ACL filter residues and packaging. Site P is currently inactive and partially covered, however, access to the site is not restricted.

Parties which USEPA alleges to have generated, disposed of, released into and/or transported wastes to Site P include:

- Edwin Cooper Petroleum Additive
- Monsanto Chemical Company

USEPA alleges that parties who potentially own, previously owned and/or operated Site P include:

- Chicago Title & Trust Company
- Gulf-Mobile & Ohio Railroad
- Metro East Sanitary District
- Sauget and Company
- Southern Railway System
- Union Electric Company

USEPA alleges that soil samples collected from Site P contain VOCs, SVOCs and metals at concentrations of up to:

VOCs (ppb)	
Toluene	413
Xylenes	450

Metals and Inorgancis (ppm)	
Lead	526
Mercury	3.9
Cyanide	15

SVOCs (ppb)	
1,2-Dichlorobenzene	3,625
1,4-Dichlorobenzene	8,875
Phenol	3,875
Di-n-butyl phthalate	16,250

2.2.3 Site Q

Site Q, a former subsurface and surface disposal area, occupies approximately 90 acres in the Villages of Sauget and Cahokia. This Site is divided by the Alton and Southern Railroad into a northern portion and a southern portion. The northern portion consists of 65 acres bordered on the north by Site R and Monsanto Avenue, The northern portion is bordered on the south by the main track of the Alton and Southern Railroad and property owned by Patgood Inc. On the east, the northern portion of the site is bordered by the Illinois Gulf Central Railroad and the US Army

Corps of Engineers (USACE) flood control levee and on the west the Site is bordered by the Mississippi River.

The southern portion consists of 25 acres, north of Cargill Road and south of the Alton and Southern Railroad. The southern portion is bounded on the west by a ten-foot wide strip of property owned by Union Electric for transmission lines and a spur track of the Alton and Southern Railroad to the Fox Terminal. A barge terminal operated by St. Louis Grain Company is located between the Union Electric property, the spur track and the Mississippi River. Southern Site Q is bordered on the east by the Illinois Central Gulf Railroad and the flood control levee.

Disposal started in the 1950s and continued until the 1970s. Sauget and Company started operation of a landfill south of the River Terminal in 1966 and terminated operations in 1973. This facility took various wastes including municipal waste, septic tank pumpings, drums, organic and inorganic wastes, solvents, pesticides and paint sludges. It also took plant trash from Monsanto, waste from other industrial facilities and demolition debris.

Most of Site Q is covered with highly permeable black cinders. Eagle Marine Industries and Peavy Company, a division of Con-Agra, operate barge terminal facilities in the central part of the northern portion of Site Q. The southern portion of Site Q is used for reclaiming rebar from concrete and for construction debris disposal. A ten-acre site on the northern portion of Site Q is currently used by Rivercity Landscape Supply as a bulk storage terminal for lawn and garden products. Raw landscape products such as mulch, rock and soil are process and packaged are also processed and packed on this portion of the site.

Access to some portions of the site is restricted by fencing and gates. Other parts of the site have unrestricted access.

Site Q is on the west side of the USCOE floodwall. In 1993, during the highest recorded flood in St. Louis' history, Site Q was flooded. USEPA conducted a CERCLA removal action at the northern portion of Site Q in 1995. USEPA conducted a second CERCLA removal action at the southern portion of Site Q beginning in October of 1999 and into early 2000. During this removal action, USEPA excavated over 2000 drums and over 7,000 cubic yards of contaminated soils containing metals, PCBs, and organics. Excavated material was transported by rail to Oklahoma for disposal at Safety Kleen's Lone Elk hazardous waste landfill.

USEPA alleges that the following parties potentially generated, disposed of, released into and/or transported wastes to Site Q;

- AALCO Wrecking Company, Inc.
- Abco Trash Service
- Able Sewer Service
- Ajax Hickman Hauling
- Atlas Service Company
- Banjo Iron Company
- Barry Weinmiller Steel Fabrication
- Becker Iron & Metal Corporation
- Belleville Concrete Cont. Company
- Bi-State Parks Airport
- Bi-State Transit Company
- Boyer Sanitation Service
- Browning-Ferris Industries of St. Louis
- C&E Hauling
- Cargill Inc.
- Century Electric Company
- Circle Packing Company
- Clayton Chemical Company
- Corkery Fuel Company
- Crown Cork & Seal Company, Inc.
- David Hauling
- Dennis Chemical Company, Inc.
- Disposal Service Company
- Dore Wrecking Company
- Dotson Disposal "All" Service
- Dow Chemical
- Edgemont Construction
- Edwin Cooper Inc.
- Eight & Trendy Metal Company
- Evans Brothers
- Finer Metals Company
- Fish Disposal
- Fruin-Colnon Corporation
- Gibson Hauling
- H.C. Fournie Inc.
- H.C. Fournie Plaster
- Hilltop Hauling
- Huffmeier Brothers
- Hunter Packing Company
- Illinois Department of Transportation
- Inmont Corporation
- Lefton Iron & Metal Company
- Mallinckrodt Chemical
- Midwest Sanitation
- Mississippi Valley Control
- Monsanto Company
- Myco-Gloss
- Obear Nestor
- Roy Baur
- Thomas Byrd
- Trash Men Inc.
- United Technologies Corporation
- U.S. Paint Corporation

USEPA alleges that the following parties potentially own, previously owned and/or operated Site Q include:

- Cahokia Trust Properties
- ConAgra, Inc. (lessee)
- Eagle Marine Industries Inc.
- Industrial Salvage & Disposal Company
- Peavey Company
- Phillips Pipe Line Company
- Pillsbury Company (lessee)
- Sauget & Company
- Union Electric Company
- Village of Cahokia
- Village of Sauget

Soil samples collected from Site Q allegedly contain VOCs, SVOCs,. metals, PCBs and dioxin. at concentrations of up to:

VOCs (ppb)	
Chlorobenzene	100,000
Ethylbenzene	790,000
4-Methyl-2-Pentanone	250,000
Toluene	2,400,000
Xylenes	2,300,000

PCBs (ppb)	
Aroclor 1248	70,000
Aroclor 1254	360,000
Aroclor 1260	16,000,000

Dioxin (ppb)	
2,3,7,8-TCDD	3.31

SVOCs (ppb)	
1,2-Dichlorobenzene	3,625
1,4-dichlorobenzene	1,200,000
Bis(2-ethylhexyl) phthalate	1,100,000
Di-n-butylphthalate	900,000

Metals (ppm)	
Antimony	17,900
Arsenic	0.216
Cadmium	152,000
Chromium	3,650
Copper	1,630
Lead	195,000
Mercury	4.9
Nickel	371
Selenium	59.5
Silver	30.2
Thallium	0.89
Zinc	9,520

Groundwater samples collected from Site Q contain VOCs, SVOCs, metals and Inorganics at concentrations of up to:

VOCs (ppb)	
Benzene	2,000
Chlorobenzene	6,700
1,2-Dichloroethane	3,000
2-Hexanone	3,500
4-methyl-2-pentanone	2,700
Toluene	1,600

Metals and Inorganics (ppm)	
Arsenic	0.100
Cyanide	1,560

SVOCs (ppb)	
4-Chloroaniline	15,000
Phenol	190,000
2-Chlorophenol	33,000
2, 4-Dichlorophenol	14,000
2,4,6-Trichlorophenol	6,000
Pentachlorophenol	35,000
4-Methylphenol	23,000
2,4-Dimethylphenol	2,800
2-Nitroaniline	2,000
Acenaphthylene	3,900

2.2.4 Site R

Site R, a closed industrial-waste disposal area owned by Solutia Inc, is located between the flood control levee and the Mississippi River in Sauget, Illinois. Its northern border is Monsanto Avenue and its southern border is Site Q. A portion of Site Q, known as the "Dog Leg", is located to the east of Site R. This site once called the "Sauget Toxic Dump" and the "Monsanto Landfill" it is now known as the "River's Edge Landfill".

Industrial Salvage and Disposal, Inc. (ISD) operated the River's Edge Landfill for Monsanto from 1957 to 1977. Hazardous and non-hazardous bulk liquid and solid chemical wastes and drummed chemical wastes from Monsanto's W.G. Krummrich plant and, to a lesser degree, its Queeny plant in St. Louis were disposed at Site R. Disposal began in the northern portion of the site and expanded southward. Wastes contained phenols, aromatic nitro compounds, aromatic amines, aromatic nitroamines, chlorinated aromatic hydrocarbons, aromatic and aliphatic carboxylic acids and condensation products of these compounds.

In 1979, Monsanto completed the installation of a clay cover on Site R to cover waste, limit infiltration through the landfill, and prevent direct contact with fill material. The cover's thickness ranges from two feet to approximately eight feet. In 1985, Monsanto installed a 2,250-foot long

rock revetment along the east bank of the Mississippi River adjacent to Site R. The purpose of the stabilization project was to prevent further erosion of the riverbank and thereby minimize potential for the surficial release of waste material from the landfill. During the 1993 flood, Site R was flooded but the clay cap was not overtopped. No erosion of the river bank or cap resulted from this flood.

Access to Site R is restricted by fencing and is monitored plant personnel.

On February 13, 1992, the State of Illinois and Monsanto signed a consent decree entered in St. Clair County Circuit Court requiring further remedial investigations and feasibility studies to be conducted by Monsanto on Site R. The results of the Remedial Investigation/Feasibility Study were submitted to Illinois EPA in 1994. Solutia made a good faith offer to the IEPA to install an engineered cap and a leachate recovery system in 1997.

Parties who allegedly own, previously owned and/or operated Site R include:

- Cahokia Trust Properties
- Monsanto Company
- Solutia Inc
- Sauget and Company

Sediment samples collected from a drainage ditch around Site R showed VOC concentrations ranging from 0.002 to 0.035 ppm. SVOC concentrations in sediments ranged from 0.045 to 3.99 ppm. PCBs were detected at concentrations ranging from 0.08 to 1.5 ppm. Elevated levels of metals, particularly aluminum, iron and magnesium, were also detected. Sediment samples collected adjacent to the Mississippi River on the west side of Site R showed SVOC concentrations ranging from 0.001 to 7.7 ppm. PCBs were also detected at concentrations ranging from .00001 to .23 ppm.

Soil samples collected from Site R showed elevated levels of VOCs ranging from 0.15 to 5,800 ppm. SVOCs were found at levels ranging from 0.017 to 19,000 ppm. Pesticides were found at levels ranging from 0.011 to 99 ppm and PCBs were detected at levels ranging from 0.075 to 4,800 ppm. Elevated levels of arsenic, chromium, lead, nickel and mercury were also detected in Site R soils.

SVOC concentrations in leachate samples ranged from 0.6 to 12.3 ppb. Pesticide concentrations ranged from 0.5 to 3.0 ppb and PCBs were detected at 0.08 ppb. Dioxin/furan concentrations ranged from 0.0001 to 0.0014 ppm. Cyanide was also detected at 71 ppb.

Groundwater samples collected from wells on and immediately downgradient of Site R had VOCs concentrations of up to 38,136 ppb. SVOCs were detected in concentrations as high as 2,973,885 ppb. Historical groundwater data is presented in the Site Sampling Plan (SSP). The following constituents were detected at the following maximum concentrations:

Upper Hydrogeologic Unit (UHU)

VOCs (ppb)	
Acetone	69,000
Benzene	11,300
Bromoform	4,700
2-Butanone	3,100
Chlorobenzene	158,000
Chloroethane	10,000
Chloroform	1,600
1,1-Dichloroethane	4,700
1,2-Dichloroethane	16,500
1,1-Dichloroethene	2,800
trans-1,2-Dichloroethene	11,300
Methylene Chloride	22,400
4-methyl-2-pentanone	3,100
1,1,2,2-Tetrachloroethane	7,000
Tetrachloroethene	4,100
Toluene	6,000
1,1,1-Trichloroethane	3,800
Trichloroethene	4,610
Vinyl Chloride	24,500

SVOCs (ppb)	
Aniline	200,000
2-Chloroaniline	300,000
3-Chloroaniline	100,000
4-Chloroaniline	200,000
2-Nitroaniline	500,000
4-Nitroaniline	500,000
1,2-Dichlorobenzene	100,000
1,3-Dichlorobenzene	100,000
1,4-Dichlorobenzene	100,000
1,2,4-Trichlorobenzene	100,000
Nitrobenzene	100,000
2-Nitrochlorobenzene	3,400,000
3-Nitrochlorobenzene	730,000
4-Nitrochlorobenzene	1,500,000
Phenol	2,000,000
2-Chorophenol	540,000
4-Chlorophenol	210,000
2,4-Dichlorophenol	340,000

2,4,6-Trichlorophenol	100,000
3-Methylphenol	280,000
4-Methylphenol	47,000
2,4-Dimethylphenol	100,000
4-chloro-3-Methylphenol	100,000
4-Nitrophenol	500,000
Naphthalene	100,000
2-Chloronaphthalene	100,000
Benzoic Acid	50,800
Benzyl Alcohol	1,830
bis(2-chloroethoxy)methane	100,000
bis(2-ethylhexyl)phthalate	100,000
4-Nitrodiphenylamine	1,250

Middle Hydrogeologic Unit (MHU)

VOCs (ppb)	
Acetone	22,000
Benzene	9,980
Chlorobenzene	60,200
Chloroform	400
Chloromethane	2,500
1,1-Dichloroethane	1,200
1,2-Dichloroethane	9,200
1,1-Dichloroethene	700
trans-1,2-Dichloroethene	11,300
Ethylbenzene	2,500
Methylene Chloride	2,260
4-methyl-2-Pentanone	3,100
Tetrachloroethene	1,050
Toluene	3,000
1,1,1-Trichloroethane	950
Trichloroethene	500

SVOCs (ppb)	
Aniline	685,000
2-Chloroaniline	329,000
3-Chloroaniline	57,200
4-Chloroaniline	105,000
1,2-Dichlorobenzene	25,000
1,3-Dichlorobenzene	25,000
1,4-Dichlorobenzene	25,000
1,2,4-Trichlorobenzene	25,000
Nitrobenzene	25,000
2-Nitrochlorobenzene	463,000
3-Nitrochlorobenzene	460,000
4-Nitrochlorobenzene	185,000
Phenol	1,100,000

Vinyl Chloride	2,500
Xylenes	2,500

2-Chorophenol	160,000
4-Chlorophenol	67,000
2,4-Dichlorophenol	83,000
2,4,6-Trichlorophenol	25,000
Pentachlorophenol	125,000
3-Methylphenol	110,000
2,4-Dimethylphenol	25,000
4-Nitrophenol	125,000
Naphthalene	25,000
Chrysene	25,000
Fluoranthene	25,000
Pyrene	25,000
n-Nitrosodiphenylamine	25,000
bis(2-ethylhexyl)phthalate	25,000

Deep Hydrogeologic Unit (DHU)

VOCs (ppb)	
Acetone	500
Benzene	613
Chlorobenzene	7,380
Chloromethane	500
1,2-Dichloroethane	1,910
trans-1,2-Dichloropropene	720
Ethylbenzene	500
Methylene Chloride	1,790
4-methyl-2-pentanone	1,190
Tetrachloroethene	1,220
Toluene	2,070
Trichloroethene	500
Xylenes	962

SVOCs (ppb)	
Aniline	48,000
2-Chloroaniline	195,000
3-Chloroaniline	52,400
4-Chloroaniline	56,900
2-Nitroaniline	5,000
4-Nitroaniline	5,000
1,2-Dichlorobenzene	9,810
1,3-Dichlorobenzene	950
1,4-Dichlorobenzene	2,250
1,2,4-Trichlorobenzene	950
Nitrobenzene	1,010
2-Nitrochlorobenzene	219,000
3-Nitrochlorobenzene	30,900

4-Nitrochlorobenzene	115,000
Phenol	33,000
2-Chorophenol	8,500
4-Chlorophenol	18,000
2,4-Dichlorophenol	12,800
2,4,6-Trichlorophenol	3,030
Pentachlorophenol	2,500
2,4-Dimethylphenol	1,400
Benzo(a)pyrene	1,300
Benzo(k)fluoranthene	1,300
Chrysene	1,300
Fluoranthene	1,100
Naphthalene	800
Pyrene	950
4-Nitrodiphenylamine	5,000
n-Nitrosodiphenylamine	1,900
bis(2-chloroethyl)Ether	2,900
bis(2-cloroisopropyl)Ether	2,900
bis(2-ethylhexyl)Phthalate	5,000
di-n-butylphthalate	5,000
3,3'-Dichlorobenzidene	8,500
Hexachlorocyclopentadiene	10,000

2.2.5 Site S

Site S is located southwest of Site O. Allegedly, the property is or was owned by the Village of Sauget and the Resource Recovery Group. In the mid-1960s, solvent recovery began on site under Clayton Chemical, which is now owned by the Resource Recovery Group (RRG). The

waste solvents were steam-stripped resulting in still bottoms that were allegedly disposed of in a shallow, on-site excavation that is now designated Site S. Historical aerial photographs indicate that Site S was potentially a waste and/or drum disposal area. The northern portion of the site is grassed and its southern portion is covered with gravel and fenced.

Soil samples collected from Site S allegedly contain VOCs, SVOCs, PCBs and metals at concentrations up to:

VOCs (ppb)	
Ethylbenzene	450,000
4-Methyl-2-Pentanone	93,000
Toluene	990,000
1,1,1-Trichloroethane	12,000
Xylenes	620,000

SVOCs (ppb)	
Naphthalene	200,000
Bis(2-ethylhexyl)phthalate	20,000,000
Butyl Benzyl Phthalate	490,000
Di-n-butyl phthalate	1,500,000
Di-n-octyl phthalate	310,000

PCBs (ppb)	
Aroclor 1248	85,000
Aroclor 1254	69,000
Aroclor 1260	41,000

Metals (ppm)	
Copper	139
Lead	0.392
Mercury	3.5
Zinc	327

Additional information regarding environmental investigation at the Sites may be found in the Site Sampling Plan.

3.0 SITE ORGANIZATION AND COORDINATION

AMEC will have overall responsibility for the conduct of the activities associated with the ecological risk assessment conducted as part of the overall remedial investigation at the Sauget Area 2 sites, including management of the performance of the necessary subcontractors. The following section details the organizational structure for this project. Key personnel and their project responsibilities are listed in Table 2, Project Organization and Roles.

3.1 SITE HEALTH AND SAFETY OFFICER (SHSO) ROLE

The SHSO reports to the Project Manager and to the HSM for all aspects of the project and is the primary on-site contact for health and safety during field activities. The SHSO oversees the on-site execution of all field activities regarding health and safety procedures and subcontractor operations. The SHSO has the authority to stop all work if conditions are judged to be hazardous to on-site personnel or to the public. The SHSO also has the authority to temporarily suspend workers from the job site for serious violations of the HASP. Other specific responsibilities are as follows:

1. Require specific health control precautions prior to work area entry by AMEC personnel, subcontract personnel, or visitors including briefing of personnel on project and potential hazards, ensuring personnel have reviewed the HASP, on-site safety meetings, and reviewing planned emergency response procedures.

2. Require any AMEC or subcontract personnel to obtain immediate medical attention in the case of a work-related injury or illness.

3. May order work to cease and require evacuation of the work area by all personnel. Has the power to re-establish safe working conditions.

4. Control access to the site by visitors. Advise visitors of their responsibility before entry is allowed.

5. Ensure the correct field execution of the HASP including workplace and personnel monitoring.

6. Perform monitoring/sampling of site hazards for exposure and hazard evaluation.

7. Advise emergency response personnel in an emergency.

8. Coordinate and minimize the number of personnel and amount of equipment in the exclusion zone required for safe and effective site operations.

9. Calibrate all monitoring equipment that will be used on a daily basis and record the results in the daily calibration log.

10. Coordinate accident prevention plan by oversight of field activities and being aware of site operations.

11. The SHSO has the primary responsibility for investigation of injuries, illnesses, and "near misses", and completion of appropriate documentation.

3.2 PROJECT HEALTH AND SAFETY MANAGER (HSM) ROLE

The HSM is responsible for coordinating development and approval of HASP and coordinating the execution of health and safety procedures. The HSM assists and advises on resolution of health and safety issues when needed. The HSM, as necessary given the complexity of site work or potential risk associated with investigation of site materials, is responsible for on-site briefing of HASP and support staff prior to commencement of work.

3.3 PROJECT MANAGER ROLE

The Project Manager is the primary on-site contact for the Sauget Area 2 project. The Project Manager will coordinate access and security to the site and will advise AMEC personnel, subcontractors, and visitors of proper access and security procedures. The Project Manager is the primary contact for changes in scope of work and coordinating efforts between AMEC and Solutia relative to ecological sampling activities at the Sauget Area 2 sites. The Project Manager will issue the Notice to Proceed with on-site investigation after approval of work plan and HASP.

3.4 FIELD TEAM LEADER

The Field Team Leader is responsible for management of AMEC field effort and oversees the field efforts of AMEC personnel and subcontractors, and provides relevant technical and project performance information to the AMEC Project Manager. The Field Team Leader is responsible for coordination with the site-health and safety officer for implementation of the HASP.

3.5 FIELD PERSONNEL ROLE

AMEC and subcontract personnel who will be involved in the on-site execution of supervision, monitoring, testing, or sampling activities are responsible for:

1. Taking all reasonable precautions to prevent injury to themselves and to their fellow employees; being alert to potentially harmful situations.
2. Performing only those tasks that they believe they can do safely, and immediately reporting any accidents and/or unsafe conditions to the SHSO and/or the Site Manager.
3. Notifying the SHSO of any special medical conditions (*e.g.*, allergies, contact lenses, pregnancy, diabetes) and, if necessary, ensuring that all on-site personnel are aware of the condition.
4. Notifying the SHSO of any prescription and/or non-prescription medication, which the worker may be taking that might cause drowsiness, anxiety or other unfavorable side affects, or may interfere with administration of medical treatment in the unlikely occurrence of an emergency or potential overexposure.
5. Preventing spillage to the extent possible. In the event spillage occurs, contain the spillage, notify the SHSO, and clean up immediately using safe clean up measures as directed by the SHSO. Do not engage in spill containment or clean up if conditions are not safe.
6. Avoid splashing materials to the extent possible.
7. Practicing good housekeeping by keeping the work area neat, clean, and orderly to the extent possible.
8. Reporting all injuries and "near misses," no matter how minor.

9. Complying with the HASP and all health and safety recommendations, precautions, and proper use of the different levels of personal protective equipment as determined by the HASP and/or the SHSO.

4.0 SCOPE OF WORK/PLANNED SITE ACTIVITIES

4.1 SAMPLING OBJECTIVES

The objective of the sampling program is to collect sufficient information to determine if contaminants from the Sauget Area 2 sites have adversely impacted either the terrestrial or the aquatic ecosystems.

4.2 SAMPLING PROCEDURES

Aquatic and terrestrial sampling will be conducted in accordance with the procedures outlined in the Sauget Area 2 Site Sampling Plan.

4.2.1 Aquatic Ecological Activities

The following sections summarize the sampling activities associated with sample collection for the aquatic ecosystem portion of the risk assessment.

4.2.1.1 Surface Water Samples

Sixty surface water samples will be collected from the Mississippi River. Sampling will be conducted from a boat; thus, activities will be performed in accordance with OSHA requirements for "working over or near water" (29 CFR 1926.106) as described in Section 6.3.12 of this HASP in addition to OSHA HAZWOPER procedures (29 CFR 1910.120).

4.2.1.2 Sediment Sampling

Sixty sediment samples will be collected from the surface water locations within the Mississippi River; thus, activities will be performed in accordance with OSHA requirements for "working over or near water" (29 CFR 1926.106) as described in Section 6.3.12 of this HASP in addition to OSHA HAZWOPER procedures (29 CFR 1910.120).

4.2.1.3 Biota Sampling

Sixty fish samples will be collected from the surface water locations within the Mississippi River; thus, activities will be performed in accordance with OSHA requirements for "working over or near water" (29 CFR 1926.106) as described in Section 6.3.12 of this HASP in addition to OSHA HAZWOPER procedures (29 CFR 1910.120).

4.2.2 Terrestrial Ecological Activities

The following sections summarize the sampling activities associated with sample collection for the terrestrial ecosystem portion of the risk assessment.

4.2.2.1 Vegetation Sampling

A total of 24 samples of vegetation will be collected, by hand using stainless steel scissors or shears, at the location of soil/waste characterization samples that will be collected as part of the remedial investigation.

4.2.2.1 Macroinvertebrate Sampling

A total of 24 earthworm samples will be taken by digging with a decontaminated hand trowel or shovel, at soil/waste characterization sample locations collected as part of the remedial investigation.

5.0 WASTE CHARACTERIZATION

WASTE TYPES: (Check all that apply)

(X) Liquid (X) Sludge ( ) Unknown

(X) Solid ( ) Gas

WASTE CHARACTERISTICS: (Check all that apply)

(X) Corrosive (X) Flammable ( ) Radioactive

(X) Toxic (X) Volatile (X) Reactive

( ) Inert (X) Carcinogenic ( ) Unknown

HAZARDOUS MATERIALS SUMMARY: (Check all that apply)

Chemicals

( ) Acids (X) Metals (X) Phenols

( ) Caustics (X) Pesticides ( ) Paints

(X) Halogen (X) PCBs (X) Solvents

(X) Other: PAHs, anilines, dioxins, phthalates

Oils/Fuels

( ) Fuel oil ( ) Gasoline ( ) Diesel

( ) Other:

Sludges

( ) Metal sludges ( ) Oily sludges (X) Septic sludges

(X) Other: diatomaceous earth filter cake

Solids

( ) Asbestos (X) Landfill refuse ( ) Tailings

( ) Other:

6.0 HAZARD EVALUATION

Chemical, physical and biological hazards occur at the site. This section addresses these hazards as well as hazards specific to work tasks.

6.1 HAZARD ANALYSIS OF WORK TASKS

Surface Water/Sediment/Biota Collection

Potential Hazards: (Check all that apply to either existing conditions or are a result of site operations)

(X) Moving Machinery	( ) Projectiles	( ) Confined Space
() Heat Stress	(X) Physical Exertion	(X) Biological
(X) Cold Stress	( ) Noise (>85 dBA)	( ) Electrical (utilities)
(X) Heavy Equipment	(X) Vehicle Traffic	(X) Chemical Exposure
( ) Intrusive Activity	( ) Fire/Explosion	(X) Slips, trips and falls

(X) Other: Working Over or Near Water

Control or Protective Measures: (Check all that apply)

(X) Tailgate Meetings	(X) PPE	(X) Safe Work Practices
(X) Operator Training	( ) Site Control	(X) Decontamination
(X) Engineering Controls	(X) Other: USCG-approved life preserver/ring buoy; fire extinguishers	

INITIAL LEVEL OF PERSONAL PROTECTIVE EQUIPMENT (PPE) FOR ASSIGNED TASK:

Initial levels of PPE have been assigned for this work task per the potential for exposure. Levels may be upgraded or downgraded depending on monitoring data and site conditions, as determined by the protocol outlined in Section 11.0 Exposure Monitoring and deemed necessary or appropriate by the SHSO.

Level of protection:	( ) A	( ) C	() Modified D
	( ) B	(X) D	
Respirator:	( ) SCBA, Airline	( ) Fullface Resp	( ) 1/2 Face Resp.
(Level C or above)	( ) OV/HEPA Combo Cart.		( ) Other Cart.:
Protective clothing:	( ) Encapsulating Suit	( ) Tyvek	( ) PE Tyvek
	( ) Saranex	(X) Splash Suit	( ) Other
Head/eye/ear:	( ) Hard Hat	( ) Safety Glasses	( ) Goggles
	( ) Splash Shield	( ) Ear Plugs	( ) Other
Gloves: (Outer/Inner)	(X) Nitrile (outer)	( ) Neoprene	( ) PVC
	(X) Latex (inner)	( ) Vinyl	( ) Other: Leather
Footwear:	( ) Safety-toed Leather		( ) Chemical Overboots
	(X) Safety-toed Rubber		() Other:

Terrestrial Vegetation/Earthworm Collection

Potential Hazards: (Check all that apply to either existing conditions or are a result of site operations)

( ) Rotating Machinery	( ) Projectiles	( ) Confined Space
( ) Heat Stress	(X) Physical Exertion	(X) Biological
(X) Cold Stress	( ) Noise (>85 dBA)	( ) Electrical (utilities)
( ) Heavy Equipment	( ) Vehicle Traffic	(X) Chemical Exposure
(X) Intrusive Activity	( ) Fire/Explosion	(X) Slips, trips and falls

( ) Other:

Control or Protective Measures: (Check all that apply)

(X) Tailgate Meetings	(X) PPE	(X) Safe Work Practices
(X) Operator Training	(X) Site Control	(X) Decontamination
( ) Engineering Controls	( ) Other:	

INITIAL LEVEL OF PERSONAL PROTECTIVE EQUIPMENT FOR ASSIGNED TASK:

Initial levels of PPE have been assigned for this work task per the potential for exposure. Levels may be upgraded or downgraded depending on monitoring data and site conditions, as determined by the protocol outlined in Section 11.0 Exposure Monitoring and deemed necessary or appropriate by the SHSO.

Level of protection:	( ) A	(X) C	( ) Modified D
	( ) B	( ) D	
Respirator:	( ) SCBA, Airline	(X) Fullface Resp	( ) 1/2 Face Resp.
(Level C or above)	(X) OV/HEPA Combo Cart.		( ) Other Cart.:

Protective clothing: ( ) Encapsulating Suit (X) Tyvek ( ) PE Tyvek

( ) Saranex ( ) Splash Suit ( ) Other

Head/eye/ear: (X) Hard Hat (X) Safety Glasses ( ) Goggles

( ) Splash Shield ( ) Ear Plugs ( ) Other

Gloves: (Outer/Inner) (X) Nitrile (outer) ( ) Neoprene ( ) PVC

(X) Latex (inner) ( ) Vinyl ( ) Other: Leather

Footwear: (X) Safety-toed Leather (X) Chemical Overboots

( ) Safety-toed Rubber ( ) Other:

The hazard analysis for the aquatic sampling (including surface water, sediment, and biota sample collection) and terrestrial sampling (including vegetation and macroinvertebrate) is summarized in Table 3.

6.2 CHEMICAL HAZARDS

The list of chemicals of concern was previously presented as Table 1 and includes the maximum constituent concentrations in soil from previous studies in Sauget Area 2. Potential relevant exposure routes, permissible exposure limits (PELs) and toxicological data for chemicals detected at Sites O, P, Q, R, and S are presented in Table 4.

6.2.1 Aquatic Sampling Activities

The primary route of exposure during surface water and sediment sampling is dermal exposure; however, based on the large volume of water and sediment flow within the Mississippi River, contaminant concentrations are not expected to be a health concern. However, as a precaution, PPE requirements will be strictly enforced and personnel and sampling equipment will be properly decontaminated to eliminate any potential for incidental exposure. Any identified changes in chemical hazards from the original site characterization will necessitate the reassessment of this plan.

6.2.2 Terrestrial Sampling Activities

Prior investigatory work performed at the sites indicate contamination of soil with PCBs, metals, dioxins, and other organic compounds (e.g., chlorobenzene, anilines, chlorophenols, BTEX[1], PAHs, phthalates, chlorinated hydrocarbons). Therefore, potential routes of exposure for the collection of terrestrial biota samples are dermal contact and inhalation exposure to contaminated soils.

6.3 PHYSICAL HAZARDS

6.3.1 Heat Stress

Heat stress monitoring of workers and the environment will be initiated by the SHSO when the ambient temperature exceeds 70°F and workers are dressed out in modified Level "D" protective clothing or greater. To prevent heat stress, personnel monitoring (checking pulse rate or body temperature) will be used as well as scheduled work/rest periods. Workers determined by the SHSO to be displaying symptoms of advanced heat stress will be promptly referred to the designated local hospital.

Work/rest periods will be adjusted based on results of personnel monitoring. The warning symptoms of heat stress include fatigue; loss of strength; reduced accuracy; comprehension and retention; and reduced alertness and mental capacity. Heat stroke represents an advanced form of heat stress and is associated with physical symptoms of hot, dry skin, elevated body temperature (>104 °F), rapid pulse rate, and advanced symptoms of dizziness, nausea, and confusion which may lead to delirium, convulsions, coma, and possible death.

Personnel shall monitor themselves for heat stress as instructed by the SHSO. Self-monitoring shall include checking of pulse rate within two to five minutes into the work break. A pulse rate >110 beats per minute (bpm) shall require shortening of the next work period by 1/3 the time. A pulse rate recorded >110 bpm upon the next rest period shall warrant the same action, and so on. The taking of oral temperatures is not planned at this time, but may be enacted when pulse rate

[1] benzene, toluene, ethylbenzene, xylenes

measurements exceed 110 bpm. An oral temperature taken during this time period which is >99.6°F shall warrant shortening the next work period by 1/3 the time. A worker recording an oral temperature >100.6°F shall be prohibited from working until their temperature returns to 98.6°F. Heat stress monitoring will be coordinated and documented by the SHSO.

6.3.2 Cold Stress

Cold stress results from the effects of low ambient temperatures and wind velocity. Windchill is the cooling effect wind has on exposed skin. Wind velocity can be estimated using the following guidelines:

- 5 mph: light flag moves
- 10 mph: light flag fully extended
- 15 mph: raises newspaper sheet
- 20 mph: blowing/drifting snow

A windchill chart is provided as Table 5.

Three types of cold stress exist. The first is hypothermia, a lowering of the body's core temperature. It is caused by low (not necessarily freezing) temperatures and is aggravated by hunger, wetness, tiredness, and overexertion. Symptoms include shivering and abnormal behavior such as decreased efficiency, decreased level of communication, forgetfulness, repetitive behavior, poor motor skills, poor judgement, and lack of concern for one's usual physical needs. Prolonged hypothermia results in listlessness, sleepiness, weakness, an inability to walk resulting in repeated falling, stupor, unconsciousness, and ultimately, death.

Treatment of hypothermia is to prevent further heat loss. Victims should be moved to warm, dry areas out of the wind, cold, snow or rain. Wet or damp clothing should be removed and replaced with dry clothing. Cover the person's head as over 60% of body heat is lost through your head. Administer hot fluids if the victim is conscious and monitor the victim's temperature every 15 minutes to ensure proper body temperature (98.6°F).

Frost nip is a local cold injury where the skin is whitened and the victim feels a burning sensation. Move the person to a warm environment and warm the affected area.

Frostbite is caused by exposure to low temperatures and affects the extremities most often. The skin will appear cold, hard, and white. Blisters may form. The victim will have no pain sensation and may have mental confusion and impaired judgement. Ultimately, the person will die.

The aquatic and terrestrial sampling is schedule for October 2001; therefore, weather conditions prevalent during this time of year are conducive to producing cold stress. Cold stress monitoring of workers and the environment will be initiated by the SHSO when the ambient temperature is below 50°F. To prevent cold stress, personnel monitoring will be used as well as scheduled work/rest periods (Table 6). Workers determined by the SHSO to be displaying symptoms of advanced cold stress will be promptly referred to the designated local hospital.

6.3.3 Confined Space

Confined spaces will not be encountered during the ecological sampling activities.

6.3.4 Slip, Trip, And Fall Hazards

Slip, trip, and fall hazards will be minimized by the following housekeeping practices at all work sites:

- Loose or light material and debris will be stored in designated areas and/or containers.
- Tools, materials, extension cords, hoses, or debris will be located so as not to cause tripping or other hazards.
- Tools, materials, and equipment subject to displacement or falling will be adequately secured.
- All efforts should be made to work on level ground. Work areas may have to be altered to ensure a safe, reasonably level area. The sites will be examined before work commences to select the safest areas to set up.

- Any incidental spillage of fuel or fuel oil will be cleaned immediately with absorbent material, and covered with sand or soil to preclude slippage.

- Rubber-soled shoes will be required for those personnel working on the boat.

6.3.5 Noise

Noise levels greater than 85 dBA are not anticipated at the Sauget Area 2 sites.

6.3.6 Lifting

- Workers will use proper lifting techniques, lifting with the legs and not the back. Loads >50 lbs. require a second person or mechanical device.

- Whenever possible, mechanical devices such as drum dollies or hand trucks should be used to lift or move heavy loads.

6.3.7 Electrical Hazards

Electrical hazards may be encountered aboard the boat during sediment sampling for the ecological risk assessment. The boat is equipped with a fire extinguisher and motor kill switch.

6.3.8 Machinery Hazards

Machinery hazards may be encountered during sediment sampling when operating the dredge. The dredge will be operated only by persons qualified by training or experience to operate such equipment or machinery in accordance with OSHA regulations (29 CFR 1926.20).

6.3.9 Engulfment

Engulfment hazards will not be encountered during sampling for the ecological risk assessment.

6.3.10 Nuisance Dusts

Nuisance dusts are not an issue at these sites. Personnel collecting samples for the terrestrial aspect will be required to conduct sampling activities with Level C protection due to the presence of chemical contaminants in soil.

6.3.11 Fire/Explosion

The boat is equipped with a fire extinguisher and motor kill switch. Smoking will not be allowed on the boat during engine operation. Complete an Incident Report (Attachment B) within 24 hrs. for all work shutdowns.

6.3.12 Inclement Weather

Work shutdown conditions are as follows:

- Poor visibility.
- Precipitation severe enough to impair safe movement or travel.
- Lightning in the immediate area.
- Terrestrial sampling: steady winds greater than 40 mph. Aquatic sampling: winds greater than 20 mph.
- Other conditions as determined by the SHSO, Field Team Leader or HSM.
- Complete an Incident Report (Appendix B) within 24 hrs. for all work shutdowns.

6.3.13 Working Over or Near Water

In accordance with OSHA regulations for working over or near water (29 CFR 1926.106) and Coast Guard boating safety regulation, persons shall wear a US Coast Guard-approved life jacket or buoyant work vest at all times while working on the boat. Prior to and after each use, the vests or life preservers shall be inspected for defects which would alter their strength or buoyancy. Defective units shall not be used.

The boat to be used during the aquatic sampling shall be equipped with proper lights, air horn, first aid kit, fire extinguisher, cell phone, railings on three sides, motor kill switch, full spare gasoline tank, oars, installed oar locks, and an anchor. Coolers packed with liquid replenishment and food are allowed on board; however, no eating or drinking will occur during the handling/collection of hazardous materials or biota samples before personnel thoroughly was their hands.

The boat captain will be assigned prior to boating activities and all personnel working on board shall follow his/her orders. The boat captain shall be responsible for knowing and following US Coast Guard guidelines relative to boat traffic and/or other boating safety issues (www.uscgboating.org/reg/regfrcontents.asp). No smoking is allowed at any time in the boat or within 50 feet of it during refueling. All personnel on board shall be responsible for being cautious of coiled and frayed lines or lines under tension while in or near the boat. In addition, all personnel shall carefully lower lines into the water and never let a weighted line free fall. The depth to the bottom of the River shall be known before deploying equipment. Personnel shall remain seated in the boat unless deploying equipment. In this case, they shall keep their center of gravity as low as possible and as near as possible to the center of the boat. Refer to the control measures listed in Table 3 (Activity Hazard Analysis) for boating operations.

For persons working on shore, ring buoys with at least 90 feet of line shall be provided and readily available for emergency rescue operations. Distances between ring buoys shall not exceed 200 feet. At least one lifesaving skiff shall be immediately available at locations where employees are working over or adjacent to water.

6.4 BIOLOGICAL HAZARDS

Biological hazards at the Sauget sites include potential exposure to poison ivy, tetanus, mosquitoes, rodents, and ticks.

To reduce the chances of dermatitis caused by poison ivy, personnel should wear long sleeves and long pants. Any exposed skin should be wiped downed several times a day with baby wipes or apply a blocking product (e.g., "Ivy Block"). Long sleeves and pants will also help to prevent mosquito bites.

Many of the study areas are former landfills and may have protruding objects. Therefore, field personnel will be required to have a current tetanus shot prior to job initiation.

Contact with rodents should be avoided during field work.

Ticks are vectors for a number of diseases such as Rocky Mountain spotted fever or Lymes disease. Lymes disease can result in permanent damage to the nervous system and joints. Ticks are found throughout the United States.

Ticks are tiny and are found in brush, woods, and tall grass. May and June are the worst months for potential exposure, but the ticks are active in all months when the temperature exceeds 45°F. Preventative measures to avoid exposure to ticks include the following:

- Field personnel should wear long pants and long-sleeved shirts. It is recommended that pant cuffs be placed inside the work boots. Shirts should be tucked in. The use of light colored clothing may make it easier to spot ticks.

- Tick repellent should be applied to clothing and skin. Permethrin-containing repellent may be sprayed near the openings on work clothes (pant bottoms and waistband); permethrins should not be used on skin. On skin, DEET (N-diethyl-metatoluamide)-containing repellent (< 33% DEET) should be applied. DEET should not be used on the face.

- All personnel should check for the presence of ticks or tick bites every day. Skin and hair should be checked for ticks. Many people get a spot on their skin in three to 30 days following a tick bite; the spot looks like a small red bullseye that is spreading out.

- If a tick is observed on the skin, it should be removed immediately: hold a tweezer on the tick as close to the skin as possible, and pull. Kill the tick and retain for examination by a physician. Performance of a blood test is appropriate following exposure to a tick bite. The Lyme blood test may not indicate positive results until two weeks or more after a tick bite. (Note: Ticks should not be killed with or stored in any liquid as this makes testing for Lymes disease impossible).

7.0 HAZARD COMMUNICATION AND TRAINING ASSIGNMENTS

Potential hazards shall be communicated during the sample handling process. Samples shall be identified as to their potential hazard and packaged to prevent spillage or breakage in accordance with Department of Transportation (DOT) regulations. Any unusual sample conditions shall be noted. In addition, the laboratory receiving the samples shall be advised of the potential contaminants present and associated hazard level (either through phone conversation or documentation with the samples).

Hazards associated with the chemicals used during the sample preservation and equipment decontamination (including hydrochloric acid, nitric acid, and methanol) shall be communicated to the field personnel.

7.1 HAZARD COMMUNICATION TRAINING

All site personnel shall be trained in the hazards associated with this project prior to commencement of work activities. Material safety data sheets (MSDSs) for all materials on-site shall be kept on-site and available to all workers at all times (refer to Attachment C). AMEC shall be responsible for acquiring all pertinent MSDSs and the SHSO shall be responsible for upkeep of the MSDS file at the job site.

7.2 HAZARDOUS WASTE WORKER TRAINING

All site staff will have completed the OSHA 40-hour Hazardous Waste Operations Training, 24-hour on-site supervised training, and appropriate annual updates [29 CFR 1910.120(e)]. In addition, Site Supervisors will have completed OSHA 8-hr Supervisory Training. First Aid and cardiopulmonary resuscitation (CPR) trained personnel shall have Bloodborne Pathogen Training as mandated by OSHA 29 CFR 1910.1030. Occasional site workers that will not receive exposures exceeding permissible exposure limits require, at a minimum, 24 hours of OSHA Hazardous Waste Operations training and one day on-site training and supervision. Documentation that training assignments have been met will be required prior to site entry for AMEC personnel and their subcontractors.

7.3 First Aid/CPR Training

A minimum of one on-site person will be trained in basic first aid and CPR as administered by American Red Cross or National Safety Council. A first aid kit will be available at all times that work is in progress and will be stored in a field vehicle and the boat. All on-site personnel will be advised of its location.

7.4 Orientation Training And Daily Safety Meetings

An on-site orientation session will be required for all site personnel, including subcontractors and visitors, prior to commencement of work and will include the following:

- Overview of planned activities, procedures and monitoring techniques
- Health effects and hazards of the chemicals identified or suspected to be on-site
- Personnel protection, including:
    - Use, care and fitting of PPE
    - Necessity for personnel protection, its effectiveness and limitations
- Decontamination procedures
- Prohibitions in work areas
    - Site layout
    - Standard safe work practices
- Emergency procedures, including:
    - Emergency contacts
    - Instructions for implementing the emergency plan
    - Map of site layout

A daily safety meeting will be conducted and documented at the beginning of each workday or work shift. Health and safety considerations for the day's activities will be discussed and protective equipment necessary will be outlined. Problems related to personal protection, inclement weather, or the interpretation of newly available environmental monitoring data will be topics typically covered during these briefings. These meetings will be documented in the field notebook.

8.0 MEDICAL SURVEILLANCE REQUIREMENTS

All AMEC personnel and their subcontractors working on-site shall participate in a medical surveillance program which is consistent with the requirements of 29 CFR 1910.120 for hazardous waste site operations. The purpose of the program is to assess and monitor employee health prior to employment, during the course of, and at the termination of employment. Subcontractor employees are required to have been certified by their employer that they are in compliance with this requirement and provide proof to AMEC prior to job initiation.

8.1 COMPREHENSIVE PHYSICAL EXAM

The program consists of scheduled baseline exams, follow-ups, termination exams, and other exams as needed. The basic exam protocol is as follows:

1. An occupational and medical history;
2. A physical exam;
3. Whole body count;
4. Laboratory tests including blood chemistry, CBC, reticulocyte count (heavy metals specific for chromium, lead and mercury, and chlorinated hydrocarbons specific for PCBs);
5. Urinalysis;
6. EKG;
7. Stool guaiac;
8. Spirometry with chest x-ray;
9. Audiometric testing;
10. Vision tests; and
11. Respirator quantitative fit test.

Copies of medical clearances, training records, and respirator qualification cards shall be made available to, and copies kept by, the SHSO prior to each individual on-site worker beginning site duties. Medical records and exposure records clearance for each on-site worker will be maintained in their individual personnel file at their respective office of employment.

8.2 EMERGENCY MEDICAL TREATMENT

Personnel who exhibit signs and symptoms of chemical or heat overexposure, or have been injured on the job, might also seek medical services. See also the Emergency Response (Section 17) for specific information regarding emergency services and logs, reports, and record keeping. Subcontractors should provide internal Workers' Compensation information to the SHSO or Project Health and Safety Manager during the pre-work meeting, for emergency use.

9.0 COMMUNICATIONS

The "buddy system" will be enforced for field activities involving potential exposure to hazardous or toxic materials. Each person will observe their partner for symptoms of chemical overexposure or cold/heat stress and provide emergency assistance when warranted. An audible emergency signaling device shall be maintained in the field.

The following emergency signals shall be used:

• Thumbs up	OK; understand
• Thumbs down	No; negative
• Grasping buddy's wrist	Leave site now
• Hands on top of head	Need assistance
• (Air Horn) One long blast	Evacuate site
• (Air Horn) Two short blasts	Return to site

10.0 SANITATION/ILLUMINATION

Washing facilities (potable water, soap, and disposable towels) shall be available on-site for use by field personnel. Local restroom facilities will be identified by the Site Superintendent/SHSO for use by project personnel. Work will only be conducted during daylight hours.

11.0 EXPOSURE MONITORING

Available site information does not warrant the anticipation of Immediately Dangerous to Life and Health (IDLH) concentrations. The levels of protection outlined in Section 6.1 (Hazard Analysis of Workers Tasks) have been assigned in accordance with known concentrations of materials. Level D has been assigned for aquatic sampling due to the potential for dermal contact with contaminated sediments. Level C has been initially assigned for terrestrial sampling due to the potential for inhalation and dermal contact with contaminated soils.

11.1 TYPE AND FREQUENCY OF AIR MONITORING

Some of the compounds detected in soil exhibit vaporous qualities; therefore, air monitoring will be conducted according to Table 3.

11.2 MONITORING INSTRUMENTS

The SHSO will maintain equipment SOPs (Attachment D) onsite that specify calibration, general use, and troubleshooting procedures. The photoionization detector will be field calibrated on a daily basis according to the manufacturers instructions, and will be recorded on the calibration log (Attachment D).

11.3 PERSONAL SAMPLING

It is not necessary to selectively monitor site workers for specific parameters with laboratory confirmation for this project.

11.4 ACTION LEVELS

Action levels shall be established for upgrading/downgrading PPE, work stoppages, and evacuation. The decision to upgrade/downgrade the level of PPE must be based upon instrument readings measured in the breathing zone of site personnel and comparison of the results to the information contained in Table 7. Readings shall be recorded in the field notebook.

12.0 WORK ZONE DELINEATION

Based on site characterization information previously obtained from the project area, hazardous materials are expected to be encountered during proposed data collection activities. A centralized area at each site (i.e., O, P, Q, R, S) will be established for equipment decontamination and disposal of used PPE. The establishment of working zones is described below.

12.1 EXCLUSION ZONE

The exclusion zone is the area where the potential for worker exposure with contaminated materials is greatest. All personnel entering this zone will be required to have the prescribed level of protective clothing as determined by the SHSO and the HSM. Access control points and delineation of entry/egress stations will be indicated on the Site Work Zone Map developed at the time of the on-site briefing conducted by the SHSO.

Prohibited items or conduct in the exclusion zone shall include, but not be limited to, the following:

1. Eating, drinking, smoking or any other activity which could lead to the possibility of hand to mouth exposure of contaminants;

2. Personal articles, e.g., watches, rings, bracelets;

3. Working when ill, or taking prescription medication without HSM prior approval; and

4. Access to the exclusion zone by any individual not having the required health and safety training in accordance with 29 CFR 1910.120.

12.2 CONTAMINATION REDUCTION ZONE

The contamination reduction zone is established outside the exclusion zone and provides a transition between the exclusion zone (potentially contaminated zone) and the support zone (clean zone). It serves as a buffer to reduce the possibility of the support zone becoming contaminated and provides additional assurance that the physical transfer of contaminating substances on personnel and equipment or in the air is limited through a combination of decontamination, distance between exclusion and support zones, air dilution, zone restrictions, and work functions.

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Items and/or conduct prohibited in the exclusion zone are also prohibited in the contamination reduction zone. Decontamination stations will be established at the boundary between the exclusion and the contamination reduction zones, for the sole purpose of personnel and equipment decontamination.

12.3 SUPPORT ZONE

The support zone shall be marked and protected against contamination from the work site. This will be accomplished by placing the support zone upwind of prevailing winds and providing adequate distance between the exclusion zone and the support zone. Primary functions of the support zone are:

1. The entry area for personnel, material, and equipment.
2. The exit area for decontaminated personnel, material, and equipment.
3. A storage area for clean safety and work equipment.
4. An area for rest breaks and the consumption of food and beverage, after washing of hands and face.

13.0 SAFE WORK PRACTICES AND EQUIPMENT

Employees and visitors will be required to follow and maintain good hygiene/work practices that include:

- Unauthorized personnel are not allowed on-site without prior briefing by the SHSO.
- Work groups will always consist of at least two team members; this may include AMEC and subcontract personnel.
- Smoking, eating, drinking, chewing gum or tobacco, taking medication, and applying cosmetics will not be permitted within any restricted areas or exclusion zone.
- Wearing of contact lenses in contaminated atmospheres is prohibited.
- Personnel under the obvious influence of alcohol or controlled substances are not allowed on-site. Those persons taken medication must notify the SHSO.
- Hands and face will be thoroughly washed before breaks or any hand to mouth activity such as eating, drinking, smoking, use of chewing tobacco, or application of cosmetics.
- Personnel will appropriately discard and replace any damaged or heavily soiled protective clothing. Discarded PPE will be drummed at the end of each day.
- Personnel should notify the on-site health and safety coordinator of any defective monitoring, emergency, or other safety equipment.
- A supply of potable water, electrolyte replacement solutions, and sufficient lighting will be maintained on-site and accessible to personnel. The location of local sanitary facilities will be identified by the SHSO.
- All unsafe conditions shall be made safe immediately. All unsafe conditions not in the scope of the project shall be reported to the PM and the condition corrected.
- All site personnel will familiarize themselves with these and the emergency procedures during daily tailgate and prework safety meetings.

- Following safe work practices reduces the likelihood of an accident, illness, or injury during field activities.
- Loose-fitting clothing or loose long hair are prohibited near moving machinery.
- Workers who are passengers or drivers of vehicles (both offsite and onsite) will wear their seat belts any time the vehicle is in motion.
- Do not fuel engines while vehicle is running.
- Install adequate onsite roads, signs, lights, and devices.
- Store tools in clean, secure areas so they will not be damaged, lost, or stolen.

The HSM shall determine the types of safety and emergency equipment needed for the various tasks at the site as well as the necessary locations for the equipment (boat, field vehicle, etc.). This equipment may include, but not be limited to, a fire extinguisher, emergency eyewash, first aid kits, and oral thermometer (for measuring temperature associated with potential heat stress).

14.0 PERSONAL PROTECTIVE EQUIPMENT

Personal protective equipment (PPE) selection will be based on three criteria: U.S. Environmental Protection Agency (USEPA) Levels of Protection as defined in Standard Operating Safety Guides, the Occupational Health and Safety Guidance Manual for Hazardous Waste Site Activities, and the provisions of OSHA 29 CFR 1910 and 1926. In the event of conflicting requirements, the most stringent level shall apply. The anticipated level of protection for aquatic sampling activities is Level D and Level C for terrestrial sampling activities. These requirements are subject to change based on contaminant monitoring, visual observations, or changes in the work or site conditions. For tasks not covered in the HASP, personnel shall wear a level of PPE which is deemed necessary by the SHSO.

PPE will be selected, upgraded, or downgraded by the SHSO, based upon the specific task and the results of environmental monitoring and will be noted in the field notebook (Table 8). Employees will be provided with the appropriate protective equipment including chemical-resistant outer gloves, hard hats, safety shoes or heavy rubber shoes, and protective eye wear for field use. Rain gear will be available in the case of inclement weather. Employees will inspect and maintain all PPE as appropriate. All PPE for site activities is based upon the potential for contact with site-specific contaminants and may involve re-assessment necessitating upgrade or downgrade in PPE. The following outlines the requirements for each level of protection.

Four levels of protection and associated PPE are identified by OSHA for hazardous waste site work: Levels D, C, B, and A. Modified Level D (Level C, without a respirator) may also be used. Level D provides the lowest level of protection. Protection increases progressively through Levels C, B, and A. Level D is implemented unless a higher level of protection is specifically indicated. It is prudent to have available on-site, sufficient PPE to upgrade to the next higher level (*i.e.*, if level D is specified, Level C PPE should be available in case unforeseen hazards are encountered).

14.1 LEVEL D PPE

Most initial field activities require Level D if direct handling of contaminated materials is not anticipated or expected. If field conditions indicate a higher level of contamination than anticipated, a higher level of protection will be directed. If higher levels are necessary, they will be selected by the SHSO and/or HSM based on the contaminant concentrations. Level D requires

minimal decontamination, including thorough washing of hands, arms, and face and boots, followed with safe work practices. Level D PPE includes the following:

1. Work shirt and work pants.
2. Safety toe work boots.
3. Hard hat (as necessary for required activity).
4. Approved safety glasses (with side shields) (as necessary for required activity)

14.2 MODIFIED LEVEL D PPE

Modified Level D includes:

1. Same as above but including the following:
2. Tyvek suits, Saranex or PE Tyvek may be required in wet conditions.
3. Safety toe, nitrile rubber high top boots (taped to Tyvek) or boot covers.
4. Inner vinyl gloves and outer nitrile rubber gloves (taped to Tyvek).
5. Face shield may be required under certain conditions (*i.e.*, splash protection).

14.3 LEVEL C PPE

The criteria for selection of Level C includes:

1. Oxygen level is greater than 19.5%.
2. Total unknown organic vapor levels at a sustained level of 5 ppm but below 50 ppm above background.
3. Doubt exists about the air quality, therefore preventing the use of Level D PPE.
4. Level A or B PPE is not indicated.

Level C PPE includes:

1. PPE same as Modified Level D.

2. Full-face air-purifying respirator with combination organic vapor/HEPA particulate (GMC-H) cartridges (or as otherwise designated); refer to AMEC respiratory protection program: SOP HSP-4.

14.4 LEVEL B PPE

The criteria for selection of Level B PPE includes:

1. Atmosphere with chemical concentrations considered IDLH.

2. Atmospheres exceeding the limits of the protection afforded by a full-face, air-purifying respirator.

3. Atmospheres containing substances with poor warning properties, substances for which air-purifying cartridges do not exist or have low removal efficiency.

4. Atmospheres containing less than 19.5% oxygen.

5. Conditions are such that small exposed areas about the head and neck will not be contacted by hazardous substances.

Level B PPE includes:

1. Same PPE as Level C, but with supplied air respirator (SCBA or airline with escape bottle); refer to AMEC respiratory protection program: SOP HSP-4.

All personnel will be required to be in appropriate PPE before entering the work zone. All disposable supplies shall be removed and disposed of in containers provided in the specified decontamination area. Personnel will be required to remove excess mud or other debris from boots and equipment before leaving the decontamination area and entering field vehicle.

15.0 PROTECTIVE CLOTHING DONNING/DOFFING PROCEDURE

The purpose of the protective clothing donning/doffing procedures is to ensure that on-site personnel are instructed in the proper way to don/doff protective clothing. Failure to adhere to these procedures may result in the protective clothing being ineffective against a potential contaminant. The following donning/doffing procedures are given as a guide and may be altered by the SHSO if improvements can be made to the procedure and these changes are warranted in the field. In addition, some articles of protective clothing/equipment detailed below may not be necessary for the particular site task.

15.1 PROTECTIVE CLOTHING DONNING PROCEDURE

1. Remove personal outer clothing in support zone and dress out in supplied clothing and rubber boots. Inspect clothing and respiratory equipment before donning.

2. Don Tyvek or splash suit, inner gloves, and outer rubber gloves as specified for the assigned task in Section 6.1. Outer chemical resistant gloves shall be sealed to the Tyvek suit using duct tape.

3. For the terrestrial sampling, don personal full-face respirator and cartridges by tightening straps. Test for fitness by completely covering cartridges and breathing in. No air should come into the respirator.

15.2 PROTECTIVE CLOTHING DOFFING PROCEDURES

1. Wash/rinse (if necessary) excess mud or other debris from outer boots, gloves, and clothing prior to leaving the exclusion zone.

2. Remove outer tape and outer layer of clothing (Tyvek suites, boots, gloves), placing disposable PPE in designated drums and reusable PPE in designated locations for donning during re-entry.

3. Remove respirator and discard cartridges prior to removing inner vinyl gloves. Remove inner gloves and move from the contamination reduction zone. Thoroughly decontaminate respirator with the manufacturer's approved detergent then store the

respirator in a protective plastic bag, which will be kept with worker or at appropriate storage facility in the support on-site at all times when not in use. Cartridges must be removed and disposed of properly at a hazardous material; do not store with the respirator.

4. Thoroughly wash hands and face with soap and water prior to eating, drinking, smoking, other hand-to-mouth contact activity.

respirator in a protective plastic bag, which will be kept with worker or at appropriate storage facility in the support on-site at all times when not in use. Cartridges must be removed and disposed of properly at a hazardous material; do not store with the respirator.

4. Thoroughly wash hands and face with soap and water prior to eating, drinking, smoking, other hand-to-mouth contact activity.

16.0 DECONTAMINATION AND DISPOSAL

The purpose of decontamination is to prevent contaminants that may be present on protective clothing and equipment from coming into contact with personnel as they remove contaminated PPE. Also, decontamination protects workers from hazardous substances that may contaminate and eventually permeate the PPE used on-site; it protects personnel by minimizing the transfer of harmful materials into clean areas. Decontamination consists of physically removing contaminants or changing their chemical nature to innocuous substances. Combining decontamination with the correct sequential method of removing PPE will prevent exposure to personnel leaving the work areas as well as off-site migration of contaminants.

Generally, decontamination is accomplished by starting at the first station with the most heavily contaminated item and progressing to the last station with the least contaminated item. Each item of protective equipment requires a separate station which is marked accordingly.

The purpose of equipment decontamination is to prevent exposure to personnel during loading, transporting, and unloading at another site. It is also to prevent off-site migration of contaminants from one site to another or during transportation of the equipment.

Waste materials shall be disposed of to prevent the spread of contamination, creating a sanitary hazard or causing litter to be left on site. Disposal of potentially contaminated materials shall involve the bagging or drumming of the materials as necessary and segregation for special disposal. In addition, all non-contaminated materials shall be collected and bagged for proper disposal as normal domestic waste.

16.1 PERSONNEL DECONTAMINATION

Removal of loose mud or other substrate from personnel will be performed in the decontamination zone. Personnel will remove any disposable PPE and dispose of it in provided containers before leaving the contamination reduction zone. Personnel shall thoroughly wash hands and face before leaving the area. A shower is not anticipated for use on-site at this time for personal decontamination. PPE and decontamination waste water will be collected and appropriately disposed.

16.2 EQUIPMENT DECONTAMINATION

All equipment (*e.g.*, hand tools, sampling equipment) shall be decontaminated at the job site prior to removal from the decontamination zone. Equipment decontamination will be conducted in accordance with USEPA protocols.

17.0 EMERGENCY RESPONSE

The purpose of this section is to safeguard human health and the environment in the event of an emergency. This section also addresses the emergency actions to be taken in response to an emergency. The responsibility of the regular day-to-day implementation of this information primarily lies with the SHSO. During an actual response situation, the SHSO will serve as the Emergency Coordinator.

17.1 PRE-EMERGENCY PLANNING

The SHSO will perform the following pre-emergency tasks before starting field activities and will coordinate emergency response with all appropriate personnel:

1. Locate nearest cellular telephone.
2. Confirm emergency telephone numbers (these numbers will be available on-site in this document for reference).
3. Inform Solutia and the local municipality or jurisdiction of the nature of the project hazards and potential emergencies.
4. Review and revise emergency response plan in the event of a failure of the plan in an emergency due to changes in site conditions, changes in the scope of work, or personnel availability.
5. Inventory and inspect on-site emergency equipment and supplies.

17.2 LINES OF AUTHORITY

The SHSO has primary responsibility for expediting Response Operations on-site to include reporting to the Ecological Project Manager and Solutia and correcting as conditions allow, and responding to and correcting emergency situations. The SHSO has the authority to stop any site activities posing an immediate health and safety hazard to site personnel and the public. Possible actions may involve notification of the HSM, the Ecological Project Manager, and Solutia, ensure

corrective measures are implemented; notification of appropriate authorities; and follow-up reporting.

17.3 EMERGENCY PREVENTION AND RECOGNITION

Prevention of emergencies will be aided by the effective implementation of the HASP, personnel awareness, contingency planning, and the briefing held with personnel at the beginning and during the execution of the field activities. Site security will be maintained during working hours by the SHSO. Communication is available by means of a cellular telephone or local phone service at neighboring commercial/retail establishments.

17.4 NOTIFICATION

In the event of an emergency, a verbal instruction or site alarm will be sounded to:

1. Notify all on-site personnel. If appropriate, direct all personnel in the affected area to evacuate and assemble upwind in a designated safe area.
2. Stop work activities and shut down all combustion equipment
3. Establish the safety of all personnel, direct the administration of first aid and provide emergency equipment as appropriate.
4. Lower noise levels to facilitate communications.
5. Begin emergency procedures.
6. Notify on-site emergency response personnel about the emergency.
7. Prohibit outside personnel from entering the evacuated area until the Fire Department arrives.

Accident reporting should be performed in accordance with AMEC SOP HSP-1.

17.5 EVACUATION ROUTES AND PROCEDURES

In the event of an emergency that requires an evacuation of the site, an alarm will be sounded or verbal instruction given by the SHSO to evacuate the area. Personnel will exit the area to the specified meeting point. At this point, the SHSO will account for all personnel, ascertain information about the emergency and advise further instructions to the on-site personnel. The SHSO will also advise responding off-site emergency personnel of the situation, if necessary. In all situations that require evacuation, personnel shall not re-enter the work area until the conditions causing the emergency have been corrected, the hazard reassessed, the HASP has been revised if necessary and reviewed with on-site personnel, and instructions given for authorized re-entry.

17.6 EMERGENCY MEDICAL TREATMENT AND FIRST AID

In the event of an emergency involving personal injury or illness, first aid should be rendered by a trained person and emergency medical services summoned as identified in Section 17.9. Personnel with injury or illness will be decontaminated to the extent possible without further injury. Life saving and first aid procedures take priority over personnel decontamination efforts. The SHSO will have final authority on the decision to require additional professional medical services (*i.e.*, paramedics, hospital visit, etc.) for any illness or injury. In the case that emergency assistance is needed, the SHSO will immediately:

- Notify emergency response (911 or other numbers listed in the emergency contacts) and give the appropriate patient information and their location
- Assist the injured party as deemed appropriate
- Designate someone to accompany the injured party to the hospital

Accident reporting shall be performed in accordance with AMEC SOP HSP-1.

In the event that an exposure to toxic or hazardous materials occurs the first responder to the victim shall, as appropriate:

- Wash/rinse the effected area thoroughly with copious amounts of soap and water, then provide appropriate medical attention. If eyes are involved, they should be rinsed for at least 15 minutes using eyewash.

- Move the exposed person to fresh air and provide medical attention.
- Provide medical attention for ingestion and puncture wound or laceration.

17.7 FIRE OR EXPLOSION

In the event of a fire or explosion; the local fire department will be summoned immediately (Section 17.9). Upon their arrival, the SHSO will advise the fire commander of the situation. If it is safe to do so, on-site personnel may use fire fighting equipment available to control and/or extinguish the fire and remove or isolate flammable or other hazardous materials which contribute to the fire or inhibit control of the fire.

17.8 SPILL OR LEAK

In the event of a spill or leak (regardless of quantity), on-site personnel will:

1. Inform the HSM and Solutia PM immediately.
2. Locate the source and stop the spillage if it can be done safely.
3. Begin containment and recovery of spilled material.

17.9 EMERGENCY CONTACTS

Emergency resources are as follows:

Local Police Department:	Sauget	618-322-6507/6997
	Cahokia	618-337-5080
Local Fire Department:	Sauget	618-332-6700
	Cahokia	618-337-5080
Local Hospital:	St. Mary's Hospital	
	100 North 8th St.	
	East St. Louis, IL	618-274-1900
Poison Control Center:		1-800-942-5969
USEPA National Response Center:		1-800-424-8802

AMEC Contacts

Ecological Project Manager:	Chuck Harman	732-302-9500, ext. 127
Project Health and Safety Manager:	Jeffrey Tasca	732-302-9500, ext. 106
Corporate Health and Safety Manager:	Denise Daggett, CIH	858-458-9044

Sauget Area 2 Group Contact

Project Manager:	Steven Smith	314-674-4922

17.9.1 Route To Nearest Hospital

Turn right on Mobile Street then turn slight right onto Monsanto Ave. Continue on Monsanto Ave. for 0.3 mile. Turn left onto IL-3 for 0.9 mile. Take the I-70E/I-64E/I-55N ramp towards IL-3N/Chicago/Indianapolis and stay right at the fork in the ramp. Merge onto South 4th St. Turn right onto East Broadway/IL-15 and continue for 0.3 mile. Turn left on North 8th St, the hospital is #100. The route to the hospital is also indicated on the map contained in Attachment E.

17.9.2 Emergency Equipment

It shall be the responsibility of the AMEC SHSO to maintain the site emergency equipment in good working order. Equipment shall be readily available, clearly identified by signs and/or labels, and workers shall be trained in its use by the SHSO. Equipment maintained by the SHSO in the field vehicle and boat shall include a first aid kit, and an emergency eye wash kit. The boat is equipped with a fire extinguisher, signaling devices (e.g., air horn, lights) and a cell phone.

18.0 STANDARD PROCEDURE FOR REPORTING EMERGENCIES

The following information should also be provided to the Corporate Health and Safety and Personnel offices in the event of an emergency.

1. Name of person making call.
2. Telephone number at location of person making call.
3. Name of person(s) exposed or injured.
4. Information provided in medical data sheet (Attachment A).
5. Nature of emergency.
6. Actions already taken.

Specific accident reporting procedures are contained in AMEC SOP HSP-1.

18.1 FOLLOW-UP AND DOCUMENTATION

Before normal activities are resumed, on-site personnel must be prepared and equipped to handle another potential emergency. The follow-up activities should be completed:

1. Notify appropriate government agencies as required (Reminder: OSHA must be notified within eight hours if there have been any fatalities or three or more hospitalizations).
2. Restock all equipment and supplies.
3. Review and revise all aspects of the HASP as necessary to address future emergencies of this type and new site conditions.

Investigation and documentation of any emergency response shall be initiated by the Field Team Leader with assistance by the SHSO. This is important in all cases, but especially so when the incident has resulted in personal injury, property damage, or environmental impact. The documentation will be a written report and will be:

1. Accurate: All information must be recorded objectively.

2. Authentic: Each person making an entry must sign and date that entry. Nothing is to be removed or erased. If details are changed or revised, the person making the change should strike out the old material and initial and date the change.

3. Titles and names of personnel involved.

4. Actions taken, decisions made, orders given, to whom, by whom, when, what, where, and how as appropriate.

5. Summary of data available (air monitoring, chemical concentrations, etc.).

6. Possible exposure of personnel.

7. Copies of all the Supervisor's Accident Investigation Reports and Employer's Report of Occupational Injury or Illness.

19.0 PROJECT DOCUMENTATION

Site investigation information shall be documented with the use of the reporting forms and logs shown in this section. The logs and records shall be the ultimate responsibility of the SHSO and shall be maintained on-site for review. However, all personnel shall be individually responsible for completion of the information required by the logs. The health and safety logs and records, included as attachments and summarized below, shall be kept on record for a period of no less than 10 years by AMEC.

Plan Approval Signature Sheet	Section 20.0
Site Map	(Figure 2, URS Figure 1)
Daily Project Log	Field Notebook
Site Monitoring Data	Field Notebook
Training/Safety Briefing Log	Field Notebook
Accident/Illnesses Reports	SOP HSP-1 Forms

Record keeping for health and safety purposes will be in accordance with the OSHA requirements published in 29 CFR 1910, including medical examination reports, accident/injury reports, exposure reports, and records/certificates of the general and specialized training courses completed by each person employed on-site.

20.0 PLAN APPROVAL

This HASP has been written for the use of AMEC employees and subcontractors on this project. AMEC claims no responsibility for its use by others. The HASP is written for the specific site conditions, purposes, dates and personnel specified and must be amended if these conditions change.

Jeffrey Tasca

Project Health and Safety Manager

Charles Harman, PWS

Project Manager

Site Health and Safety Officer

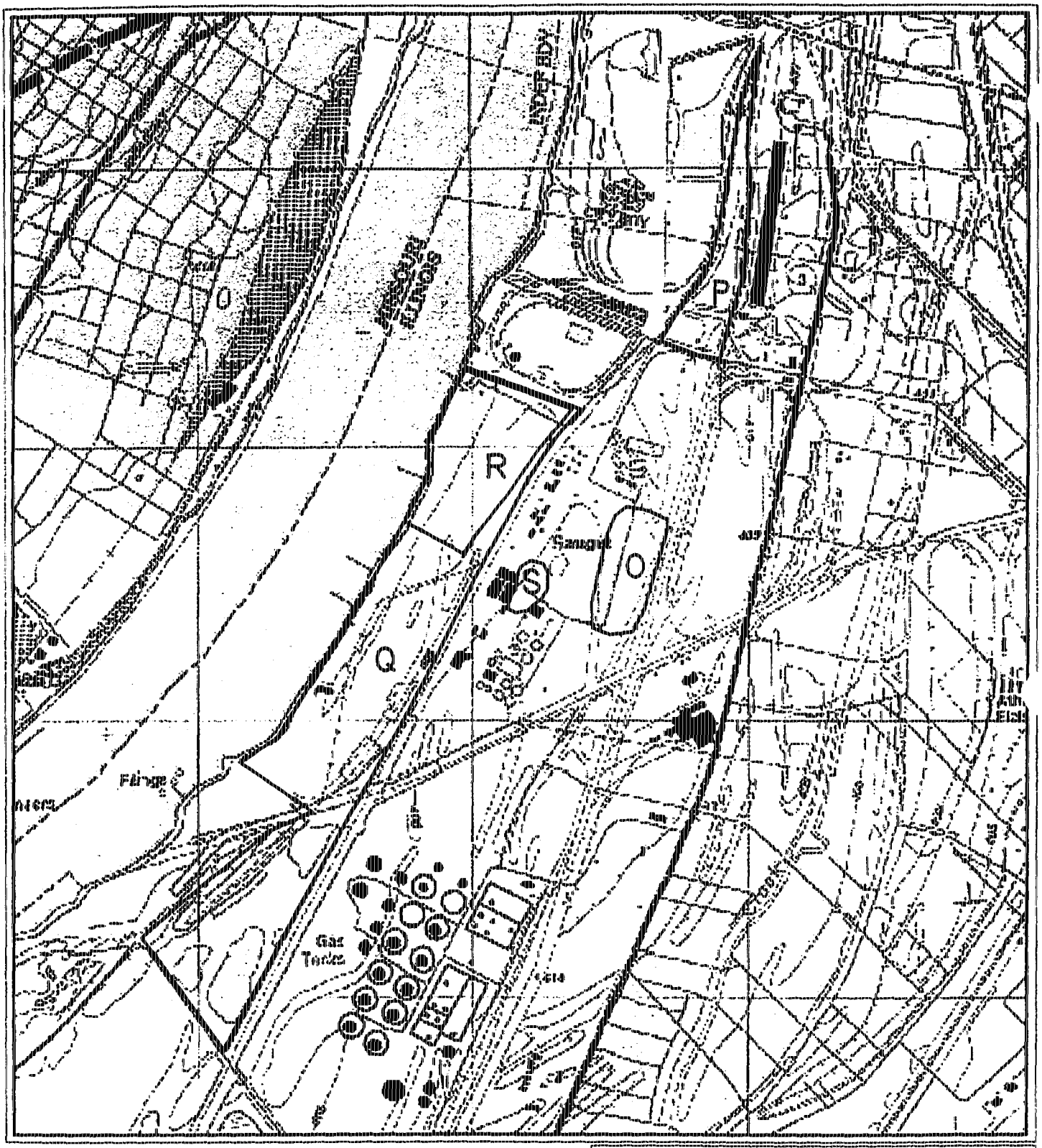
TBD

21.0 HEALTH AND SAFETY PLAN ACCEPTANCE

I have had the opportunity to read and ask questions about this HASP. My signature certifies that I understand the procedures, equipment, and restrictions of this plan and agree to abide by them.

SIGNATURE*	PRINTED NAME	COMPANY	DATE

This acceptance form is required for all routine site staff and subcontracting personnel. Visitors and non-routine subcontractors are required to receive and sign the appropriate Health and Safety Orientation Form located in Attachment F.



SOURCE: USGS QUADRANGLE (CAHOKIA, IL–MO), 1998
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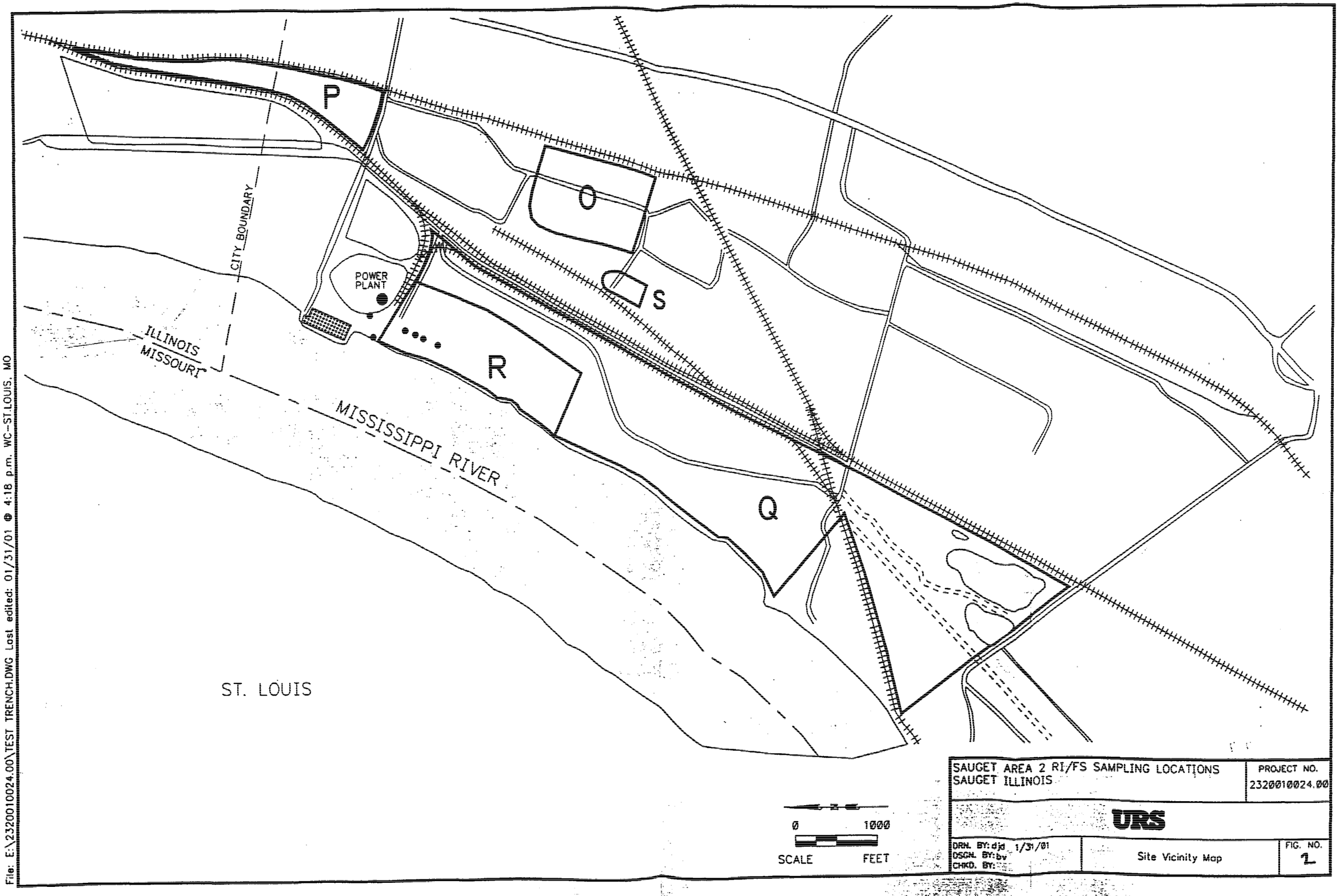
FIGURE 1

SITE LOCATION MAP

SAUGET AREA 2
SITES O, P Q, R, S
SAUGET AND CAHOKIA, ILLINOIS



SAUGET AREA 2 RI/FS SAMPLING LOCATIONS
SAUGET ILLINOIS

PROJECT NO. 2320010024.00

URS

DRN. BY: djd 1/31/01
DSGN. BY: bv
CHKD. BY:

Site Vicinity Map

FIG. NO. 1

File: E:\2320010024.00\TEST TRENCH.DWG Last edited: 01/31/01 @ 4:18 p.m. WC-ST.LOUIS, MO

Table 1
Maximum Constituent Concentrations in Soil from Previous Studies
Sauget Area 2

Constituent	Site O	Site P	Site Q	Site R ^	Site S
VOCs				5800	
benzene	30.8				
chlorobenzene	58.9		100		
ethylbenzene	167		790		450
4-methyl-2-pentanone	7.69		250		93
toluene	29.5	0.413	2400		990
1,1,1-trichloroethane	1.41				12
o-xylene			2300		
xylenes(total)	615.4	0.45			620
SVOCs				19000	
1,4-dichlorobenzene	1030	8.87	1200		
1,2-dichlorobenzene	606	3.625			
1,2,4-trichlorophenol	26.9				
naphthalene	34.6				200
2-methylnapthalene	160				
N-nitrosodiphenylamine	50				
pentachlorophenol	1620				
phenanthrene	230				
fluoranthene	74				
pyrene	282				
butyl benzyl phthalate	3846				490
benzo(a)anthracene	121.7				
1,2,4-trichloroebenzene	65.3				
chrysene	282				
phenol		3.875			
di-n-butyl-phthalate		16.25	900		1500
di-n-octyl-phthalate					310
bis(2-ethylhexyl)phthalate			1100		20000
PCBs				4800	
Aroclor 1232	30.3				
Aroclor 1242	1871				
Aroclor 1248			70		85
Aroclor 1254			360		69
Aroclor 1260			16000		41
Dioxins					
2,3,7,8-TCDD	0.00017		0.0033		
Metals					
antimony			17900		
arsenic			0.216		
cadmium	31		152000		
chromium			3650		
copper	341		1630		139
cyanide		15			
lead		526	195000		0.392
mercury	6.3	3.9	4.9		3.5
nickel	136		371		
selenium			59.9		
silver			30.2		
thallium			0.89		
zinc	1398		9520		327

all concentrations in ppm

^ - Soil sampling at Site R showed VOC concentrations ranging from .15 to 5800 ppm. SVOCs were found at levels ranging from 0.017 to 19,000 ppm. Pesticides were found at levels ranging from 0.11 to 99 ppm and PCBs were detected at levels ranging from 0.75 to 4,800 ppm. Elevated levels of arsenic, chromium, lead, nickel, and mercury were also detected in Site R soils.

Table 2

Project Organization and Roles

NAME	PROJECT ROLE	PHONE NUMBER
Solutia, Inc.		
Steven Smith	Project Manager	314-674-4922
AMEC Earth and Environmental, Inc.		
Charles Harman, PWS	Project Manager	732-302-9500
Laurie Gneiding, CEP	Terrestrial Field Team Leader	732-302-9500
J. David Dean	Aquatic Field Team Leader	770-420-2100
TBD	SSHO	
Jeffrey Tasca	Project H &S Manager (HSM)	(732) 302-9500
Denise Daggett, CIH	Corporate H &S Officer	(858) 458-9111

Table 3
Activity Hazard Analysis

Project Identification Sauget Area 2	**Location** Sauget and Cahokia Illinois	**Estimated Start Date** April, 2001
TASK	**POTENTIAL HAZARDS**	**CONTROL MEASURES**
Aquatic Sampling (surface water, sediment and biota collection)	Exposure to Chemical Hazards	* Wear appropriate PPE * Practice contamination avoidance * Follow proper personal and sample decontamination procedures. * Wash hands/face immediately as part of decontamination * Wear chemical safety goggles when handling chemical sample preservatives and samples * Avoid splashing. If inevitable, personnel should stay out of splash radius. * Wear chemical protective gloves (nitrile).
	Manual Lifting and Material Handling	* Use proper lifting techniques * Team lifting will be used for heavy loads (> 60 lbs.)
	Heat Stress	* Personnel must be aware of sign/symptoms * Personnel must drink plenty of fluids * Practice heat stress prevention per HSP
	Splashing	* Use safety glasses or goggles; and * All personnel should stay out of the splash radius.
	Slips/Trip/Falls	* Work areas and means of access shall be maintained neat and orderly * Even terrain will be utilized as unloading areas
	Boating Operations	* Individuals operating boasts must be experienced and qualified * Boats are to be occupied during use by not less than one qualified operator plus one additional person. * The designated boat operator will provide a safety briefing to all boat occupants prior to disembarking * Maximum weight load for a boat will not exceed manufacturer's specified capacity. * All persons on board will remain seated except when sampling * All gear will be stowed securely against unexpected shifts. * All personnel on board will wear a Coast Guard approved Type II personal floation devices. * On-board personnel must be able to contact shore either by cellular phone or radio

Table 3 (continued)
Activity Hazard Analysis

Project Identification Sauget Area 2	Location Sauget and Cahokia Illinois	Estimated Start Date April, 2001
TASK	**POTENTIAL HAZARDS**	**CONTROL MEASURES**
Terrestrial Sampling (vegetation and macroinvertebrate sample collection)	Exposure to Chemical Hazards	* Wear appropriate PPE per HASP * Practice contamination avoidance * Follow proper personal and sample decontamination procedures * Wash hands/face immediately as part of decontamination * Wear chemical safety goggles when handling chemical sample preservatives and samples * Avoid splashing. If inevitable, personnel should stay out of splash radius * Hazard communication training
	Manual Lifting, Material Handling, and Hand Auger Usage	* Use proper lifting techniques * Team lifting will be used for heavy loads (> 60 lbs.)
	Heat Stress	* Personnel must be aware of sign/ symptoms of heat stress * Personnel will drink plenty of fluids * Practice heat stress prevention per HASP
	Slips/Trip/Falls	* Work areas and means of access shall be maintained neat and orderly * Even terrain will be utilized as unloading areas

Table 4
Chemical Hazard Properties and Exposure Information

CHEMICAL NAME/ SYNONYM	OSHA PEL[1]/ ACGIH TLV[2]	STEL[3]/ IDLH[4]	IP[5] (eV)	LEL/UEL[6]	RELEVANT EXPOSURE PATHWAY	SYMPTOMS	PROPERTIES/ CHARACTERISTICS	INCOMPATABILITIES/ REACTIVITIES
Aniline	5 ppm	Ca (100 ppm)	ND	11%/1.3%	Inh Con	Headache, weakness, dizziness, fast heartbeat, cyanosis[7]; eye irritant	colorless to brown, oily liquid with aromatic amine-like odor. Solid below 21°F	strong oxidizers, strong acids, toluene diisocyanate, alkalis
Antimony	0.5 mg/m^3	50 mg/m^3	NA	NA/NA	Inh Con	Irritant to eyes, skin, nose, throat, and mouth. Coughing, dizziness, headache, and loss of olfactory function (smell).	Silver-white, lustrous, hard, brittle solid. Scale like crystals or a dark-gray lustrous powder.	strong oxidizers, acids, halogenated acids.
Arsenic (inorganic compounds)	0.01 mg/m^3/ 0.5 mg/m^3	0.002 mg/m^3/ 5 mg/m^3	NA	NA/NA	Inh Abs Con	Ulceration of nasal septum, dermatitis, respiratory irritation, darkening of skin, [carc]	Metal: Silver-gray or tin-white brittle, odorless solid.	Strong oxidizers, bromine, azide
Benzene	0.5 ppm/1 ppm	1 ppm/ 500 ppm	9.24	1.2%/7.7.8%	Inh Abs Con	Irritation oft eyes, skin, nose, respiratory system; giddiness; headache, nausea, staggered gait; fatigue, anorexia, tiredness; dermatitis; [carc]	Colorless to light-yellow liquid with an aromatic odor. [Note: A solid below 42° F.]	Strong oxidizers, fluorides, perchlorates, nitric acid
Bis(2-ethylhexyl)phthalate	5 mg/m^3	10 mg/m^3/ 5000 mg/m^3	ND	0.3%/ND	Inh Con	Irritant to eyes, mucous membranes.	Colorless, oily liquid with a slight odor.	
Butyl benzyl phthalate	ND	5 mg/m^3/ ND	ND	ND	Inh Con	ND	clear, oily liquid	Oxidizers
Cadmium	0.01 ppm 0.005 mg/m^3	9 mg/m^3	NA	NA/NA	Inh Ing	Fluid in lungs, difficulty breathing, coughing, chest tightness, headache, chills, muscle aches, nausea, vomiting, diarrhea, mild anemia, [carc]	Metal: Silver-White, blue-tinged, lustrous, odorless solid.	strong oxidizers, sulfur, selenium, tellurium
Chlorobenzene	10ppm 750 ppm	1000 ppm	9.07	1.3%/9.6%	Inh Con	Irritated eyes, skin, and nose. Drowsiness and incoherence.	Colorless liquid with an almond like odor.	strong oxidizers

NA: Not applicable
ND: No data available
[1] OSHA PEL = OSHA Permissible Exposure Limit is a promulgated concentration that cannot be exceeded during an 8-hour time shift over a 40-hour work week.
[2] ACGIH TWA = Guideline not to be exceeded during an 8-hour time shift over a 40-hour work week.
[3] STEL = short-term exposure limit. Concentration not to be exceeded for 15-minute time-weighted average.
[4] IDLH = immediately dangerous to life and health. Represents the maximum concentration from which, in the even of respirator failure, one could escape within 30 minutes without a respirator and without experiencing any escape-impairing or irreversible health effects.
[5] IP = ionization potential. Given as a guideline to determine which lamp to use with a photoionization detector (PID).
[6] LEL/UEL = lower explosive limit/upper explosive limit. Represents the upper and lower flammable limits in air at room temperature.
[7] Cyanosis = purple discoloration of skin due to lack of oxygen

Table 4
Chemical Hazard Properties and Exposure Information

CHEMICAL NAME/ SYNONYM	OSHA PEL[1]/ ACGIH TLV[2]	STEL[3]/ IDLH[4]	IP[5] (eV)	LEL/UEL[6]	RELEVANT EXPOSURE PATHWAY	SYMPTOMS	PROPERTIES/ CHARACTERISTICS	INCOMPATABILITIES/ REACTIVITIES
Chromium (as Cr^{3+} dust)	0.5 mg/m^3 1 mg/m^3	25 mg/m^3	NA	NA/NA	Inh Con	Irritant to eyes, skin, and lungs	Blue-white to steel-gray, lustrous, brittle, hard, odorless solid.	compound specific
Copper (dusts)	1 mg/m^3 1 mg/m^3	100 mg/m^3	NA	NA/NA	Inh Con	Irritant to eyes, nose, pharynx, and nasal passages.	Reddish, lustrous, malleable, odorless solid.	oxidizers, alkalis, acetylene
Cyanide	10 ppm	50 ppm	13.6	5.6%/40%	Inh Abs Con	Asphyxiation, weakness, headache, confusion, nausea, vomiting, increased rate and depth of respiration, or slow and gasping respiration.	Colorless or pale blue liquid or gas above 78° F. Bitter, almond like odor.	
Di-n-butylphthalate	5 mg/m^3	4000 mg/m^3	ND	0.5%/ND	Inh Abs Con	Eye and upper respiratory tract irritant.	Colorless to faint yellow oily liquid	nitrates, strong oxidizers, alkalis, acids, liquid chlorine
Dichlorobenzenes (1,2-; 1,4)	1,2-: 50 ppm 1,4-: 75 ppm	1,2-: 200 ppm 1,4-: 150 ppm [Ca]	8.98	2.5%/NA	Inh Abs Con	Irritation to eyes, and nose. Liver and kidney damage. Skin blisters; headache	Colorless to pale yellow liquid with a pleasant aromatic odor./ Colorless or white crystalline solid with a mothball like odor.	
Di-n-octylphthalate	5 mg/m^3	10 mg/m^3/ ND	ND	0.3%/ND	Inh Con	Respiratory tract, skin and eye irritant	Light-colored, colorless liquid	Strong oxidizers, acids, and alkalis
Ethyl benzene	100 ppm/ 100 ppm	125 ppm/ 800 ppm	8.76	0.8%/ 6.7%	Inh Con	Irritant to eyes, skin, mucous membranes; headache; dermatitis; narcosis (tiredness), coma	Colorless liquid with an aromatic odor.	Strong oxidizers
Isopropanol (70%)	400 ppm 400 ppm	2000 ppm	10.10	2.0%/12.7%	Inh Con	Irritation eyes, nose, throat; drowsiness, dizziness, headache; dry cracking skin;	Colorless liquid with the odor of rubbing alcohol.	Strong oxidizers, acetaldehyde, chlorine, ethylene oxide, acids, isocyanates
Lead	0.1 mg/m^3 0.05 mg/m^3	100 mg/m^3	NA	NA/NA	Inh Con	Weakness, insomnia, facial pallor, irritated eyes, and hypotension.	A heavy, ductile, soft, gray solid.	Strong oxidizers, hydrogen peroxide, acids
Mercury (except organomercury)	Hg vapor: 0.05/0.1 mg/m^3 Other: 0.1 mg/m^3	10 mg/m^3	NA	NA/NA	Inh Abs Con	Irritation to eyes, and skin. Coughing, chest pain, difficulty breathing, bronchitis, tremors, insomnia, irritability, indecision, headache, fatigue, weakness	Metal: Silver-white, heavy, odorless liquid.	acetylene, ammonia, chlorine dioxide, azides, calcium, sodium carbide, lithium, copper

Table 4
Chemical Hazard Properties and Exposure Information

CHEMICAL NAME/ SYNONYM	OSHA PEL[1]/ ACGIH TLV[2]	STEL[3]/ IDLH[4]	IP[5] (eV)	LEL/UEL[6]	RELEVANT EXPOSURE PATHWAY	SYMPTOMS	PROPERTIES/ CHARACTERISTICS	INCOMPATABILITIES/ REACTIVITIES
4-Methyl-2-pentanone	50 ppm 100 ppm	500 ppm	9.34	?/8%	Inh Con	Irritation to eyes, nose, and skin. Headaches, sleepiness, coma,	Colorless liquid with a pleasant odor.	
Nickel (metals or other compounds except nickel carbonyl)	0.015 mg/m^3 1 mg/m^3	10 mg/m^3	NA	NA/NA	Inh Con	Dermal sensitivity, allergic asthma, and pneumonitis[8] [carc]	Metal: Lustrous, silvery odorless solid	Strong acids, sulfur, selenium, wood and other combustibles.
N-nitrosodiphenylamine	ND	ND	ND	ND	Inh Con	Eye irritant, skin redness, skin edema, [Carc]	greenish crystals	Oxidizers
PAHs (as coal tar pitch volatiles)	0.2 mg/m^3 / 0.2 mg/m^3	ND/80 mg/m^3	ND	ND	Inh Con	Dermatitis, bronchitis [carc]	Black or dark brown amorphous residue	
PCBs	0.5 mg/m^3 1 mg/m^3	5 mg/m^3	ND	ND	Inh Abs Con	Irritation to eyes, chloracne, liver damage, reproductive effects. [carc]	Colorless to light colored viscous liquid with a slight hydrocarbon odor.	Strong oxidizers
Pentachlorophenol	0.5 mg/m^3 0.5 mg/m^3	2.5 mg/m^3	ND	ND	Inh Abs Con	Irritation to eyes, nose, throat. Sneezing, coughing, sweating, headache, dizziness, nausea, *vomiting*, difficulty breathing chest pain, high fever, dermatitis.	Colorless to white, crystalline solid with a benzene like odor. [fungicide]	Strong oxidizers, acids, alkalis
Phenol	5 ppm 5 ppm	250 ppm	8.5	1.8%/8.6%	Inh Abs Con	Irritation to eyes, nose, and throat. Weakness, muscle aches, pain, dark urine, cyanosis , skin burns, dermatitis, ochronosis[9], *tremors, convulsions, twitching.*	Colorless to light pink crystalline solid with a sweet acrid odor.	Strong oxidizers, calcium hypochlorite, acids
Selenium	0.2 mg/m^3 0.2 mg/m^3	1 mg/m^3	NA	NA/NA	Inh Con	Irritation to eyes, nose, and throat. Vision disturbances, headache, chills, fever, bronchitis, dermatitis, skin burns	Amorphous or crystalline red to gray solid.	Acids, Strong oxidizers, chromium trioxides, potassium bromate, cadmium
Silver (metal dust and soluble compounds)	0.1mg/m^3 0.1 mg/m^3	10 mg/m^3	NA	NA/NA	Inh Con	Blue-gray eyes, nasal septum, throat, skin irritation, skin ulceration,	Metal; white lustrous solid	Ethyleneimine, acetylene, hydrogen peroxide, bromoazide, chlorine trifluoride

[8] Pneumonitis = inflammation of the lungs.
[9] Ochronosis = pigment deposits in the cartilages, ligaments, and tendons associated with alkaptonuria (alkaptons in urine).

Table 4
Chemical Hazard Properties and Exposure Information

CHEMICAL NAME/ SYNONYM	OSHA PEL[1]/ ACGIH TLV[2]	STEL[3]/ IDLH[4]	IP[5] (eV)	LEL/UEL[6]	RELEVANT EXPOSURE PATHWAY	SYMPTOMS	PROPERTIES/ CHARACTERISTICS	INCOMPATABILITIES/ REACTIVITIES
2,3,7,8-tetrachlorodibenzo-p-dioxin	ND	ND	ND	ND	Inh Abs Con	Irritation to eyes, skin, mucous membranes, thermal skin burns, headache, sore throat.	Colorless to white crystalline solid	
Thallium	0.1 mg/m^3 0.1 mg/m^3	15 mg/m^3	NA	NA	Inh Abs Con	Ptosis[10], strabismus[11], tremor, chest pain and tightness, pulmonary edema, seizures, uncontrolled movements, psychosis, hair loss, numbness in legs.	Appearance and odor vary depending upon the specific soluble thallium compound..	
Toluene	50 ppm [skin]	150 ppm/ 500 ppm	8.82	1.1%/ 7.1%	Inh Abs Con	Irritant to eyes, nose; fatigue, weak, confusion, euphoria, dizziness, headache; dilated pupils, tearing eyes; nervousness, muscle fatigue, insomnia; dermatitis	Colorless liquid with a sweet pungent, benzene-like odor.	Stron oxidizers
1,2,4-Trichlorobenzene	5 ppm	ND	ND	ND/6.6%	Inh Con	Irritation to eyes, skin, mucous membranes	Colorless liquid or crystalline solid (below 63 F) with an aromatic odor.	Acids, acid fumes, oxidizers, steam
1,1,1-Trichloroethane	350 ppm	700 ppm	11	7.5%/12.5%	Inh Con	Irritation to eyes, and skin. Headache, lassitude, CNS[12] depression, poor equilibrium, dermatitis, cardiac arrhythmia: liver damage	Colorless liquid with a mild chloroform like odor.	ND
1,2,4-Trichlorophenol	ND	ND	ND	ND	ND	ND	ND	ND
Xylenes (o, m, p)	100 ppm/ 100 ppm	150 ppm/ 900 ppm	8.56 (o,m 8.44(p)	m,p: 1.1%/7.0% o: 0.9%/6.7%	Inh Abs Con	Irritant to eyes, skin, nose, throat; dizziness, excitement, drowsiness, incoordination, staggering gait; dermatitis.	Colorless liquid with an aromatic odor.	Strong oxidizers, strong acids
Zinc (as zinc oxide)	5 mg/m^3 5 mg/m^3	10/500 mg/m^3	NA	NA/NA	Inh Con	Irritant to eyes, skin, upper respiratory system: coughing.	White, odorless solid	water

[10] Ptosis = sagging organ or parts, e.g., drooping upper eyelid
[11] Strabismus = inability for one eye to focus for binocular vision
[12] CNS = central nervous system

TABLE 5

Wind Chill Factors

Wind Speed (mph)	Local Temperature, °F										
	32	23	14	5	-4	-13	-22	-31	-40	-49	-58
	Equivalent Temperature, °F										
Calm	32	23	14	5	-4	-13	-22	-31	-40	-49	-58
5	29	20	10	1	-9	-18	-28	-37	-47	-56	-65
10	18	7	-4	-15	-26	-37	-48	-59	-70	-81	-91
15	13	-1	-13	-25	-37	-49	-61	-73	-85	-97	-109
20	7	-6	-19	-32	-44	-57	-70	-83	-96	-109	-117
25	3	-10	-24	-37	-50	-64	-77	-90	-104	-117	-121
30	1	-13	-27	-41	-54	-68	-82	-97	-109	-123	-137
35	-1	-15	-29	-43	-57	-71	-85	-99	-113	-127	-142
40	-3	-17	-31	-45	-59	-74	-87	-102	-116	-131	-145
45	-3	-18	-32	-46	-61	-75	-89	-104	-118	-132	-147
50	-4	-18	-33	-47	-62	-76	-91	-105	-120	-134	-148
LITTLE DANGER FOR PROPERLY CLOTHED PERSONS			**CONSIDERABLE DANGER**				**VERY GREAT DANGER**				
Maximum danger of false sense of security			Danger from freezing of exposed flesh within one minute				Flesh may freeze within 30 seconds				
Trenchfoot and immersion foot may occur at any point on this chart											

TABLE 6
Sauget Area 2 Sites
Health and Safety Plan

WORK/WARM-UP SCHEDULE FOR FOUR-HOUR SHIFT

Air Temp. - Sunny Sky	No Noticeable Wind		5 mph Wind		10 mph Wind		15 mph Wind		20 mph Wind	
°F (approx.)	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks
-15° to -19°	norm.breaks	1	norm. breaks	1	75 min.	2	55 min.	3	40 min.	4
-20° to -24°	norm. breaks	1	75 min.	2	55 min.	3	40 min.	4	30 min.	5
-25° to -29°	75 min.	2	55 min.	3	40 min.	4	30 min.	5	Non-emergency work should cease ↓	
-30° to -34°	55 min.	3	40 min.	4	30 min.	5	Non-emergency work should cease ↓		↓	
-35° to -39°	40 min.	4	30 min.	5	Non-emergency work should cease ↓		↓		↓	
-40° to -44°	30 min.	5	Non-emergency work should cease ↓		↓		↓		↓	
-45° & below	Non-emergency work should cease		↓		↓		↓		↓	

NOTES:

1. Schedule applies to any 4-hour work period with moderate to heavy work activity, with warm-up periods in a warm location and with an extended break (e.g., lunch) a the end of the 4-hour work period in a warm locations. For light-to-moderate work (limited physical movement): apply the schedule one step lower.

2. TLV applies only for workers in dry clothing.

Source: ACGIH TLVs and BEIs. 1992/1993

Table 7
Type and Frequency of Air Monitoring

TYPE	MINIMUM RECOMMENDED FREQUENCY
Background:	Once per day in the work area and perimeter using direct-reading instruments, prior to any intrusive activities or equipment startup.
Perimeter:	Once per hour using direct-reading instruments during intrusive activities.
Personnel:	At least twice per day in the breathing zone of those with the highest anticipated exposure during intrusive activities.
Area:	At least twice per day in each work zone and at the onset of any new intrusive activities, or at new locations.
Environmental:	Periodic field screening of selected samples as per the Sampling and Analysis Plan.

Table 8
Action Levels for Change in PPE

EQUIPMENT	ACTION LEVEL	ACTION TO BE TAKEN
PID	>5 ppm sustained for greater than 5 minutes	Upgrade to level C
PID	>50 ppm	Upgrade to level B
PID	>500 ppm	Stop all work activities, evacuate the site and call proper authorities.

ATTACHMENT A

MEDICAL DATA SHEET

MEDICAL DATA SHEET

This brief Medical Data Sheet will be completed by all onsite personnel and will be kept in the support zone by the Site Health and Safety Officer during the conduct of the site operations. Completion of this form is required in addition to compliance with the Medical Surveillance Program requirements. This data sheet will accompany any personnel when medical assistance is needed or if transport to a hospital facility is required.

Project:		
Name:		Home Telephone:
Address		
Age:	Height:	Weight:
Blood Type:		
Emergency Contact:		
Drug or Other Allergies:		
Particular Sensitivities		
Do you wear contacts?		
Provide a checklist of previous illnesses		
Exposure to Hazardous Chemicals		
What medications are you presently using?		
Do you have any medical restrictions?		
Name/Address/Phone Number of Personal Physician		

ATTACHMENT B

INCIDENT REPORT

1.0 INTRODUCTION

Accident investigation is a useful tool for discovering the cause(s) of an accident and to prevent future accidents. A thorough investigation of the contributing circumstances that might have caused an accident (including both injury accidents and non-injury accidents [incidents]) is crucial. The real or potential extent of injury or property damage dictates the thoroughness of the investigation. Causative factors are then evaluated to determine the appropriate corrective action. Detailed corrective action information is provided in the Injury and Illness Prevention Program, Volume I, of this manual.

Accident investigation is not to be confused with injury or illness reporting (discussed in SOP A-3, *Injury or Illness Reporting*).

2.0 SCOPE

This procedure applies to both accidents which occur in office operations or field jobsites. This procedure pertains to accidents involving OEES personnel, personnel subcontracted to OEES, and visitors, including clients, who may work on or visit OEES jobsites.

OEES reserves the right to investigate and document any accident involving a subcontractor.

3.0 RESPONSIBILITIES

3.1 CORPORATE HEALTH AND SAFETY DIRECTOR (CHSD)

The CHSD manages an accident investigation, either parallel to or in conjunction with the Project Manager (PM)/Supervisor, as the circumstances warrant. In instances of a severe injury or fatality, or a potentially catastrophic near miss, the CHSD will

investigate and report to the Executive Vice President of Operations. The CHSD serves in an advisory capacity for all accident investigations. The CHSD also periodically analyzes the accident investigation data for the purpose of identifying underlying causes and general patterns.

3.2 Health and Safety Manager (HSM)

The HSM is responsible for initiation and oversight of accident investigations. Depending on the breadth of the investigation, the HSM may conduct the accident investigation.

3.3 PM/Supervisor

The PM/Supervisor is responsible for conducting accident investigations relating to projects or activities under his/her supervision. The PM/Supervisor is also responsible for ensuring that corrective actions (identified through the accident investigation) are implemented within the targeted time frame and that the appropriate injury/illness or incident reports are made.

3.4 Health and Safety Coordinator (HSC)/Site Health and Safety Coordinator (SHSC)

The HSC/SHSC shall assist the PM/Supervisor during accident investigations and shall verify the PM/Supervisor's findings.

3.5 Employees and Subcontractors

Employees and subcontractor employees are responsible for cooperating with accident investigators during the investigation and for being truthful and answering questions to the best of their recollection. Employees and subcontractor employees shall also comply with corrective action recommendations within the targeted time frame.

4.0 GOALS OF ACCIDENT INVESTIGATION

The PM/Supervisor should make a personal investigation of all accidents because:

- An investigation is the best way to determine the true cause of an accident.
- The PM/Supervisor should know what the employee was doing, the proper way to do it, and what the employee probably did or did not do to cause the accident.
- The PM/Supervisor is best qualified to evaluate the information gathered and determine the cause of the accident.
- The PM/Supervisor has the authority to investigate the accident and initiate corrective action.

5.0 INVESTIGATIVE PROCEDURES

The PM/Supervisor shall conduct an investigation of all incidents, injuries, and work-related illnesses; talk with the victim and/or co-workers who witnessed the accident or conditions; and examine the doctor's medical report if available. Each investigation shall be conducted as soon after the accident/incident as possible. Delays of even a few hours can allow for the destruction/alteration of evidence (whether intentional or unintentional).

Fairness and impartiality are essential during the investigation. The purpose of the investigation is to obtain information and prevent a recurrence of the incident/accident, not to place blame.

5.1 REPORTING

The PM/Supervisor shall report verbally and in writing any injury, illness, or incident on the appropriate forms and to the appropriate parties as defined in SOP A-3.

5.2 INVESTIGATING

The investigation phase of the accident investigation procedure is initiated in response to reports of an accident (either injury or non-injury). Important information that should be obtained through the investigation include:

- Project
- Location of accident
- Employee(s) involved
- Narrative description of the accident
- Equipment associated with the accident
- Task being performed when accident happened
- Time factors (e.g., time of day, hours into shift, type of shift)
- Preventive measures
- Characteristics of injury or property damage

5.2.1 Interviewing

After reviewing the Supervisor's Report of Injury or Illness, First-aid Incident Report, and/or Incident Report, or after verbal report of an accident, the PM/Supervisor and HSC/SHSC shall interview individually all witnesses to the accident. It is recommended to prepare a set of questions and ask each witness to answer the same questions. The details of the interview shall be recorded on the Accident Investigation Interview Report (Attachment 1). The completed form shall be forwarded to the appropriate HSM, office HSC, SHSC, and CHSD.

5.2.2 Identifying Causal Factors and Corrective Actions

The PM/Supervisor shall identify contributing factors to the accident using the National Safety Council's *Guide for Identifying Causal Factors and Corrective Actions* (Attachment 2). Completed guides shall be forwarded to the appropriate HSM, office HSC, SHSC, and CHSD.

Possible corrective actions are suggested on the guide, but keep in mind that these are not the only possible corrective actions that may be implemented. The recommended (and mandatory) corrective action(s) will be decided upon by the PM/Supervisor, with the

assistance of the HSC/SHSC and CHSD (when necessary), and stated in the appropriate column of the guide.

Corrective actions will be selected based upon effectiveness, cost versus benefit, feasibility, reliability, acceptance, effect on productivity, time required to implement, and any other factor deemed significant. Corrective actions can be monitored for implementation and completion using the Corrective Action Program Schedule (Attachment 3).

5.3 ANALYSIS

The CHSD periodically analyzes the accident investigation data for the purpose of identifying underlying causes or general patterns. The analysis will also identify inadequate policies, procedures, or management systems that are not always readily evident when reviewing each individual accident.

5.4 DOCUMENTATION

Accident investigations will be documented using photographs and by sequestering faulty tools and equipment that were involved, as well as by completing the appropriate forms (see Section 6.0, Record Keeping).

6.0 RECORD KEEPING

An Accident Investigation Interview Report (Attachment 1), Guide for Identifying Causal Factors and Corrective Actions (Attachment 2), and Corrective Action Program Schedule (Attachment 3) will be completed for each work-related incident, injury, or illness that occurs.

7.0 REFERENCES

Fed-OSHA. 1997. 29 CFR 1904, *Recording and Reporting Occupational Injuries and Illnesses.*

Cal-OSHA. 1997. 8 CCR 3203, *Injury and Illness Prevention Program.*

National Safety Council. 1997. Accident Prevention Manual for Business and Industry: Administration and Programs. 10th ed.

8.0 ATTACHMENTS

1. Accident Investigation Interview Report
2. Guide for Identifying Causal Factors and Corrective Actions
3. Corrective Action Program Schedule

ATTACHMENT 1

ACCIDENT INVESTIGATION INTERVIEW REPORT

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ACCIDENT INVESTIGATION INTERVIEW REPORT

Name of Interviewer: _____ Name of Interviewee: _____

Department of Interviewer: _____ Department of Interviewee: _____

Project: _____ Time shift began: _____ a.m/p.m.

Project activity during this shift: _____

Accident date: _____ Time of accident: _____ a.m/p.m.

Date accident was reported? _____

Any injuries involved? Yes ❑ No ❑ _____

Location of accident (include address, city, county, zip code): _____

Name of witnesses: _____

What was/were employee(s) doing when accident occurred? Be specific (i.e., walking, lifting, operating machinery, etc.)?

Please describe fully the events that resulted in the accident. Tell what happened and how it happened. (Do not describe the nature of the injury or property damage.).

What machine, tool, or object was most closely connected to the accident? (e.g, machine employee struck against or which struck him, vehicle employee was driving)?

Nature of injury or property damage.

Additional information or drawings can be included on the reverse side of this form.

Forward completed form to the appropriate HSM, office HSC, SHSC, and CHSD.

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ATTACHMENT 2

GUIDE FOR IDENTIFYING CAUSAL FACTORS AND CORRECTIVE ACTIONS

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GUIDE FOR IDENTIFYING CAUSAL FACTORS and CORRECTIVE ACTIONS

Project/Department: _____ Investigators: _____

Accident Date/Time: _____ Date/Time of Investigation: _____

PART 1 - EQUIPMENT

Answer questions by placing an x in the appropriate box or circle (Y=Yes, N=No)

○ Y ☐ N 1.0 WAS A HAZARDOUS CONDITION(S) A CONTRIBUTING FACTOR?

	CAUSAL FACTORS	COMMENTS	POSSIBLE CORRECTIVE ACTIONS	RECOMMENDED CORRECTIVE ACTIONS	TARGET COMPLETION DATE
☐ Y ☐ N	1.1 Did any defect(s) in equipment/tool(s)/material contribute to hazardous condition(s)?		Review procedures for inspecting, reporting, maintaining, repairing, replacing, or recalling defective equipment/tool(s)/material used.		
☐ Y ○ N	1.2 Was the hazardous condition(s) recognized? *If yes, answer A and B. If no, proceed to 1.3.*		Perform job hazard analysis. Improve employee ability to recognize existing or potential hazardous conditions. Provide test equipment, as required, to detect hazard. Review any change or modification of equipment/tool(s)/ material.		
☐ Y ○ N	A. Was the hazardous condition(s) reported?		Train employees in reporting procedures. Stress individual acceptance of responsibility.		
☐ Y ○ N	B. Was employee(s) informed of the hazardous condition(s) and the job procedures for dealing with it as an interim measure?		Review job procedures for hazard avoidance. Review supervisory responsibility. Improve supervisor-employee communications. Take action to remove or minimize hazard.		
☐ Y ○ N	1.3 Was there an equipment inspection procedure(s) to detect the hazardous condition(s)?		Develop and adopt procedures (for example, an inspection system) to detect hazardous conditions. Conduct test.		
☐ Y ○ N	1.4 Did the existing equipment inspection procedure(s) detect the hazardous condition(s)?		Review procedures. Change frequency or comprehensiveness. Provide test equipment as required. Improve employee ability to detect defects and hazardous conditions. Change job procedures as required.		
☐ Y ○ N	1.5 Was the correct equipment/ tool(s)/material used?		Specify correct equipment/tool(s)/ material in job procedures.		
☐ Y ○ N	1.6 Was the correct equipment/tool(s)/ material readily available?		Provide correct equipment/tool(s)/ material. Review purchasing specifications and procedures. Anticipate future requirements.		
☐ Y ○ N	1.7 Did employee(s) know where to obtain equipment/tool(s)/material required for the job?		Review procedures for storage, access, delivery, or distribution. Review job procedures for obtaining equipment/ tool(s)/material.		
○ Y ☐ N	1.8 Was substitute equipment/tool(s)/ material used in place of correct one?		Provide correct equipment/tool(s)/ material. Warn against use of substitutes in job procedures and in job instruction.		
☐ Y ○ N	1.9 Did the design of the equipment/ tool(s) create operator stress or encourage operator error?		Review human factors engineering principles. Alter equipment/tool(s) to make it more compatible with human capability and limitations. Review purchasing procedures and specifications. Check out new equipment and job procedures involving new equipment before putting into service. Encourage employees to report potential hazardous conditions created by equipment design.		
○ Y ☐ N	1.10 Did the general design or quality of the equipment/tool(s) contribute to a hazardous condition?		Review criteria in codes, standards, specifications, and regulations. Establish new criteria as required.		
○	1.11 List other causal factors in "Comment" column.				

SOURCE: National Safety Council, Accident Prevention Manual for Business and Industry: Administration and Programs, 1988

OGDEN

PART 2 - ENVIRONMENTAL

Answer questions by placing an *x* in the appropriate box or circle (Y=Yes, N=No)

2.0 WAS THE LOCATION/POSITION OF EQUIPMENT/MATERIALS/EMPLOYEE(S) A CONTRIBUTING FACTOR ?

CAUSAL FACTORS	COMMENTS	POSSIBLE CORRECTIVE ACTIONS	RECOMMENDED CORRECTIVE ACTIONS	TARGET COMPLETION DATE
2.1 Did the location/position of equipment/material/employee(s) contribute to a hazardous condition?		Perform job hazard analysis. Review job procedures. Change the location/position or layout of the equipment. Change position of employee(s). Provide guardrails, barricades, barriers, warning lights, signs, or signals.		
2.2 Was the hazardous condition(s) recognized? If yes, answer A and B. If no, proceed to 2.3.		Perform job hazard analysis. Improve employee ability to recognize existing or potential hazardous conditions. Provide test equipment, as required, to detect hazard. Review any change or modification of equipment/tool(s)/ material.		
A. Was the hazardous condition(s) reported?		Train employees in reporting procedures. Stress individual acceptance of responsibility.		
B. Was employee(s) informed of the job procedure for dealing with the hazardous condition as an interim action?		Review job procedures for hazard avoidance. Review supervisory responsibility. Improve supervisor-employee communications. Take action to remove or minimize hazard.		
2.3 Was employee(s) supposed to be in the vicinity of the equipment/material?		Review job procedures and instruction. Provide guardrails, barricades, barriers, warning lights, signs, or signals.		
2.4 Was the hazardous condition created by the location/position of equipment/material visible to employee(s)?		Change lighting or layout to increase visibility of equipment. Provide guardrails, barricades, barriers, warning lights, signs, or signals, floor stripes, etc.		
2.5 Was there sufficient workspace?		Review workspace requirements and modify as required.		
2.6 Were environmental conditions a contributing factor (for example, illumination, noise levels, air contaminant, temperature extremes, ventilation, vibration, radiation)?		Monitor, or periodically check environmental conditions as required. Check results against acceptable levels. Initiate action for those found unacceptable.		
2.7 List other causal factors in "Comment" column.				

PART 3 - PEOPLE

3.0 WAS THE JOB PROCEDURE(S) USED A CONTRIBUTING FACTOR?

CAUSAL FACTORS	COMMENTS	POSSIBLE CORRECTIVE ACTIONS	RECOMMENDED CORRECTIVE ACTIONS	TARGET COMPLETION DATE
3.1 Was there a written or company procedure (rules) for this job ? If yes, answer A, B, and C. If no, proceed to 3.2.		Perform job hazard analysis and develop safe job procedures.		
A. Did job procedures anticipate the factors that contributed to the accident?		Perform job hazard analysis and change job procedures.		
B. Did employee(s) know the job procedure?		Improve job instruction. Train employees in correct job procedures.		
C. Did employee(s) deviate from the known job procedure?		Determine why. Encourage all employees to report problems with an established procedure to supervision. Review job procedure and modify if necessary. Counsel or discipline employee. Provide closer supervision.		
3.2 Was employee(s) mentally and physically capable of performing the job?		Review employee requirements for the job. Improve employee selection. Remove or transfer employees who are temporarily, either mentally or physically, incapable of performing the job.		
3.3 Were any tasks in the job procedure too difficult to perform (for example, excessive concentration or physical demands)?		Change job design and procedures.		
3.4 Is the job structured to encourage or require deviation from job procedures (for example, incentive, piecework, work pace)?		Change job design and procedures.		
3.5 List other causal factors in "Comment" column.				

3.6 WAS LACK OF PERSONAL PROTECTIVE EQUIPMENT OR EMERGENCY EQUIPMENT A CONTRIBUTING FACTOR IN THE INJURY?

SOURCE: National Safety Council, Accident Prevention Manual for Business and Industry: Administration and Programs, 1988

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PART 3 - PEOPLE (Continued)

Answer questions by placing an x in the appropriate box or circle (Y=Yes, N=No)

	CAUSAL FACTORS	COMMENTS	POSSIBLE CORRECTIVE ACTIONS	RECOMMENDED CORRECTIVE ACTIONS	TARGET COMPLETION DATE
	NOTE: THE FOLLOWING CAUSAL FACTORS RELATE TO THE INJURY.				
Y N	3.7 Was appropriate personal protective equipment (PPE) specified for the task or job? If yes, answer A, B, and C. If no, proceed to 3.8.		Develop methods to specify PPE requirements.		
Y N	A. Was appropriate PPE available?		Provide appropriate PPE. Review purchasing and distribution procedures.		
Y N	B. Did employee(s) know that wearing of specified PPE was required?		Review job procedures. Improve job instruction.		
Y N	C. Did employee(s) know how to use and maintain the PPE?		Improve job instruction.		
Y N	3.8 Was the PPE used properly when the injury occurred?		Determine why and take appropriate action. Implement procedures to monitor and enforce use of PPE.		
Y N	3.9 Was the PPE adequate?		Review PPE requirements. Check standards, specification, and certification of the PPE.		
Y N	3.10 Was emergency equipment specified for this job (for example, emergency showers, eyewash fountains)? If yes, answer the following. If no, proceed to Part 4.		Provide emergency equipment as required.		
Y N	A. Was emergency equipment readily available?		Install emergency equipment at appropriate locations.		
Y N	B. Was emergency equipment properly used?		Incorporate use of emergency equipment in job procedures.		
Y N	C. Did emergency equipment function properly?		Establish inspection/monitoring system for emergency equipment. Provide for immediate repair of defects.		
	3.11 List other causal factors in "Comment" column.				

PART 4 - MANAGEMENT

Y N 4.0 WAS A MANAGEMENT SYSTEM DEFECT A CONTRIBUTING FACTOR?

	CAUSAL FACTORS	COMMENTS	POSSIBLE CORRECTIVE ACTIONS	RECOMMENDED CORRECTIVE ACTIONS	TARGET COMPLETION DATE
Y N	4.1 Was there a failure by supervision to detect, anticipate, or report a hazardous condition?		Improve supervisor capability in hazard recognition and reporting procedures.		
Y N	4.2 Was there a failure by supervision to detect or correct deviations from job procedure?		Review job hazard analysis and job procedures. Increase supervisor monitoring. Correct deviations.		
Y N	4.3 Was there a supervisor/employee review of hazards and job procedures for tasks performed infrequently? (Not applicable to all accidents.)		Establish a procedure that requires a review of hazards and job procedures (preventive actions) for tasks performed infrequently.		
Y N	4.4 Was supervisor responsibility and accountability defined and understood?		Define and communicate supervisor responsibility and accountability. Test for understandability and acceptance.		
Y N	4.5 Was supervisor adequately trained to fulfill assigned responsibility in accident prevention?		Train supervisors in accident prevention fundamentals.		
Y N	4.6 Was there a failure to initiate corrective action for a known hazardous condition that contributed to this accident?		Review management safety policy and level of risk acceptance. Establish priorities based on potential severity and probability of recurrence. Review procedure and responsibility to initiate and carry out corrective actions. Monitor progress.		
	4.7 List other causal factors in "Comment" column.				

(X) ITEM IS A CAUSAL FACTOR

[X] ITEM IS NOT A CAUSAL FACTOR

SOURCE: National Safety Council, Accident Prevention Manual for Business and Industry: Administration and Programs, 1988

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ATTACHMENT 3

CORRECTIVE ACTION PROGRAM SCHEDULE

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CORRECTIVE ACTION PROGRAM SCHEDULE

LOCATION:

ATTENTION:

DATE:

ITEM NO.	DESCRIPTION OF ITEM	ASSIGNED TO	DATE ASSIGNED	COMPLETION TARGET	DATE COMPLETED	STATUS

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ATTACHMENT C

MATERIAL SAFETY DATA SHEETS

MSDSs

SYSTEM.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 1 - Slight
Flammability Rating: 4 - Extreme (Flammable)
Reactivity Rating: 2 - Moderate
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. Higher concentrations can produce central nervous system depression, narcosis, and unconsciousness.
Ingestion:
Swallowing small amounts is not likely to produce harmful effects. Ingestion of larger amounts may produce abdominal pain, nausea and vomiting. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms are expected to parallel inhalation.
Skin Contact:
Irritating due to defatting action on skin. Causes redness, pain, drying and cracking of the skin.
Eye Contact:
Vapors are irritating to the eyes. Splashes may cause severe irritation, with stinging, tearing, redness and pain.
Chronic Exposure:
Prolonged or repeated skin contact may produce severe irritation or dermatitis.
Aggravation of Pre-existing Conditions:
Use of alcoholic beverages enhances toxic effects. Exposure may increase the toxic potential of chlorinated hydrocarbons, such as chloroform, trichloroethane.

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing

is difficult, give oxygen. Get medical attention.
Ingestion:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately.
Skin Contact:
Immediately flush skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting upper and lower eyelids occasionally. Get medical attention.

5. Fire Fighting Measures

Fire:
Flash point: -20C (-4F) CC
Autoignition temperature: 465C (869F)
Flammable limits in air % by volume:
lel: 2.5; uel: 12.8
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire.
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Vapors can flow along surfaces to distant ignition source and flash back. Contact with strong oxidizers may cause fire. Sealed containers may rupture when heated. This material may produce a floating fire hazard. Sensitive to static discharge.
Fire Extinguishing Media:
Dry chemical, alcohol foam or carbon dioxide. Water may be ineffective. Water spray may be used to keep fire exposed containers cool, dilute spills to nonflammable mixtures, protect personnel attempting to stop leak and disperse vapors.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert

material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB(R) solvent adsorbent is recommended for spills of this product.

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Acetone:
-OSHA Permissible Exposure Limit (PEL):
1000 ppm (TWA)

-ACGIH Threshold Limit Value (TLV):
500 ppm (TWA), 750 ppm (STEL) A4 - not classifiable as a human carcinogen

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, a half-face organic vapor respirator may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece organic vapor respirator

may be worn up to 50 times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-face piece positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Clear, colorless, volatile liquid.

Odor:
Fragrant, mint-like

Solubility:
Miscible in all proportions in water.

Specific Gravity:
0.79 @ 20C/4C

pH:
No information found.

% Volatiles by volume @ 21C (70F):
100

Boiling Point:
56.5C (133F) @ 760 mm Hg

Melting Point:
-95C (-139F)

Vapor Density (Air=1):
2.0

Vapor Pressure (mm Hg):
400 @ 39.5C (104F)

Evaporation Rate (BuAc=1):
ca. 7.7

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.

Hazardous Decomposition Products:
Carbon dioxide and carbon monoxide may form when heated to

decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Concentrated nitric and sulfuric acid mixtures, oxidizing materials, chloroform, alkalis, chlorine compounds, acids, potassium t-butoxide.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

11. Toxicological Information

Oral rat LD50: 5800 mg/kg; Inhalation rat LC50: 50,100mg/m3; Irritation eye rabbit, Standard Draize, 20 mg severe; investigated as a tumorigen, mutagen, reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC
Acetone (67-64-1)	No	No	N

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to readily biodegrade. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material is expected to readily biodegrade. When released to water, this material is expected to quickly evaporate. This material has a log octanol-water partition coefficient of less than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material may be moderately degraded by photolysis. When released into the air, this material is expected to be readily removed from the atmosphere by wet deposition.
Environmental Toxicity:
This material is not expected to be toxic to aquatic life. The LC50/96-hour values for fish are over 100 mg/l.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as

hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: ACETONE
Hazard Class: 3
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 350LB

International (Water, I.M.O.)

Proper Shipping Name: ACETONE
Hazard Class: 3.1
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 350LB

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\----------

Ingredient	TSCA	EC	Japan
Acetone (67-64-1)	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\----------

Ingredient	Korea	--Canada-- DSL	NDSL
Acetone (67-64-1)	Yes	Yes	No

--------\Federal, State & International Regulations - Part 1\------

Ingredient	-SARA 302- RQ	TPQ	------SARA List	Chem
Acetone (67-64-1)	No	No	Yes	

--------\Federal, State & International Regulations - Part 2\------

Ingredient	CERCLA	-RCRA- 261.33	-TS 8(

Acetone (67-64-1)	5000	U002	No

Chemical Weapons Convention: No TSCA 12(b): Yes CDTA: Yes
SARA 311/312: Acute: Yes Chronic: No Fire: Yes Pressure: No
Reactivity: No (Pure / Liquid)

Australian Hazchem Code: 2[Y]E
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

16. Other Information

NFPA Ratings: Health: 1 Flammability: 3 Reactivity: 0
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor.
Avoid contact with eyes, skin and clothing.
Label First Aid:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 8.
Disclaimer:
****

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Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

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MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

MALLINCKRODT J.T.Baker

24 Hour Emergency Telephone: 800-488-8351
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

NITRIC ACID 1.0 N AND 2.0 N VOLUMETRIC SOLUTIONS

MSDS Number: N3659 --- *Effective Date: 10/15/99*

1. Product Identification

Synonyms: Azotic acid solution; nitric acid 6.3%; nitric acid 1.0 N volumetric solution; nitric acid 2.0 N volumetric solution; nitric acid 12.6%
CAS No.: 7697-37-2
Molecular Weight: 63.00
Chemical Formula: HNO3 in H2O
Product Codes:
J.T. Baker: 5639
Mallinckrodt: 3510

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent
Nitric Acid	7697-37-2	6 - 13%
Water	7732-18-5	> 87%

3. Hazards Identification

Emergency Overview

POISON! DANGER! OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL

IF SWALLOWED. HARMFUL IF INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Poison)
Flammability Rating: 0 - None
Reactivity Rating: 3 - Severe (Oxidizer)
Contact Rating: 4 - Extreme (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: Yellow (Reactive)

Potential Health Effects

Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison.

Inhalation:
Corrosive! Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract.
Ingestion:
Corrosive! Swallowing nitric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract.
Skin Contact:
Corrosive! Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color.
Eye Contact:
Corrosive! Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Chronic Exposure:
Long-term exposure to concentrated vapors may cause erosion of teeth and lung damage. Long-term exposures seldom occur due to the corrosive properties of the acid.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders, eye disease, or cardiopulmonary diseases may be more susceptible to the effects of this substance.

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4. First Aid Measures

Immediate first aid treatment reduces the health effects of this substance.
Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

5. Fire Fighting Measures

Fire:
Not combustible, but substance is a strong oxidizer and its heat of reaction with reducing agents or combustibles may cause ignition. Can react with metals to release flammable hydrogen gas.
Explosion:
May react explosively with combustible organic or readily oxidizable materials such as: alcohols, turpentine, charcoal, organic refuse, metal powder, hydrogen sulfide, etc.
Fire Extinguishing Media:
If involved in a fire, use water spray.
Special Information:
Increases the flammability of combustible, organic and readily oxidizable materials. In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Neutralize with alkaline material (soda ash, lime), then absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting

spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker NEUTRASORB(R) or TEAM(R) 'Low Na+' acid neutralizers are recommended for spills of this product.

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect from physical damage and direct sunlight. Isolate from incompatible substances. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
For Nitric Acid:
OSHA Permissible Exposure Limit (PEL):
2 ppm (TWA)
ACGIH Threshold Limit Value (TLV):
2 ppm (TWA); 4 ppm (STEL)
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Nitric acid is an oxidizer and should not come in contact with cartridges and canisters that contain oxidizable materials, such as activated charcoal. Canister-type respirators using sorbents are ineffective.
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Colorless to yellowish liquid.
Odor:
Suffocating, acrid.
Solubility:
Infinitely soluble.
Specific Gravity:
No information found.
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100 (as water and acid)
Boiling Point:
No information found.
Melting Point:
No information found.
Vapor Density (Air=1):
No information found.
Vapor Pressure (mm Hg):
No information found.
Evaporation Rate (BuAc=1):
No information found.

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Containers may burst when heated.
Hazardous Decomposition Products:
When heated to decomposition, emits toxic nitrogen oxides fumes and hydrogen nitrate.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
A dangerously powerful oxidizing agent, concentrated nitric acid is incompatible with most substances, especially strong bases, metallic powders, carbides, hydrogen sulfide, turpentine, and combustible organics.
Conditions to Avoid:
Heat and incompatibles.

11. Toxicological Information

For Nitric Acid: Investigated as a mutagen and reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC
Nitric Acid (7697-37-2)	No	No	N
Water (7732-18-5)	No	No	N

12. Ecological Information

Environmental Fate:
No information found.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste facility. Although not a listed RCRA hazardous waste, this material may exhibit one or more characteristics of a hazardous waste and require appropriate analysis to determine specific disposal requirements. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)
Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 20L

International (Water, I.M.O.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)

Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 20L

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\----------

Ingredient	TSCA	EC	Japan
Nitric Acid (7697-37-2)	Yes	Yes	Yes
Water (7732-18-5)	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\----------

Ingredient	Korea	--Canada-- DSL	NDSL
Nitric Acid (7697-37-2)	Yes	Yes	No
Water (7732-18-5)	Yes	Yes	No

--------\Federal, State & International Regulations - Part 1\-------

Ingredient	-SARA 302- RQ	TPQ	List	------SARA Chem
Nitric Acid (7697-37-2)	1000	1000	Yes	
Water (7732-18-5)	No	No	No	

--------\Federal, State & International Regulations - Part 2\-------

Ingredient	CERCLA	-RCRA- 261.33	-TS 8(
Nitric Acid (7697-37-2)	1000	No	No
Water (7732-18-5)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: No Pressure: No
Reactivity: Yes (Mixture / Liquid)

Australian Hazchem Code: 2PE
Poison Schedule: S6
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

16. Other Information

NFPA Ratings: Health: 3 Flammability: 0 Reactivity: 0 Other: **Oxidizer**
Label Hazard Warning:
POISON! DANGER! OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED. HARMFUL IF INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.
Label Precautions:
Do not get in eyes, on skin, or on clothing.
Do not breathe vapor or mist.
Use only with adequate ventilation.
Wash thoroughly after handling.
Keep from contact with clothing and other combustible materials.
Store in a tightly closed container.
Label First Aid:
In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In all cases call a physician.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 3.
Disclaimer:
****

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Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

ATTACHMENT D

EQUIPMENT SOPs/CALIBRATION LOG

TITLE FIELD INSTRUMENTATION: PHOTOVAC MICROTIP HL-2000 CALIBRATION AND OPERATION

1.0 PURPOSE

This procedure is intended to provide general operational guidelines for the MicroTIP HL-2000. This instrument was designed as a handheld air monitor/photoionization detector. It measures and displays the concentration of airborne photoionizable gases and vapors. The MicroTIP layout is provided in Attachment 1.

2.0 SCOPE

This document applies to all OEES personnel involved in the operation, calibration, and maintenance of the MicroTIP HL-2000.

3.0 DEFINITIONS

ALARM Key - Shows set-point for concentration alarm

AUDIO Key - Selects alarm or tone or no audio

BATT Key - Shows battery voltage (normally 6-8.5V)

CAL Key - Calibrates with zero air and span gas

CLEAR Key - Erases the last number pressed

DISPLAY Key - Displays concentration as a graph or numeral

ENTER Key - Confirms the display and then continues

EVENT Key - Sets options for recording data

EXIT Key - Cancels the key with no more changes

GRAPH Key - Prints graph of the recorded data

Keypad - Used to set up and calibrate the MicroTIP, and allows the user to manipulate the concentrations measured and recorded in various ways. The MicroTIP has 16 keys for direct numeric entry and for using the instrument's functions.

LIGHT Key - Shows intensity of detector lamp

MAX Key - Displays highest concentration measured

PLAY Key - Replays recorded data on the display

PRINT Key - Prints table of recorded data

SETUP Key - Sets data time and options for keys

Span Gas - Gas that will be used to calibrate the MicroTIP. The span gas contains a known concentration of a photoionizable gas or vapor and is used to set the sensitivity. Usually isobutylene at 100 parts per million (ppm) in air is recommended as span gas.

TUTOR Key - Begins tutorial session (While in the tutorial, keypresses have no effect on MicroTIP's operation. Press the EXIT key to end the session.)

Zero Air - Contains no ionizable gases or vapors and is used to set the zero point of the MicroTIP during calibration. Usually clean ambient air will be suitable as zero air. However, if there is any doubt as to the quality of the ambient air, then a commercial source of zero grade air and a sampling bag must be used.

4.0 RESPONSIBILITIES

4.1 HEALTH AND SAFETY MANAGER (HSM)

The HSM is responsible for approving instrument procedures for issue and general implementation of this procedure.

4.2 Health And Safety Coordinator (HSC)

The HSC arranges for the training of personnel in the use of the instruments covered by the procedures.

4.3 Site Health And Safety Coordinator (SHSC)

The SHSC is responsible for general field implementation of the procedures, which includes instrument calibration, operation, maintenance, and recordkeeping.

5.0 PROCEDURES

5.1 Check Battery Voltage

Before any instrument is used by field personnel, the battery voltage will be checked.

Turn on the MicroTIP by pressing the back of the power switch. The pump will start and the instrument will need to warm up (less than three minutes). Press the BATT key. The normal operating voltage is between 6 and 8.5 volts.

5.2 Calibration

5.2.1 Parts Required

- Calibration kit, part no. 390033, consisting of:
  - Gas pressure regulator with contents gauge, to fit a 6D size cylinder of span gas
  - Gas sampling bag
  - Adapter tubing with fittings for regulator and MicroTIP inlet (attached to gas sampling bag)
- Span gas cylinder of isobutylene at 100 parts per million (ppm) ±5% in air.

5.2.2 Steps Required

The instrument should be calibrated at least once a day. Zero air is used to set the zero point. Clean, ambient air is suitable as zero air. Span gas is used to set the sensitivity.

Isobutylene at 100 ppm in air is recommended as span gas. To calibrate the instrument use the calibration kit as follows:

1. Connect the supplied regulator to the span gas cylinder (typically 100 ppm isobutylene). Hand tighten the fittings.

2. Open the valve on the gas bag by turning the valve stem fully counterclockwise.

3. Attach the nut to the regulator. Hand tighten the fittings.

4. Turn the regulator knob counterclockwise about half a turn to start the flow of gas.

5. Fill the gas bag about half full and then close the regulator. Turn the valve stem fully clockwise to stop the flow of gas.

6. Disconnect the bag from the adapter and press gently on the bag to empty it. Flush the bag a few more times with the span gas and then fill it with the span gas.

7. Close the gas bag by turning the valve clockwise.

8. Press SETUP and select the desired Cal Memory (based on which span gas is used) with the arrow keys and press ENTER. Press EXIT to leave setup.

9. Press CAL and enter the desired response factor (refer to the user's manual section on response factors for specific response factors of gases and vapors). If a specific compound is not being looked for, then enter 1.00. Section 5.3.3 of this SOP explains interpretation of results using the response factor.

10. Expose MicroTIP to zero air (clean, ambient air is suitable). Press ENTER and the instrument sets its zero point.

11. MicroTIP then asks for the span gas concentration. Enter the known span gas concentration and then connect the span gas bag adapter to the inlet.

12. Press ENTER and the instrument sets its sensitivity.

13. When the display reverts to normal, the instrument is calibrated and ready for use. Remove the span gas bag from the inlet.

14. Record readings on the Instrument Calibration Log (Attachment 2).

The MicroTIP has 10 Cal Memories and can be calibrated with 10 different span gases or response factors if desired. Only one Cal Memory can be used at a time. Each stores a different response factor, zero point, and sensitivity. To program:

1. Press SETUP and select the desired Cal memory (1-10) with the arrow keys.
2. Exit from SETUP and press the CAL key.
3. Enter the desired response factor and press ENTER.
4. Follow the displayed calibration instructions.

5.3 MICROTIP HL-2000 OPERATION

Check to see that the MicroTIP HL-2000 User's Manual is in the instrument case before taking the monitor in the field or shipping the device to the field.

The MicroTIP layout and normal display are shown in Attachment 1. Turn the instrument on by pressing the back of the power switch (see Attachment 1). The pump will start and the message "Warming up now, please wait" will be displayed. Within three minutes the following information will appear as part of the normal display (see Attachment 1): instrument status, current detected concentration, event number (if the datalogger is on), time, and date.

The MicroTIP operates automatically. The user reads the concentration directly from MicroTIP's display which updates itself each half second.

The minimum, maximum, and average concentrations measured are measured over 15-second periods and are automatically recorded in MicroTIP's datalogging memory if the datalogger is turned on. MicroTIP's memory holds 12 hours of concentration data.

All information entered from the keypad and stored in the MicroTIP memory is retained when the instrument is switched off. The clock and calendar continue to operate.

5.3.1 Specific Functions

The following is a description of each key function:

DISPLAY - If a numerical display is shown, pressing the DISPLAY key will change it to a bar graph. If the bar graph is shown, pressing the DISPLAY key changes it to a numerical display. The bar graph range is selected with the SETUP key.

LIGHT - LIGHT switches the backlighting between high and normal intensity and will turn backlighting off. The current intensity of the detector ultraviolet (UV) lamp is also displayed when the key is pressed. The brighter backlighting consumes more power and is recommended for use only in very dark locations.

BATT - Pressing the BATT key displays the current battery level. When "LoBat" is displayed there is approximately 10 minutes of operation left. To continue normal operation, replace the discharged battery pack with a fully charged one.

MAX - Maximum concentration is shown when the MAX key is pressed. The maximum concentration, event during which it was encountered, time, and date of the occurrence will be displayed for 15 seconds. By pressing the MAX key and then pressing the CLEAR key twice, the max register will be cleared.

CLEAR - If a number is entered in error, press CLEAR to erase the entry and re-enter the correct number.

EVENT - The EVENT key may be used to identify a particular sample or sampling location in memory. Recorded data may be displayed, printed, and removed from the datalogger by specifying a start and stop event number (refer to the MicroTIP User's Manual for complete instructions and limitations).

EXIT - EXIT terminates all MicroTIP functions except DISPLAY. When this key is pressed the display reverts to normal. Most functions exit automatically if no key is pressed for 15 seconds.

SETUP - SETUP allows the instrument to be set up for a specific application. The current date and time are also set with this key.

To set up the instrument:

1. Press the SETUP key.

2. The first option sets the full scale range for the bar graph display, graph output, audio output, and the 1 volt analog output. Use the up and down arrow keys to select the 20, 200, or 2000 ppm range and press ENTER. This does not affect the instrument's ability to read up to 2500 ppm.

3. Select Cal Memory 1 with the up and down arrow keys and press ENTER.

4. Enter the correct values for the current time, pressing ENTER after each value.

5. Enter the numerical values for the day, month, and year, pressing ENTER after each selection.

TUTOR - Pressing this key begins a tutorial session which displays a two-line description of the function of each key. Press each key and read the tutorial display. Pressing EXIT ends the tutorial session. While in the tutorial session, key presses have no effect on MicroTIP's operation.

AUDIO - To connect the headset (part no. 395030), remove the dust cap from the I/O connector and plug in the headset. Press the AUDIO key and use the arrow keys to select one of three options for audio output and press ENTER.

ALARM - Pressing ALARM will display the current alarm level and allows a new alarm level to be entered. If the alarm value is correct, press EXIT to return to the normal display. If a new value is to be set, enter the value and press ENTER.

PLAY - This key plays back previously recorded data.

1. Press PLAY and two options will be available. Pressing the ENTER key begins playback where it was last stopped. Press the SETUP (*) key to set the playback options.

2. Enter the start event.

3. Select which value is to be displayed.

4. The data can be played back in either numerical or graphical display by pressing the DISPLAY key.

When the instrument is playing back recorded data it is also measuring and recording real-time concentrations even though the instrument status is "play." Press EXIT to return to the normal display.

PRINT and GRAPH - PRINT key allows the data to be printed while the MicroTIP is measuring real-time concentrations. When used with a compatible printer, the GRAPH key prints the recorded data in graphical form. Refer to the MicroTIP User's Manual for complete instructions for these operations.

5.3.2 Detector Lamp Selection

The MicroTIP is supplied with a UV lamp that produces an energy of 10.6 electron-volts (eV). With this standard lamp installed, MicroTIP responds well to gases and vapors that ionize at 10.6 eV or less. If necessary, the MicroTIP may be equipped with lamps that produce different amounts of energy. The lamp selected must have an energy (in eV) greater than the ionization potential of the analyte it is detecting.

5.3.3 Interpreting the Results

The MicroTIP will indicate a 1:1 response ratio if exposed to the same gas as the gas to which it was calibrated. However, if the MicroTIP is exposed to a gas or vapor (or mixture of gases/vapors) that are not the same as the calibration gas, it will not detect at a 1:1 response ratio. When the MicroTIP has been calibrated to the span gas, the response factor is:

$$\frac{\text{known concentration of target gas}}{\text{displayed concentration of target gas (as a ppm -- equivalent)}}$$

A response factor may be used to relate a particular gas to the calibration gas (such as methane as compared to isobutylene as the calibration gas). Response factors can be input when calibrating the MicroTIP (see Section 5.2.2, Step 9). When computing the concentration, the MicroTIP multiplies the detected concentration by the response factor

and displays the result. If the response factor is 1.0 then the concentration is not changed. The instruction manual provides response factors for common gases that may be encountered.

If the MicroTIP response factor is set at 1.0 when using the instrument to detect gases or vapors other than the span gas, then the person reading the display should manually perform the following computation:

Actual airborne concentration of target gas =
displayed concentration x response factor of target gas

Record results on the Sir Air Surveillance Record (Attachment 3).

5.4 High Sensitivity Operation

The MicroTIP can be used as a high sensitivity leak detector for photoionizable chemicals. In High Sensitivity operation, MicroTIP does not read directly in ppm but displays a reading proportional to the concentration of photoionizable gases and vapors detected.

During high sensitivity calibration, no span gas is required. MicroTIP zeros its reading with zero air and then sets itself to the maximum sensitivity.

1. Press SETUP. Select the 0-20 ppm display range with the arrow keys and press ENTER.

2. Select High Sensitivity with the arrow keys and press ENTER.

3. Press EXIT. Select the bar graph with the DISPLAY key.

4. Press CAL and calibrate the MicroTIP with zero air. High Sensitivity operation does not require a span gas.

5. Press AUDIO and select "continuous audio" with the arrow keys.

5.5 Warning Limitations

The MicroTIP does not distinguish between individual pollutants. The reading displayed represents the total concentration of all photoionizable chemicals present in the sample.

This device is not intended for constant use with flammable concentrations of gases.

Calibration, maintenance, and servicing of this device, including battery charging, must be performed in a safe area away from hazardous locations.

Exercise care in handling battery packs in order not to short the terminals with conducting materials such as rings, bracelets, and keys. The battery or conductor may overheat and cause burns.

5.6 Accessories And Other Devices

The MicroTIP User's Manual contains detailed instructions for the following:

A. Printer
B. Computer
C. Chart recorder
D. Sample bag
E. 3-meter sample line (for remote sampling)
F. Shoulder strap
G. Headset
H. Replacement detector lamps

6.0 MAINTENANCE

6.1 Charging The Battery

When the instrument status reads "LoBat," the MicroTIP battery pack requires recharging. A fully charged battery powers the instrument for 7 hours. If the instrument is to be used for more than 7 hours, carry a spare battery pack. Approximately 8 hours is required to recharge a fully discharged battery pack (NOTE: use only the HL-2000 battery charger).

If the MicroTIP is left for more than three days without a charged battery, recorded data and setup parameters will be lost. To avoid loss of data, charge the battery pack for at least 8 hours and attach it back to the instrument.

Note: If MicroTIP is not used regularly, the battery should be charged at least once a month for between 8 and 72 hours.

6.2 Cleaning The Lamp Window

During the course of normal operation a film builds up on the window of the detector lamp. The window should be cleaned every 24 hours of operation using the following procedure:

1. Turn off the instrument.

2. Hold the black detector housing in one hand and unscrew it from the body of the instrument. Remove the housing, being careful not to lose the O-ring seal on top to the photoionization detector. The detector cell, lamp holder, and high frequency (HF) driver circuit board are now exposed.

3. Unplug the red and yellow wires from the HF driver circuit board.

4. Locate the black ground wire. Loosen the screw on the HF driver circuit board and disconnect the black wire.

5. Hold the lamp holder in one hand so it will not rotate and carefully unscrew the detector cell with the red, yellow, and black wires attached. (NOTE: Do not touch the fine wire mesh inside the detector cell).

6. Leaving the lamp spring in place, remove the lamp from the lamp holder.

7. To remove the film, gently rub the window of the lamp with a lint-free tissue moistened with HPLC or spectroscopic grade methanol.

8. Allow the window to dry. Then, using a lint-free tissue (touching the lamp as little as possible), replace it in the lamp holder.

9. Replace the detector cell squarely on the lamp holder. Finger tighten the detector cell, being careful not to overtighten.

10. Replace the black wire below the screw on the HF driver circuit board and tighten the screw down. Plug the yellow wire into the gold pin and the red wire into the silver pin on the HF driver circuit board.

11. Check the lamp holder and ensure it is securely seated by hand. Check that the O-ring seal is in position.

12. Replace the detector housing and tighten by hand.

6.3 Additional Maintenance

The MicroTIP User's Manual contains detailed instructions for the following:

A. replacing the detector UV lamp
B. replacing the inlet filter
C. replacing the sample pump

7.0 RECORDS

7.1 Procedure

A copy of each approved procedure and revision shall be retained as a permanent record in accordance with company procedures. Monitoring results will be documented on the Site Air Surveillance Record (Attachment 3).

7.2 Calibration

The Instrument Calibration Log (Attachment 2) will be maintained in the Health and Safety portion of the project files and/or on a log with the instrument.

8.0 SHIPPING FEDERAL EXPRESS

The MicroTIP does not contain any hazardous materials or components. However, some of the calibration gases used for calibrating the instrument are classified as hazardous materials.

Any person involved with the handling or transportation of hazardous materials or who has the potential to be exposed to hazardous materials must receive Department of Transportation (DOT) HM-126F training. Shipping labels must comply with DOT regulations. The following information will need to be listed on both the package being shipped and the paperwork accompanying the shipment (from 49 CFR Hazardous Materials, Table 172.101):

- proper shipping name
- hazard class
- identification number
- packing group
- labels required
- special provisions
- packaging authorization
- quantity limitations by air

9.0 REFERENCES

Photovac. 1992. MicroTIP HL-2000 User's Manual.

10.0 ATTACHMENTS

1. MicroTIP Layout and Normal Display
2. Instrument Calibration Log
3. Site Air Surveillance Record

ATTACHMENT 1

MICROTIP LAYOUT AND NORMAL DISPLAY

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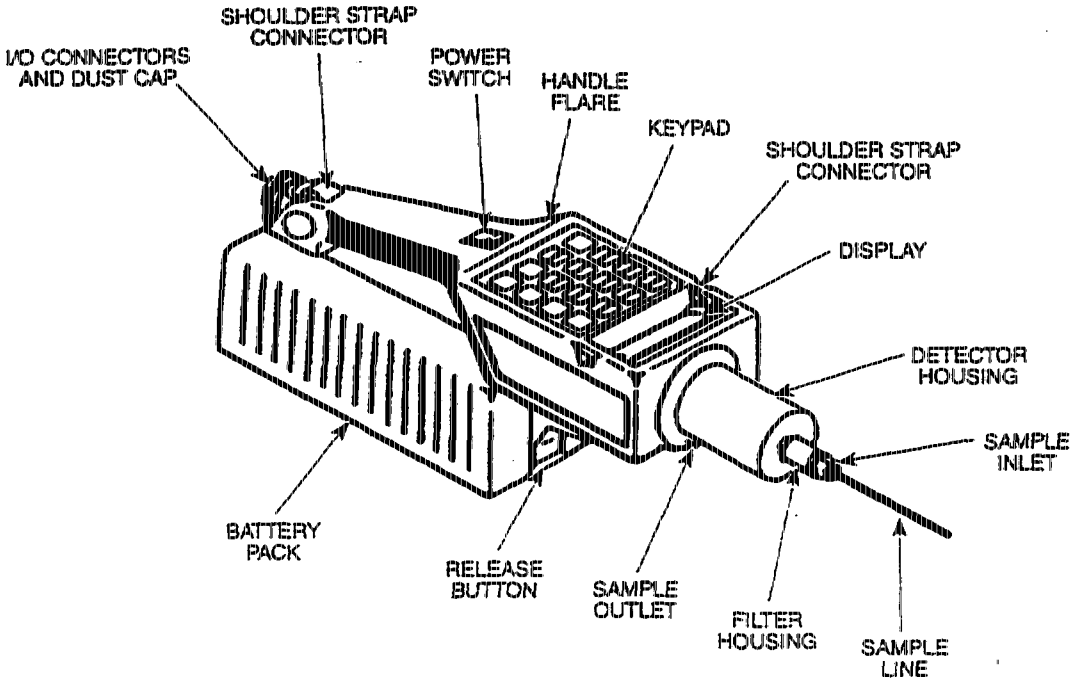
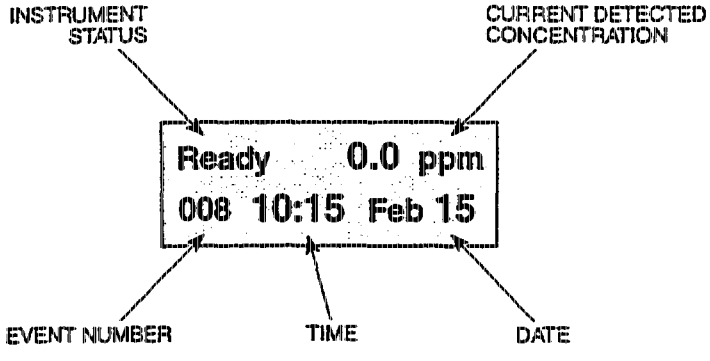


A. MicroTIP Layout



B. Normal Display

SOURCE: Photovac, "MicroTIP HL-2000 User's Manual"

OGDEN

MicroTIP Layout and Normal Display

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ATTACHMENT 2

INSTRUMENT CALIBRATION LOG

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INSTRUMENT CALIBRATION LOG

Project: ____________________

Location: ____________________

JOB #_ ____________________

Date/Time	Instrument	Serial Number	Span Gas Substance	Span Conc.	Instrument Reading at Calibration	[illegible]	Comments

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ATTACHMENT 3

SITE AIR SURVEILLANCE RECORD

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SITE AIR SURVEILLANCE RECORD

SITE INFORMATION		Page _____of___
JOB #: SITE MANAGER:	DATE: H&S COORDINATOR:	SITE LOCATION:

SITE CONDITIONS	SITE PERSONNEL:
TEMPERATURE: RELATIVE HUMIDITY: WIND SPEED & DIRECTION:	

SAMPLE NO.	TIME	SAMPLE DESCRIPTION	LOCATION	INSTRUMENT	READING	COMMENTS

OGDEN

SAMPLE NO.	TIME	SAMPLE DESCRIPTION	LOCATION	INSTRUMENT	READING	COMMENTS

OGDEN

TITLE

FIELD INSTRUMENTATION: THERMO ENVIRONMENTAL INSTRUMENTS INC. OVM MODEL 580B CALIBRATION AND OPERATION

1.0 PURPOSE

This procedure is intended to provide general operational guidelines for the Thermo Environmental Instruments Inc. organic vapor meter 580B. The 580B detects and quantitates most organic vapors with a photoionization detector. The 580B can detect up to 2000 parts per million (ppm).

2.0 SCOPE

This document applies to all OEES personnel involved in the operation, calibration, and maintenance of the 580B.

3.0 DEFINITIONS

Key Pad - There are seven switches on the key pad which operate the 580B. The switch marked ON/OFF is used to turn the pump and lamp on and off. The switch marked LIGHT will turn on backlighting for the two-line display. The other five switches perform various functions.

LIGHT switch - Illuminates the display.

MODE/STORE switch - Causes the 580B to return to the RUN mode. When the 580B is already in the RUN mode, it causes it to enter the LOG mode.

ON/OFF switch - Toggles the lamp and pump power between on and off.

Power Plug - The power plug is used to run the instrument off of its internal batteries. There is a chain attached to the power plug so that it will not be lost.

Pump - The 580B pump draws the sample into the detector.

Sample Exit Port - The 580B sample is drawn into the detector by a positive displacement pump and then sent back out through the exit port.

Sample Inlet - Sample is drawn into the detector through the sample inlet at the front of the 580B.

SPKR switch - Normally is used to toggle the instrument speaker between on and off.

4.0 RESPONSIBILITIES

4.1 HEALTH AND SAFETY MANAGER (HSM)

The HSM is responsible for approving instrument procedures for issue and general implementation of this procedure.

4.2 HEALTH AND SAFETY COORDINATOR (HSC)

The HSC arranges the training of personnel in the use of the instruments covered by this procedure.

4.3 SITE HEALTH AND SAFETY COORDINATOR (SHSC)

The SHSC is responsible for general field implementation of this procedure.

5.0 PROCEDURES

5.1 OPERATING

Attachment 1 depicts the operating features of the 580B.

Turn the OVM Model 580B on. When the instrument is first turned on the display will indicate that the lamp is not lit. Pressing the ON/OFF switch will tell the microprocessor to turn on the lamp and the pump. If the lamp is not lit after 14 seconds the microprocessor will indicate a "lamp out" condition. (If a lamp out condition is indicated check the seating of the lamp.)

Once the lamp is lit the display will show the concentration (in ppm) on the bottom line.

Turn the lamp and pump off by pressing the ON/OFF switch.

When the instrument is operated in RUN mode, a bar graph will show at the top of the screen to show a rough visual indication of the current concentration (in ppm). Remember, the concentration detected by the instrument are a ppm equivalent as compared to the calibrating gas. When the instrument is operated in the MAX HOLD mode, the top line of the display will indicate the maximum reading and the bottom line will indicate the current concentration. Whenever a new maximum is seen the top line will be updated. The MAX HOLD reading may be reset by pressing the RESET button while in the RUN mode.

Audible indication of concentration can be obtained by pressing the SPKR switch while in the RUN mode. The speaker will generate a "clicking" that will increase in speed as the concentration increases. The speaker rate can be changed by changing the switches located inside the side door. Only one of the four speaker rate switches should be on (in the down position) at any time. The 580B will display an overrange warning if the concentration goes above 2000 ppm. The top line of the display will show "overrange" when this occurs. The overrange warning will continue until the instrument is brought to an area with an organic vapor concentration of less than 20 ppm.

The alarm will sound if the concentration rises above the alarm setting. If the speaker is not activated then the alarm sound will not be heard. See Section 5.4 for the procedure for alarm setting. The alarm will turn off once the concentration drops below the alarm setting.

5.2 Run Mode Selection

The 580B has two run modes, MAX HOLD and CONCENTRATION METER. When the display reads "Conc. Meter" on the top line the bottom line will show "RESET TO CHG" and will alternate every two seconds with "MAX HOLD." In its current setup, the 580B is in the "MAX HOLD" mode. To select the RUN mode, press the RESET switch to cause the 580B to show "MAX HOLD +=USE/-=NO." Press the +/INC switch to select the "MAX HOLD" mode. To select the CONCENTRATION METER NORMAL mode, press the -/CRSR switch. The 580B will then return to the previous screen.

5.3 Average Time Selection

The 580B can be configured to display the average concentration from once a second up to once every four minutes. The display will show "AVERAGE = 0:01 RESET TO CHG." When the RESET switch is pressed the display will show "AVERAGE = 0:01 RESET WHEN DONE." Press the +/INC switch to increment the number above the cursor and the -/CRSR switch to move the cursor. The average time format is M:SS (where M is minutes and S is seconds).

5.4 Alarm Setting

The 580B has an alarm that will sound if the concentration rises above the alarm setting. The alarm will not be heard if the speaker has not been activated. The top line of the display will, however, indicate when there is an alarm condition. The 580B will display the current alarm setting on the top line of the display. The setting may be changed by simultaneously pressing the RESET switch with either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor. The alarm setting is entered in the Parameters mode.

5.5 Lamp Selection

The lamp setting can be changed depending on the lamp selected. Refer to the instruction manual for information on changing this setting.

5.6 Response Factor Setting

The 580B will indicate a 1:1 response ratio if exposed to the same gas as the gas to which it was calibrated. However, if the 580B is exposed to a gas or vapor (or mixture of gases/vapors) that are not the same as the calibration gas, it will not detect at a 1:1 response ratio. When the 580B has been calibrated to the span gas, the response factor is:

$$\frac{\text{known concentration of target gas}}{\text{displayed concentration of target gas (as a ppm-equivalent)}}$$

The current Response Factor Setting will be displayed on the top line of the display. Thus, a response factor may be used in order to relate a particular gas to the calibration gas. Response factors can be input when calibrating the MicroTIP. When computing the

concentration the microprocessor multiplies the detected concentration by the response factor and displays the result. If the response factor is 1.0 then the concentration is not changed. The instruction manual provides response factors for common gases that may be encountered.

If the 580B Response Factor Setting is set at 1.0 when using the instrument to detect gases/vapors other than the span gas, then the person reading the display should manually perform the following computation:

Actual airborne concentration of target gas =
displayed concentration x response factor of target gas

5.7 CALIBRATION

To calibrate the 580B, perform the following steps:

1. The 580B will display "RESET TO CALIBRATE." Press the RESET switch to enter the calibration mode.

2. The display will show "RESTORE BACKUP +=YES." The previous calibration information may be restored by pressing the +/INC switch. The 580B will then return to the previous screen. If the backup is not desired, press -/INC to continue the calibration routine.

3. The display will show "ZERO GAS RESET WHEN READY." Introduce the zero gas and press RESET. The 580B will then zero the instrument.

4. The display will then read "MODEL 580B ZEROING."

5. When zeroing is complete the display will show "SPAN PPM=0000." Then enter the span gas concentration by simultaneously pressing the RESET switch and either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor.

6. Once the span gas concentration has been entered the +/INC switch should be pressed.

7. The display will then read "SPAN GAS RESET WHEN READY." Introduce the span gas and then press RESET. The 580B will then calibrate the instrument and display "MODEL 580B CALIBRATING."

8. Once calibration is complete, the 580B will go back to the beginning and display "RESET TO CALIBRATE."

9. If, during the zeroing or calibrating of the 580B, a steady reading was not seen, then the 580B will display "CAL ERROR RESET WHEN READY."

10. Press RESET to return the 580B to zeroing or calibrating (depending on which it came from).

11. When calibration is completed, record the readings on an Instrument Calibration Log (Attachment 2).

5.8 Lamp Cleaning

Occasionally the lamp may need to be removed for cleaning. Refer to the instruction manual for information about lamp removal and insertion.

5.9 Low Battery

The 580B will display a warning when the battery is low. The warning will be a flashing B in the left-hand corner of the bottom line of the display when in the RUN mode.

5.10 Parameters Mode

All of the 580B operating parameters are entered in the Parameters mode. Calibration is also performed within the Parameters mode. To enter the Parameters mode, press the -/CRSR switch from the main menu. Pressing the +/INC switch will advance the 580B to the next selection or setting. Pressing the -/CRSR switch will advance the 580B to the previous selection/setting. Once a setting has been selected, the +/INC and -/CRSR switches will have different functions.

6.0 RECORDS

6.1 PROCEDURE

A copy of each approved procedure and revision shall be retained as a permanent record in accordance with company procedures. Monitoring results will be recorded on the Site Air Surveillance Record (Attachment 3).

6.2 CALIBRATION

Instrument Calibration Logs (Attachment 2) will be maintained in the health and safety portion of the project files and/or on a log with the instrument.

7.0 REFERENCES

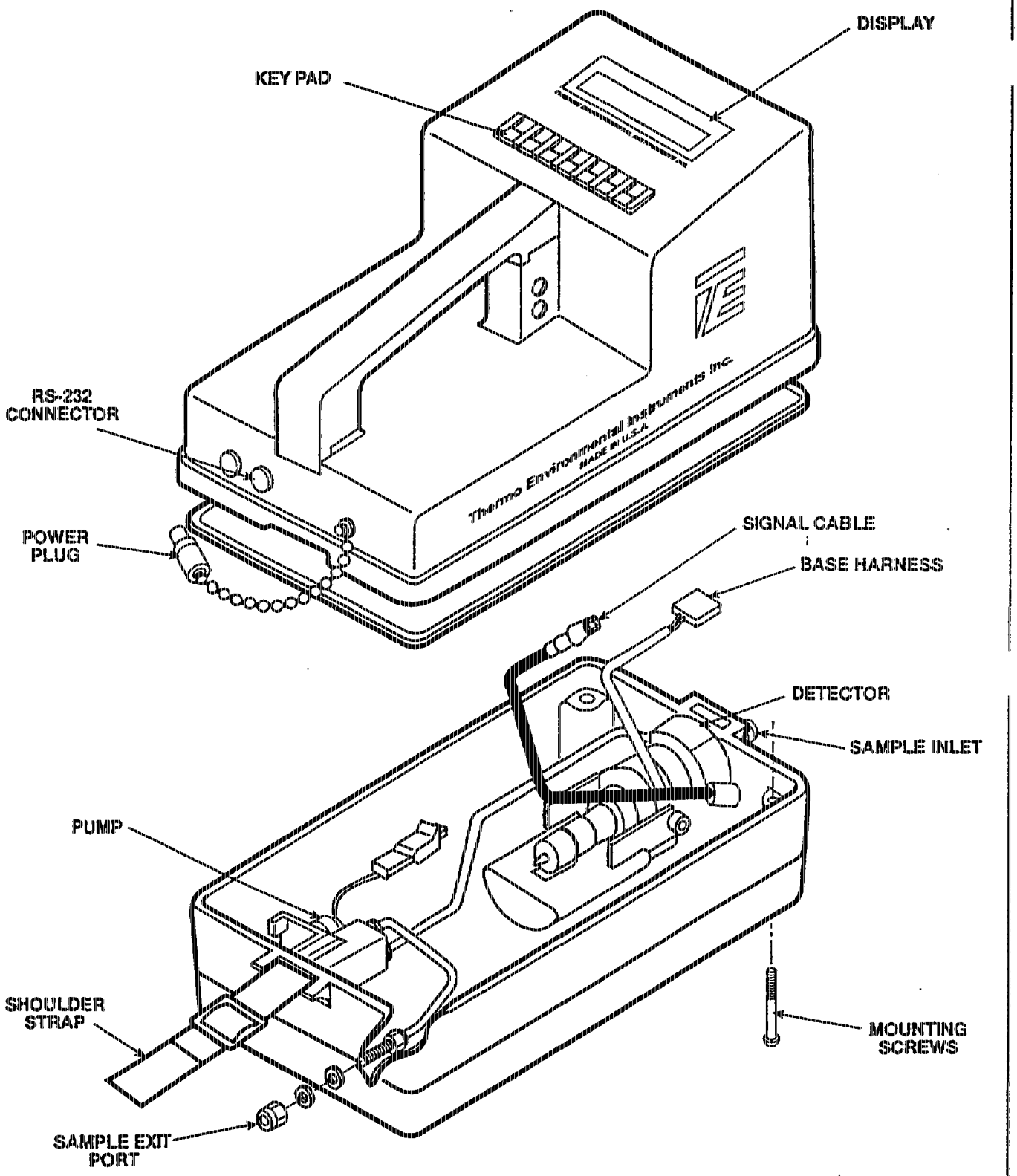
Thermo Environmental Instruments Inc. OVM/Datalogger Model 580B Instruction Manual.

8.0 ATTACHMENTS

1. Thermo 580B Operating Features
2. Instrument Calibration Log
3. Site Air Surveillance Record

ATTACHMENT 1

THERMO 580B OPERATING FEATURES


DISPLAY
KEY PAD
RS-232
CONNECTOR
POWER
PLUG
Thermo Environmental Instruments Inc.
MADE IN U.S.A.
SIGNAL CABLE
BASE HARNESS
DETECTOR
SAMPLE INLET
PUMP
SHOULDER
STRAP
MOUNTING
SCREWS
SAMPLE EXIT
PORT

ATTACHMENT 2

INSTRUMENT CALIBRATION LOG

OGDEN

INSTRUMENT CALIBRATION LOG

Project: ____________________

JOB #_ ____________________

Location: ____________________

Date/Time	Instrument	Serial Number	Span Gas Substance	Span Conc.	Instrument Reading at Calibration	Instrument Calibration Reading Prior Sampling	Comments

ATTACHMENT 3

SITE AIR SURVEILLANCE RECORD

SITE AIR SURVEILLANCE RECORD

SITE INFORMATION		Page ____of____
JOB #: SITE MANAGER:	DATE: H&S COORDINATOR:	SITE LOCATION:
SITE CONDITIONS TEMPERATURE: RELATIVE HUMIDITY: WIND SPEED & DIRECTION:	SITE PERSONNEL:	

SAMPLE NO.	TIME	SAMPLE DESCRIPTION	LOCATION	INSTRUMENT	READING	COMMENTS

ATTACHMENT E

HOSPITAL ROUTE MAP AND EMERGENCY CONTACTS

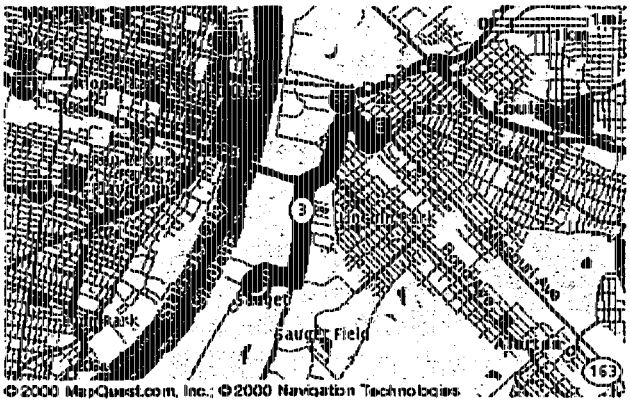
WWW.MAPQUEST.COM

Driving Directions Results

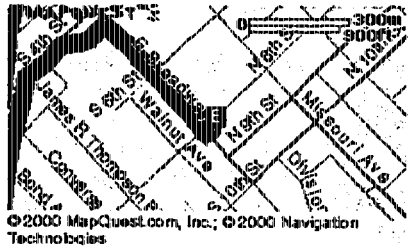
< Back SEND TO PRINTER

FROM:	TO:
MOBILE ST EAST SAINT LOUIS, IL 62201 US	100 N 8TH ST EAST SAINT LOUIS, IL 62201-2989 US

DIRECTIONS	DISTANCE
1: Start out going Northeast on MOBILE ST towards MONSANTO AVE by turning right.	0.2 miles (0.3 km)
2: Turn SLIGHT RIGHT onto MONSANTO AVE.	0.3 miles (0.5 km)
3: Turn LEFT onto IL-3.	0.9 miles (1.5 km)
4: Take the I-70 E/I-64 E/I-55 N ramp towards IL-3 N/CHICAGO/INDIANAPOLIS.	0.6 miles (0.9 km)
5: Keep RIGHT at the fork in the ramp.	0.2 miles (0.4 km)
6: Merge onto S 4TH ST.	0.2 miles (0.3 km)
7: Turn RIGHT onto E BROADWAY/IL-15.	0.3 miles (0.4 km)
8: Turn LEFT onto N 8TH ST.	0.0 miles (0.0 km)
TOTAL ESTIMATED TIME: 8 minutes	**TOTAL DISTANCE:** 2.7 miles (4.3 km)



TO:
100 N 8TH ST EAST SAINT LOUIS, IL 62201-2989 US



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Coupons Along Your Route

These directions are informational only. No representation is made or warranty given as to their content, road conditions or route usability or expeditiousness. User assumes all risk of use. MapQuest and its suppliers assume no responsibility for any loss or delay resulting from such use.

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EMERGENCY CONTACTS

Emergency resources are as follows:

Local Police Department:	Sauget	618-322-6507/6997
	Cahokia	618-337-5080
Local Fire Department:	Sauget	618-332-6700
	Cahokia	618-337-5080
Local Hospital:	St. Mary's Hospital	
	100 North 8th St.	
	East St. Louis, IL	618-274-1900
Poison Control Center:	1-800-942-5969	
USEPA National Response Center:	800-424-8802	

AMEC Contacts

Ecological Project Manager:	Chuck Harman	732-302-9500, ext. 127
Project Health and Safety Manager:	Jeffrey Tasca	732-302-9500, ext. 106
Corporate Health and Safety Manager:	Denise Daggett, CIH	858-458-9044

Solutia Contact

Project Manager:	Steven Smith	314-674-4922

ATTACHMENT F

HEALTH AND SAFETY ORIENTATION FORM

HEALTH AND SAFETY ORIENTATION FORM

(Visitors And Non-Routine Subcontractors)

I understand that I am entering a site where hazardous materials have been detected in the environmental media. Chemicals detected include, but are not limited to, polychlorinated biphenyls (PCBs), phenols, anilines, solvents, metals, and polyaromatic hydrocarbons (PAHs). In addition, due to the nature of the work being conducted on the site, physical hazards are present.

The information summarized in this sheet is important for you to read and fully understand. It has been extracted from the Site-specific Health and Safety Plan for the Sauget Area 2 Sites and has been compiled for your health and safety. If you have any questions regarding the information presented below, please ask your escort for identification.

Health, Safety, And Security Information

1. You must sign in and out of the Visitor Log Book maintained at the site. This assists in identifying all personnel at the site in the event of an emergency.

2. You must remain in the support ("clean") zone unless you have an escort. If you are observed alone in any unauthorized area, you will be asked to leave immediately.

3. Areas marked "Caution - DO NOT ENTER" demarcate where the Exclusion ("contaminated") areas begin. You are not authorized to enter these areas.

4. Access to the Exclusion Zone is strictly forbidden to ALL visitors unless they have written approval from the client and proof of adequate OSHA training and medical surveillance PRIOR to arrival on site.

5. Please read and follow all safety signs onsite which will alert you to possible physical and chemical hazards.

6. Eating and smoking are not allowed onsite. You may eat or smoke in designated areas only or in your vehicle.

7. There may be heavy equipment and/or moving vehicles. Please be aware.

8. No one under the age of 18 is permitted onsite without prior written approval from the client.

9. Report any accident or injury to your escort

10. No domestic animals are permitted onsite.

11. Please do not disrupt site activities or contribute to any unnecessary delays.

Emergency Notification

1. in the event of a site emergency, please walk immediately to the designated meeting area for the site. You will receive further instructions at this location. Please stay in this meeting area until the all-clear signal is given from the Field Team Leader or off-site emergency support personnel.

2. Please cooperate fully with those in authority in the event of an emergency.

ACKNOWLEDGEMENT OF INFORMATION

I have read and understand the above information provided by AMEC Earth & Environmental, Inc. and have had an opportunity to direct questions of a health and safety nature and have received adequate answers/explanations from my escort or other site staff member. My signature also indicates that my employer assumes the risk of any injury or property damage that may occur to me or by me.

Print Name	Signature	Affiliation	Date	Time In	Time Out