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LWG
LOWER WILLAMETTE GROUP

## Portland Harbor RI/FS

# ROUND 2
# QUALITY ASSURANCE PROJECT PLAN
# ADDENDUM 3: GROUNDWATER PATHWAY ASSESSMENT PILOT STUDY

December 16, 2004

**Prepared for:**
The Lower Willamette Group

**Prepared by:**
Kennedy/Jenks Consultants
Integral Consulting, Inc.

# SECTION A: PROJECT MANAGEMENT

## A1 TITLE AND APPROVAL SHEET

### PORTLAND HARBOR RI/FS
### ROUND 2 GROUNDWATER PATHWAY ASSESSMENT PILOT STUDY

Quality Assurance Plan Approvals

| | | | |
|---|---|---|---|
| U.S. EPA Project Manager: | Chip Humphrey | _____ | Date: _____ |
| U.S. EPA Project QA Manager | Ginna Grepo-Grove | _____ | Date: _____ |
| CERCLA Project Coordinator: | Keith Pine | _____ | Date: _____ |
| Integral Chemistry QA Manager: | Maja Tritt | _____ | Date: _____ |
| CAS Project Manager: | Abbie Spielman | _____ | Date: _____ |
| CAS Laboratory QA Manager: | Lee Wolf | _____ | Date: _____ |

## A2 TABLE OF CONTENTS

## A2.1 List of Tables

## A2.2 List of Acronyms

| | |
|---|---|
| ACG | Analytical concentration goals |
| CAS | Columbia Analytical Services |
| CERCLA | Comprehensive Environmental Response, Compensation, and Liability Act |
| COI | chemicals of interest |
| CVAA | cold vapor atomic absorption spectrometry |
| DQO | data quality objective |
| EPA | U.S. Environmental Protection Agency |
| ERA | ecological risk assessment |
| FSP | field sampling plan |
| GC/ECD | gas chromatography/electron capture detector |
| GC/FPD | gas chromatography/flame photometric detection |
| GC/LRMS | gas chromatography/low resolution mass spectrometry |
| GC/MS | gas chromatography/mass spectrometry |
| HHRA | human health risk assessment |
| ICP | inductively coupled plasma |
| ICP-AES | Inductively coupled plasma – atomic emission spectrometry |
| ICP/MS | inductively coupled plasma/mass spectrometry |
| ISA | initial study area |
| LWG | Lower Willamette Group |
| LWR | lower Willamette River |
| µg/L | micrograms per liter |
| µg/kg | micrograms per kilogram |
| MDL | method detection limit |
| mg/kg | milligrams per kilogram |
| mg/L | milligrams per liter |
| MID | multiple ion detection |
| MRL | method reporting limit |
| MSD | matrix spike duplicate |
| PAH | polycyclic aromatic hydrocarbon |
| PARCC | precision, accuracy, representativeness, completeness, and comparability |
| PSFSP | pilot study field sampling plan |
| QA/QC | quality assurance/quality control |
| QAPP | quality assurance project plan |
| RI/FS | remedial investigation/feasibility study |
| RM | river mile |
| RPD | relative percent difference |
| SIM | selected ion monitoring |
| SOP | standard operating procedure |
| SVOC | semivolatile organic compound |
| TIC | tentatively identified compound |
| VOC | volatile organic compound |

## A2.1 List of Attachments

## A3 DISTRIBUTION LIST

| | |
|---|---|
| U.S. EPA Project Officer: | Chip Humphrey |
| U.S. EPA Oregon Operations Office: | Eric Blischke |
| U.S. EPA Project QA Manager: | Ginna Grepo-Grove |
| Oregon Department of Environmental Quality: | Jim Anderson |
| NOAA: | Helen Hillman |
| U.S. Fish & Wildlife Service: | Ted Buerger |
| Oregon Department of Human Services | Dave Stone |
| Oregon Department of Fish & Wildlife: | Rick Kepler |
| Columbia River Inter-Tribal Fish Commission: | Patti Howard |
| Yakama Nation: | Paul Ward |
| Confederated Tribes of the Warm Springs Reservation of Oregon: | Brian Cunninghame |
| Confederated Tribes of the Umatilla Indian Reservation: | Audie Huber |
| Confederated Tribes of the Siletz Indians: | Tom Downey |
| Nez Perce Tribe: | Rick Eichstaedt |
| Confederated Tribes of the Grand Ronde Community of Oregon: | Rod Thompson |
| Environment International: | Valerie Lee |
| Port of Portland: | Jim McKenna |
| Port of Portland: | David Ashton |
| Northwest Natural: | Bob Wyatt |
| Anchor Environmental LLC: | Susan Thompson |
| Integral Project Manager: | Keith Pine |
| Integral Field Sampling and Analysis Coordinator: | Gene Revelas |
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# A4 INTRODUCTION AND PROJECT ORGANIZATION

## A4.1 Introduction

This quality assurance project plan (QAPP) addendum describes procedures that will be used to complete the Transition Zone water sampling included in the groundwater pathway assessment pilot study for Round 2 of the remedial investigation and feasibility study (RI/FS) for the Portland Harbor Superfund Site (Site) in Portland, Oregon. The groundwater pathway assessment pilot study field sampling plan (PSFSP) (Integral 2004a) describes the objectives and sampling methods to be used in the pilot study. The Round 2 QAPP describes procedures and requirements for the generation of data of documented acceptable quality that will be used for the RI/FS, including the ecological and human health risk assessments. This addendum supplements the Round 2 QAPP (Integral and Windward 2004) and addresses procedures that will be used for the Transition Zone water pilot study sampling that are not described in the Round 2 QAPP.

Supplemental information to Sections A and B of the Round 2 QAPP is provided in this QAPP addendum. Documents and records are described in Section A9 of the Round 2 QAPP and are not addressed further in this QAPP addendum.

Procedures for project assessment and oversight will be completed as described in Section C of the Round 2 QAPP, with one exception: Additional routine technical systems audits of analytical laboratories will not be required because all of the laboratories selected for this work have been audited in connection with Round 1 fish tissue analyses and Round 2 sediment analyses. Laboratories will be audited only if serious problems are encountered, as described in the Round 2 QAPP.

Procedures for data validation will be completed as described in Section D of the Round 2 QAPP.

The section numbers in this QAPP addendum correspond to U.S. Environmental Protection Agency (EPA) guidance for QAPP preparation (EPA 2002a) and, in most cases, correspond to the Round 2 QAPP.

## A4.2 Project and Task Organization

The organizational structure for activities associated with the Round 2 investigation is provided in Section A4.2 and Figure A4-1 of the Round 2 QAPP, with the following changes:

- The sampling and analysis coordinator for the pilot study will be Todd Martin of Integral.

- The field coordinator will be Nick Varnum of Integral.
- The Laboratory Project Manager will be Abbie Spielman of Columbia Analytical Services (CAS).

Contact information related to the Transition Zone water pilot study sampling is provided in Table A4-1 of this QAPP addendum.

## A5 PROBLEM DEFINITION AND BACKGROUND

An overview of the Portland Harbor RI/FS is provided in Section A5.1 of the Round 2 QAPP. A primary objective of the groundwater assessment pilot study is to assess the feasibility and effectiveness of methods for the collection of sediment Transition Zone water samples within zones of active groundwater discharge to the Willamette River.

The scope and anticipated schedule of the pilot study sampling efforts are discussed in the PSFSP (Integral 2004a). In summary, the pilot study sampling will be conducted in November – December 2004 at two sites within the ISA – ARCO Bulk Terminal (ARCO) and Arkema Chemicals Inc. (Arkema; formerly ATOFINA Chemicals Inc.). Details regarding site selection and background are included in the PSFSP.

Available methods for sampling Transition Zone water in riverbed sediments and the rationale for selecting methods for the pilot study are described in the PSFSP (Section 3.2). The following sampling methods were selected for the pilot study (see PSFSP Table 3-2 for a summary of the advantages and disadvantages of available methods):

### In-Situ Collection

*Diffusion-Based*

- Small-volume Peeper
- Large-volume Peeper
- Vapor Diffusion Sampler.

*Direct Sampling*

- Suction via Trident system
- UltraSeep (seepage meter)

### Ex-Situ Collection

*Sediment Sampling and Transition-Zone Water Extraction*

- Grab Sampling and Centrifuge.

Except for the vapor diffusion sampler, each of these methods will be used to sample Transition Zone water within the upper 1 foot of the sediment column at each of the pilot study locations (one location at ARCO and two locations at Arkema). The vapor diffusion sampler will be used to collect vapor samples at the two locations where volatile organic compounds (VOCs) are documented in groundwater. Results of the sampling effort will be compared to identify the benefits and limitations of the technologies and will be used to facilitate scoping of the future groundwater pathway assessment. In addition to Transition Zone water sampling via these methods, samples of underlying shallow groundwater will be collected at the ARCO site using a barge-mounted, direct-push drill rig and screen-point sampler.

## A6 TASK DESCRIPTION

The tasks to be completed for the pilot study include sample collection, field measurements, laboratory analyses, data quality evaluation, data management, and reporting. Summaries of field and laboratory tasks and references to detailed descriptions are provided in this section. Procedures for data quality evaluation, data management, and report preparation are described in the Round 2 QAPP.

### A6.1 Field Tasks

Six methods will be used during the pilot study to collect Transition Zone water:

- Small-volume Peeper
- Large-volume Peeper
- Trident Probe™
- UltraSeep
- Grab Sampling and Centrifuge Extraction.

One method will be used to collect underlying shallow groundwater:

- Direct-push.

In addition to Transition Zone water and shallow groundwater, vapor samples will be collected using a vapor diffusion technique, and bulk sediment samples will be collected for analysis.

### A6.2 Laboratory Analyses and Deliverables

Chemical analysis of Transition Zone water, shallow groundwater, vapor, and sediment samples will be completed by CAS (Kelso, WA). A summary of analyses for Transition Zone water, shallow groundwater, vapor and sediment samples is provided in Table A6-1. The analyte list for Transition Zone water and shallow groundwater, along with proposed pilot study analytical concentration goals (ACGs), method detection limits (MDLs), and method reporting limits (MRLs), is provided in Table A6-2. The analyte list for vapor samples and associated MRLs is provided in Table A6-3. Pilot study ACGs, MDLs, and MRLs for sediment analyses are included in Table A6-4.

Analyses will be completed using EPA methods and other established methods as indicated in Table A6-1. Laboratory data deliverables are described in the Round 2 QAPP.

### A6.3 Project Schedule

The pilot study sampling (hereinafter meant to include all four media to be collected) will be initiated following EPA's approval of the PSFSP and this QAPP addendum. The pilot study schedule will be strongly influenced by weather conditions and seasonal variations in the river temperature and water level. The schedule considerations due to these factors and limitations due to equipment availability are described in Section 5.0 of the PSFSP. The general timeframe for pilot study sampling is November – December 2004. Schedules for delivery of data and reports are described in the Programmatic Work Plan (Integral et al. 2004).

## A7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

### A7.1 The Data Quality Objective Process

Data quality objectives (DQOs) for the groundwater pathway assessment are described in the Round 2 QAPP. Final ACGs for the groundwater pathway assessment are under development in consultation with EPA and its partners. ACGs for the groundwater pilot study, however, have been proposed to identify analytical sensitivity levels that will be sufficient to evaluate risks to in-water ecological receptors that may be exposed to chemicals dissolved in groundwater flowing through the sediment transition zone. Although the pilot study ACGs can be met for many analytes, modifications to optimize laboratory MDLs and MRLs are not sufficient to meet the pilot study ACGs in all cases. (See Section B4.2 for additional discussion of method modifications.) MDLs, MRLs and ACGs for Transition Zone water and shallow groundwater in the pilot study are provided in Table A6-2.

## A7.2 Data Quality Indicators

The overall quality objective for Round 2 is to develop and implement procedures that will ensure the collection of representative data of known and acceptable quality. An objective of the pilot study is to evaluate which sampling methods yield data of acceptable quality for evaluation of the groundwater pathway. Some of the methods may not yield the optimum volume of Transition Zone water for analysis, which may result in elevated detection limits, reduction in the number of chemical analyses that can be completed, and/or fewer quality control samples analyzed. Additional detail on estimated sample volumes for the various sampling methods is provided in Section B3 of this QAPP addendum. The implications of the limited sample volumes on data quality and completeness will be a component of the overall evaluation of the pilot study methods. The QA procedures and measurements that will be used for this project are based on EPA guidance (EPA 1983, 1996b, 2004) and on established laboratory methods from other sources (APHA 1997).

QC samples and procedures are specified in each method protocol that will be used for this project. Methods are summarized in Table A6-1. All QC requirements will be completed by each laboratory as described in the protocols and in the Round 2 QAPP, unless limited by sample volume. Laboratory control limits for QC samples and procedures are provided in Tables A7-1 and A7-2 and in the laboratory QA manual (Appendix C of the Round 2 QAPP). Data validation criteria and procedures are described in Sections D1 and D2 of the Round 2 QAPP. During data validation, low recoveries will be evaluated for analytes with wide laboratory control limits to evaluate potential bias of the data. Data will be qualified on this basis if a bias is identified. PARCC parameters (i.e., precision, accuracy or bias, representativeness, completeness, comparability) are described in the Round 2 QAPP.

Target MRLs for this study are summarized in Tables A6-2 through A6-4. Laboratory methods are described in Section B4. MDLs will be determined by each laboratory for each analyte, as described in the Round 2 QAPP. MDLs are provided in Tables A6-2 through A6-4. The MDLs and MRLs are based on standard preparation and extraction volumes. If limited sample volumes are available from some of the water sampling methods, analyses will be prioritized and/or the volume available for laboratory QC samples will be reduced. Additional discussion on limited sample volumes and possible adjustments is provided in Section B3 below.

Analyte concentrations for pilot study samples will be reported to the MDL, as described in the Round 2 QAPP, except for vapor samples. Vapor sample results will be reported to the MRL, and detections below the MRL will be qualified as estimated values.

## A8 SPECIAL TRAINING/CERTIFICATION

In addition to requirements for experience and training provided in Section A8 of the Round 2 QAPP, training and certification requirements for the in-water pilot study field work include appropriate SCUBA certifications for divers and experience with Trident and UltraSeep equipment for operators of these devices. Trident and UltraSeep equipment will be operated by personnel from Coastal Monitoring. Christopher Smith, Jim Groves, and Ron Paulson, the team from Coastal Monitoring, are the inventors of the ultrasonic groundwater seepage meter. They have extensive experience deploying the Trident and UltraSeep equipment at various projects around the world. Further, they have over 70 years of combined experience working in the field of aquatic science, and all are certified scuba divers, including training by the U.S. Navy in dive operations.

# SECTION B: DATA GENERATION AND ACQUISITION

Section B of this Round 2 QAPP addendum includes the following supplemental sections:

- B1 – Sampling Process Design
- B2 – Sampling Methods
- B3 – Sample Handling and Custody
- B4 – Field and Laboratory Methods
- B5 – Quality Control.

Procedures related only to the instrumentation that will be used for field measurements of water quality conditions are additionally addressed in the following sections, while laboratory instrumentation is discussed in the Round 2 QAPP:

- B6 – Instrument/Equipment Testing, Inspection, and Maintenance
- B7 – Instrument/Equipment Calibration and Frequency.

The following information is provided in the Round 2 QAPP and is not addressed further in this QAPP addendum:

- B8 – Inspection/Acceptance of Supplies and Consumables
- B9 – Non-direct Measurements
- B10 – Data Management.

Details regarding field documentation for Transition Zone water, vapor, sediment and shallow groundwater sampling are provided in the PSFSP and in SOP H: Field Documentation, which will be submitted under separate cover.

## B1 SAMPLING PROCESS DESIGN

During the pilot study sampling, samples will be collected from three anticipated groundwater plume discharge locations in the Willamette River [one at ARCO and two at Arkema]. Details on the pilot study sampling design are provided in the PSFSP (Integral 2004a).

## B2 SAMPLING METHODS

Sample collection methods, equipment, and sample analysis requirements are summarized in the following sections. Additional details not provided here are included in the PSFSP (Integral 2004a) and in SOPs submitted separately to EPA and its partners. Sample containers, sample size requirements, preservation, and holding times for water, vapor and sediment samples and equipment rinse blanks are summarized in Table B2-1. Corrective actions are addressed in Sections B2.3 and C1 of the Round 2 QAPP.

### B2.1 Transition-Zone Water and Shallow Groundwater Sample Collection

Transition Zone water and/or shallow groundwater samples will be collected at each location by five methods, each briefly described below.

#### Trident Probe™

The Trident is a simple, direct-push system equipped with temperature and water sampling probes. The water sampling probe is used to collect in-situ water samples for Transition Zone water characterization. Transition Zone water is collected by syringe or vacuum pump extraction through a small-diameter, Teflon®-coated, stainless-steel probe (see FSP Figure 4-5). On the side of the tube near the tip, there is a sample port consisting of a slot covered by a small mesh (241-μm), stainless-steel screen. The Trident system can be deployed by hand near the surface, by push pole to depths of 30 to 40 feet, and by diver at greater depths. All Transition Zone water samples will be pumped into pre-preserved sample containers, which will be stored at 4°C until analysis. Detailed procedures are included in SOP B: Trident Probe Groundwater Discharge Mapping and Transition Zone Water Sampling.

#### UltraSeep

The UltraSeep system is a seepage meter that incorporates continuous flow measurement with automated sample collection. The meter relies on a Teflon®-coated, stainless-steel, open-bottomed chamber, measuring 48 x 46 cm, to funnel the seepage water to the flow sensor. The flow sensor is connected to the high point of the funnel via 12-mm Teflon® tubing, allowing free flow of water between the funnel and the outside environment. Based on the measured flow conditions, the water sampling system is activated to pump water to up to six sequential Teflon® sampling bags mounted around the perimeter of the meter. The UltraSeep systems will be diver-deployed and programmed to collect continuous flow record and multiple samples over the course of 1 to 4 days. At the end of the deployment period, divers will retrieve the UltraSeep systems and associated samples. All operational activities relating to the UltraSeep will be performed by Coastal Monitoring Associates, with oversight by Integral personnel. All samples will be pumped into appropriate pre-preserved sample bottles, which will be stored at 4°C until analysis. Detailed procedures are included in SOP C: UltraSeep Groundwater Discharge Flow

Measurement and Transition Zone Water Sampling, which will be submitted under separate cover.

### Small- and Large-Volume Peepers

The small-volume peeper is capable of collecting approximately 200 mL of water over a 38-cm sediment depth. The large-volume peepers will be specially constructed and can collect approximately 1,000 mL of water. Prior to deployment, both the small- and large-volume peepers will be filled with anoxic DI water spiked to roughly the equivalent salinity of the site Transition Zone water. Both peeper types will be deployed by divers. The small-volume peepers will be driven directly into the sediments to a total depth of approximately 1 foot. The large-volume peepers will be deployed by placing the peeper in a 7- to 8-inch deep hole excavated in the sediment and backfilling the hole with sediment. More than one of each type of peeper may be deployed at each location, if necessary, to achieve sufficient sample volume by compositing. All of the peepers will be left in place to equilibrate for a 3-week period. After this equilibration period, the peepers will be retrieved and brought to surface. Water will be extracted from each of the sample chambers of the small-volume peepers by inserting a needle through the membrane and extracting with a syringe. Each sample bottle will be filled with water from sample chambers distributed across the entire peeper to ensure that the sample is vertically representative of the entire 1-foot depth of sediment. Water will be transferred from the large-volume peepers by inserting a needle through the membrane, extracting the water with a syringe, and discharging the syringe into the appropriate, pre-preserved sample bottles. Once filled, the sample bottles will be stored at 4°C until analysis. Detailed procedures are included in SOP D: Diffusion-Based Transition Zone Water Sampling.

### Bulk Sediment Sampling and Centrifugation

Bulk sediment samples will be collected using a power grab sampler from the upper 30 cm of sediment at each of the sampling locations. The grab sampler will be brought to the surface, and sub-cores will be collected using butyrate tubing, also known as cellulose acetate butyrate (CAB) tubing. The subsamples will be shipped overnight to the analytical laboratory where the subsamples will be handled in an oxygen-free atmosphere. At the laboratory, some of the subsamples will be sectioned and centrifuged for Transition Zone water extraction. The remaining subsamples will be used for analysis of bulk sediment COI concentrations. Detailed procedures are included in SOP E: Bulk Sediment and Ex-Situ Transition Zone Water Extraction. The CAS procedure for extraction of Transition Zone water from bulk sediment is included as Attachment 1 to this QAPP Addendum. The remaining subsamples may be used for analysis of bulk sediment chemical of interest (COI) concentrations, as described below.

The sediment remaining in the grab sampler following subsample collection will be inspected, and the following physical characteristics of the sediment will be recorded

on field logs or sample description forms: sediment texture; sediment color; presence, type, and strength of odors; core penetration depth (nearest cm); degree of leakage or sediment surface disturbance; and any obvious abnormalities such as wood/shell fragments or large organisms.

### B2.2 Vapor Sample Collection

Vapor samples will be collected by the vapor diffusion method. The vapor diffusion samplers, consisting of three uncapped, 40-mL VOA vials sealed inside two layers of zip-seal bags, will be deployed at the same time as the peeper samplers. Each vapor diffusion sampler will be pushed into the sediment to an approximate depth of 6 inches and allowed to equilibrate for a 3-week period prior to retrieval. Upon return of the samplers to the surface, the outer zip-seal bag will be removed and a septum cap will be crimp-sealed over the bottle with the inner bag still in place. Detailed procedures are included in SOP D: Diffusion-Based Transition Zone Water Sampling.

### B2.3 Shallow Groundwater Sample Collection

In addition to Transition Zone water and vapor diffusion samples, shallow underlying groundwater will be collected using the direct-push method. Direct-push borehole pairs will be installed at each of the three transition-zone water sampling stations identified for the ARCO site. An initial borehole will be advanced to a minimum depth of 15 feet and maximum depth of 40 feet to collect continuous sediment core samples for visual inspection and logging. Logging results will be used to identify potential groundwater discharge zones and define target depths for shallow groundwater sampling. The target depths will be selected such that the two intervals sample physically distinct groundwater intervals, if possible. Following termination of the first borehole, a second borehole will then be advanced nearby (1-2 feet away) to collect discrete groundwater samples from the target sampling depths. Samples will be pumped to the surface using a peristaltic pump. Three pore volumes of the screen point sampler and tubing will be purged using a peristaltic pump prior to collection of the samples. All samples will be pumped into appropriate pre-preserved sample bottles, which will be stored at 4°C until analysis. Detailed procedures are included in SOP F: Geoprobe® direct-push sampling.

Sampling will follow the guidelines in EPA Method 1669, *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA 1996a), and in the *Field Sampling Manual for the Regional Monitoring Program for Trace Substances* (David et al. 2001).

## B3 SAMPLE HANDLING AND CUSTODY

Sample custody procedures are described in Section B3 of the Round 2 QAPP (Integral and Windward 2004). Sample preservation and storage requirements and

holding times for the water, vapor, and sediment samples are provided in Table B2-1 of this QAPP addendum. SOP I: Sample Labeling, Custody, and Handling, describes these procedures and will be submitted under separate cover.

Some of the Transition Zone water sampling methods will collect a limited volume of water that may not provide enough to meet the standard volumes submitted for analysis. Table B3-1 provides information on prioritization of analyses and target volumes that will allow analyses to be conducted for COIs with method modifications, such as reduction of sample size and final extract volume, or with elimination of laboratory QC samples (such as duplicates and matrix spikes) and/or volume for reanalysis. Based on the target volumes shown in Table B3-1 and information from Table 4-1 of the PSFSP, the number of samples per sampling method and analytical method for each of the pilot study locations is shown in Table B3-2.

## B4 FIELD AND LABORATORY METHODS

### B4.1 Field Measurements

Measurements of temperature, conductivity, and pH will be taken at Trident Probe and UltraSeep sampling stations. Measurements of conductivity, pH, temperature, dissolved oxygen, and oxidation-reduction potential will be taken at Geoprobe® direct-push sampling locations using a YSI Multi Probe and flow-through cell. Details regarding the operation of these instruments, including calibration, measurement, quality control procedures, and decontamination, are provided in the PSFSP SOPs.

### B4.2 Laboratory Analyses

Laboratory methods to be used for Round 2 are consistent with requirements provided in EPA method protocols (EPA 1983, 1996b, 2004). Analytes, MDLs, MRLs, and ACGs for water and sediment samples are provided in Tables A6-2 and A6-4, respectively. Analytes and MRLs for vapor samples are provided in Table A6-3. MRLs and ACGs for analytes to be analyzed in the pilot study sediment samples are provided with MRLs and ACGs for all Round 2 sediment analytes in the Round 2 QAPP (Integral and Windward 2004). ACGs will not be reached for some analytes due to technical limitations. MRLs may be elevated for additional analytes for some sampling methods if sample volumes are limited. Modifications will be made to these methods, as necessary and technically feasible, to improve MRLs. Method modifications will not be sufficient to reduce MRLs to the level of the ACGs for several analytes, and ACGs will not be attained in these cases. MRLs achievable for the various sampling methods will be one factor considered in the evaluation of pilot study results.

Analytical methods and modifications for pilot study water and vapor samples are described below. Methods and modifications for sediment samples are described in the Round 2 QAPP (Integral and Windward 2004).

Water samples will be analyzed for a subset of the following analytes, depending on the location and sampling method:

- Metals (arsenic, chromium, copper, lead, manganese, and zinc)
- Cations (calcium, magnesium, potassium and sodium)
- Conventionals (chloride, sulfate, alkalinity and pH)
- Perchlorate
- Organochlorine pesticides (DDT, DDD, DDE)
- Polycyclic aromatic hydrocarbons (PAHs)
- Total petroleum hydrocarbons (TPH) [diesel-range organics (TPH-diesel) and gasoline-range organics (TPH-gas)]
- VOCs [analyte list as provided in Integral and Windward (2004), plus 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,2,4-trichlorobenzene, and hexachlorobutadiene].

Vapor samples will be analyzed for VOCs only (benzene, toluene, ethylbenzene, xylenes at ARCO; chlorobenzene at Arkema).

Bulk sediment samples will be analyzed for a subset of the following analytes, depending on the location:

- Metals (arsenic, chromium, copper, lead and zinc)
- Perchlorate
- Organochlorine pesticides (DDT, DDD, DDE)
- PAHs
- TPH [diesel-range organics (TPH-diesel) and gasoline-range organics (TPH-gas)]
- VOCs [analyte list as provided in Integral and Windward (2004), plus 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,2,4-trichlorobenzene, and hexachlorobutadiene].

The total number of samples and the analyses that will be conducted on each sample are indicated in Table B3-2. The laboratory methods for sample preparation and analysis are summarized in Table A6-1.

### B4.2.1 Metals and Cations in Transition Zone Water and Shallow Groundwater

The methods that will be used to prepare and analyze Transition Zone water and shallow groundwater samples for metals and cations are shown in Table A6-1. Digestion with nitric and hydrochloric acids will be used to prepare samples for analysis of metals. Analysis for these metals will be completed by inductively coupled plasma/mass spectrometry (ICP/MS).

### B4.2.2 Conventional Analyses in Transition Zone Water and Shallow Groundwater

Conventional analyses of surface water samples will include chloride, sulfate, alkalinity as $CaCO_3$, and pH. Perchlorate will also be analyzed on samples from the Arkema Chlorate Plant area. EPA methods will be used as shown in Table A6-1.

Alkalinity as $CaCO_3$ will be measured using titration according to EPA Method 310.1, and pH will be measured electrometrically according to EPA Method 150.1.

Chloride and sulfate will be analyzed according to EPA Method 300.0. Perchlorate will be analyzed according to EPA Method 314.0. Samples will be filtered at the laboratory as specified in the method and analyzed by ion chromatography.

### B4.2.3 Organic Compounds in Transition Zone Water and Shallow Groundwater

Organochlorine pesticides will be extracted from samples using continuous liquid-liquid extraction procedures. Samples will be analyzed by gas chromatography/electron capture detector (GC/ECD). Florisil® column clean-up will be performed on the sample extracts.

Sample extractions for PAHs will be completed using continuous liquid-liquid extraction. Analyses for PAHs will be completed by gas chromatography/mass spectrometry (GC/MS) with a large-volume injector. Selected ion monitoring (SIM) will be used to enhance sensitivity.

VOC analyses will be conducted according to EPA Method 8260B, purge-and-trap followed by GC/MS analysis.

### B4.2.3 VOCs in Vapor Samples

Vapor samples will be analyzed by EPA Method TO-15. The samples will be preconcentrated using cold traps in a two-step cryofocusing procedure. Analysis will be completed using GC/MS.

## B5 QUALITY CONTROL

QC samples will be prepared in the field and at the laboratories to monitor the bias and precision of the sample collection and analysis procedures.

Field QC samples for this study will include field replicates, equipment rinse blanks, decon blanks, and temperature blanks. A summary of field QC samples that will be collected for each sampling method at each location is provided in Table B5-1.

Field replicates, equipment rinse blanks, and temperature blanks are described in the Round 2 QAPP. Prior to the start of sample collection activities for each sampling event, a decon blank will be generated by the laboratory that conducts decontamination of the peristaltic pump sampling equipment, as described in Appendix B of the Round 2 QAPP. The decon blank will be analyzed for metals and organic parameters.

Field QC samples to be collected at each location for each sampling method and analysis are summarized in Table B5-1. Replicate samples will be collected for appropriate analytical methods at each pilot study location for all sampling methods except the UltraSeep method. Equipment rinsate blanks will be collected at each pilot study location for applicable analytical methods (excluding conventionals and cations) for equipment that is decontaminated and reused for sample collection. These methods include the Trident Probe and UltraSeep for Transition Zone water collection, direct-push for shallow groundwater collection, and the bowl and spoon used to composite bulk sediment samples. An equipment blank will not be required for the power grab sampler because the sediment that contacts the sampler will not be collected. Prior to the start of fieldwork for each sampling event, decon blanks will be analyzed to ensure that the decontamination procedure used for pre-cleaned sampling equipment (peepers, butyrate tubing for collecting sediment subsamples from the grab sampler, discharge tubing, etc.) is adequate. The procedures for decon blanks are described in the PSFSP SOPs.

Laboratory QC samples and procedures will be completed as described in the Round 2 QAPP and the various method protocols (Table A6-1).

## B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Maintenance of the instruments that will be used for field measurements will be completed as described in the manufacturer's instructions.

Testing, inspection, maintenance, setup, and calibration of laboratory instruments are described in the Round 2 QAPP.

## B7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Calibration checks on instruments to be used for field measurements (i.e., conductivity, pH, temperature, dissolved oxygen, and oxidation reduction potential) will be performed prior to use each day that measurements will be made, as described in the PSFSP SOPs and in the manufacturer's instructions. Calibration checks will be completed twice daily at a minimum. The instruments will be checked for calibration before each daily sampling begins and again at the end of the day. If measurements are made at more than 10 stations in a single day, calibration checks will be made after every 10 stations are measured and at the end of the day. Calibration information will be recorded in the field logbook.

Calibration procedures for laboratory instruments are described in the Round 2 QAPP.

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# TABLES

Table A4-1. Project Team Contact Information.

| Name | Project Role | Phone | Fax | Email |
|---|---|---|---|---|
| **EPA Region 10** | | | | |
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Table A6-1. Laboratory Methods for Water, Vapor, and Sediment Samples.

| Analytes | Laboratory | Sample Preparation | | Quantitative Analysis | |
|---|---|---|---|---|---|
| | | Protocol | Procedure | Protocol | Procedure |
| **Water samples** | CAS Kelso | | | | |
| Conventional Analyses | | | | | |
| pH | | None | -- | 150.1 | Electrometric |
| Chloride | | 300.0 | Filtration, 0.45 um filter, if needed | 300.0 | Ion chromatography |
| Sulfate | | 300.0 | Filtration, 0.45 um filter, if needed | 300.0 | Ion chromatography |
| Alkalinity as $CaCO_3$ | | None | -- | 310.1 | Titration |
| Perchlorate | | EPA 314.0 | Filtration, 0.45 um filter, if needed | EPA 314.0 | Ion chromatography |
| Metals | | | | | |
| Arsenic, copper, chromium, lead, zinc | | EPA 3005 | Acid digestion | EPA 200.8 | ICP/MS |
| Calcium, magnesium, manganese, potassium, sodium | | EPA 3005 | Acid digestion | EPA 6010B | ICP/OES |
| Volatile Organic Compounds | | | | | |
| Full project list | | EPA 5035 | Purge and trap | EPA 8260B | GC/MS |
| BTEX | | EPA 5035 | Purge and trap | EPA 8260B | GC/MS |
| Total Petroleum Hydrocarbons | | | | | |
| TPH-gas | | NWTPH-Gx | Purge and trap | NWTPH-Gx | GC/FID |
| TPH-diesel | | NWTPH-Dx | Extraction | NWTPH-Dx | GC/FID |
| Organochlorine Pesticides | | | | | |
| DDT, DDE, DDD | | EPA 3520C<br>EPA 3630C | Continuous liquid-liquid extraction<br>Florisil® cleanup | EPA 8081A | GC/ECD |
| Semivolatile Organic Compounds | | | | | |
| PAHs | | EPA 3520C | Continuous liquid-liquid extraction | EPA 8270C | GC/MS-SIM |
| **Vapor Samples** | CAS Simi Valley | | | | |
| Volatile Organic Compounds | | | | | |
| Full project list | | EPA TO-15 | Cold traps | EPA TO-15 | GC/MS |
| BTEX | | EPA TO-15 | Cold traps | EPA TO-15 | GC/MS |

Table A6-1. Laboratory Methods for Water, Vapor, and Sediment Samples.

| Analytes | Laboratory | Sample Preparation | | Quantitative Analysis | |
|---|---|---|---|---|---|
| | | Protocol | Procedure | Protocol | Procedure |
| **Sediment Samples** | CAS Kelso | | | | |
| Conventional Analyses | | | | | |
| Total solids | | NA | -- | PSEP 1986 | Balance |
| Perchlorate | | Laboratory SOP | Deionized water leach | EPA 314.0 | Ion chromatography |
| Metals | | | | | |
| Arsenic, lead | | EPA 3050 | Strong acid digestion | EPA 6020 | ICP/MS |
| Chromium, copper, zinc, calcium, magnesium, manganese, potassium, sodium | | EPA 3050 | Strong acid digestion | EPA 6010B | ICP-OES |
| Organochlorine Pesticides | | | | | |
| DDT, DDE, DDD | | EPA 3540<br>EPA 3620B<br>EPA 3660B | Soxhlet extraction<br>Florisil® cleanup<br>Sulfur cleanup | EPA 8081A | GC/ECD |
| Volatile Organic Compounds | | | | | |
| Full project list | | EPA 5035 | Purge and trap | EPA 8260B | GC/MS |
| BTEX | | EPA 5035 | Purge and trap | EPA 8260B | GC/MS |
| Total Petroleum Hydrocarbons | | | | | |
| TPH-gas | | NWTPH-Gx | Methanol extraction; Purge and trap | NWTPH-Gx | GC/FID |
| TPH-diesel | | NWTPH-Dx | Sonication extraction; Silica Gel Cleanup (as needed) | NWTPH-Dx | GC/FID |
| Semivolatile Organic Compounds | | | | | |
| Polycyclic Aromatic Hydrocarbons | | EPA 3550B<br>EPA 3640A<br>EPA 3630C | Sonication extraction<br>Gel permeation chromatography<br>Silica gel cleanup | EPA 8270C | GC/MS - SIM |

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting and Detection Limits for Groundwater and Transition-Zone Water Samples.

| Analyte | MRL | MDL | NRWQC[1] | ORNL [2] | ACG[3] | Ratio of MRL to AGC | Ratio of MDL to ACG |
|---|---|---|---|---|---|---|---|
| **Conventional Analyses, µg/L (ppb)** | | | | | | | |
| Chloride | 200 | 40 | 230000 | | 230000 | 0.0009 | 0.0002 |
| Sulfate | 200 | 40 | | | NE | NA | NA |
| Alkalinity as $CaCO_3$ | 2000 | 1000 | 20000 | | 20000 | 0.1000 | 0.0500 |
| Perchlorate | 2 | 0.5 | | | NE | NA | NA |
| **Metals/Inorganics, mg/L (ppm)** | | | | | | | |
| Arsenic | 0.005 | 0.002 | 0.150 | 0.914 | 0.150 | 0.0333 | 0.0133 |
| Calcuim | 0.05 | 0.03 | | 116 | 116 | 0.0004 | 0.0003 |
| Chromium[4] | 0.0002 | 0.00006 | 0.011 | 0.002 | 0.002 | 0.1000 | 0.0300 |
| Copper[5] | 0.0001 | 0.00004 | 0.0027 | 0.00023 | 0.00023 | 0.4348 | 0.1739 |
| Lead[5] | 0.00002 | 0.00001 | 0.0005 | 0.01226 | 0.0005 | 0.0370 | 0.0185 |
| Magnesium | 0.02 | 0.02 | | 82 | 82 | 0.0002 | 0.0002 |
| Manganese | 0.05 | 0.01 | | <1.1 | NE | NA | NA |
| Potassium | 2.00 | 0.7 | | 53 | 53 | 0.0377 | 0.0132 |
| Sodium | 0.10 | 0.07 | | 680 | 680 | 0.0001 | 0.0001 |
| Zinc[5] | 0.0005 | 0.0002 | 0.0365 | 0.03 | 0.03 | 0.0167 | 0.0067 |
| **Organochlorine Pesticides, µg/L (ppb)** | | | | | | | |
| 4,4'-DDE | 0.01 | 0.003 | | | NE | NA | NA |
| 4,4'-DDD[6] | 0.01 | 0.004 | | 1.69 | 1.69 | 0.00592 | 0.00237 |
| 4,4'-DDT | 0.01 | 0.005 | 0.001 | 0.3 | 0.001 | 10.000 | 5.000 |
| **Total Petroleum Hydrocarbons** | | | | | | | |
| TPH-gas | 250 | 12 | | | NE | NA | NA |
| TPH-diesel (DRO) | 250 | 36 | | | NE | NA | NA |
| **Volatile Organic Compounds, µg/L (ppb)** | | | | | | | |
| Acetone | 20 | 1.8 | | 507640 | 507640 | 0.0000 | 0.0000 |
| Acrolein | TBD | TBD | | | NE | NA | NA |
| Acrylonitrile | TBD | TBD | | | NE | NA | NA |

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting and Detection Limits for Groundwater and Transition-Zone Water Samples.

| Analyte | MRL | MDL | NRWQC[1] | ORNL[2] | ACG[3] | Ratio of MRL to AGC | Ratio of MDL to ACG |
|---|---|---|---|---|---|---|---|
| Benzene | 0.5 | 0.11 | | 525000 | 525000 | 0.0000 | 0.0000 |
| Bromochloromethane | 0.5 | 0.12 | | | NE | NA | NA |
| Bromodichloromethane | 0.5 | 0.08 | | | NE | NA | NA |
| Bromoform | 0.5 | 0.09 | | | NE | NA | NA |
| Bromomethane | 0.5 | 0.17 | | | NE | NA | NA |
| 2-Butanone | 20 | 1.6 | | 282170 | 282170 | 0.0001 | 0.0000 |
| Carbon Disulfide | 0.5 | 0.12 | | 244 | 244 | 0.0020 | 0.0005 |
| Carbon Tetrachloride | 0.5 | 0.14 | | 1970 | 1970 | 0.0003 | 0.0001 |
| Chlorobenzene | 0.5 | 0.09 | | 1203 | 1203 | 0.0004 | 0.0001 |
| Chlorodibromomethane | TBD | TBD | | | NE | NA | NA |
| Chloroethane | 0.5 | 0.2 | | | NE | NA | NA |
| 2-Chloroethyl Vinyl Ether | TBD | TBD | | | NE | NA | NA |
| Chloroform | 0.5 | 0.1 | | 1240 | 1240 | 0.0004 | 0.0001 |
| Chloromethane | 0.5 | 0.14 | | | NE | NA | NA |
| trans-1,4-Dichloro-2-butene | TBD | TBD | | | NE | NA | NA |
| 1,2-Dibromoethane | 2 | 0.1 | | | NE | NA | NA |
| Dibromomethane | 0.5 | 0.09 | | | NE | NA | NA |
| 1,2-Dichlorobenzene | 0.5 | 0.09 | | | NE | NA | NA |
| 1,3-Dichlorobenzene | 0.5 | 0.11 | | | NE | NA | NA |
| 1,4-Dichlorobenzene | 0.5 | 0.09 | | | NE | NA | NA |
| 1,1-Dichloroethane | 0.5 | 0.1 | | 14680 | 14680 | 0.0000 | 0.0000 |
| 1,2-Dichloroethane | 0.5 | 0.09 | | 15200 | 15200 | 0.0000 | 0.0000 |
| 1,1-Dichloroethene | 0.5 | 0.14 | | >2800 | >2800 | 0.0002 | 0.0001 |
| trans-1,2-Dichloroethene | 0.5 | 0.1 | | 9538 | 9538 | 0.0001 | 0.0000 |
| 1,2-Dichloropropane | 0.5 | 0.1 | | | NE | NA | NA |
| cis-1,3-Dichloropropene[7] | 0.5 | 0.08 | | 244 | 244 | 0.0020 | 0.0003 |
| trans-1,3-Dichloropropene[7] | 0.5 | 0.07 | | 244 | 244 | 0.0020 | 0.0003 |
| Dichlorodifluoromethane | 0.5 | 0.12 | | | NE | NA | NA |
| Ethylbenzene | 0.5 | 0.12 | | >440 | >440 | 0.0011 | 0.0003 |
| Hexachlorobutadiene | 2 | 0.28 | | | NE | NA | NA |
| 2-Hexanone | 20 | 2.1 | | 32783 | 32783 | 0.0006 | 0.0001 |
| Iodomethane | TBD | TBD | | | NE | NA | NA |

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting and Detection Limits for Groundwater and Transition-Zone Water Samples.

| Analyte | MRL | MDL | NRWQC[1] | ORNL[2] | ACG[3] | Ratio of MRL to AGC | Ratio of MDL to ACG |
|---|---|---|---|---|---|---|---|
| Isopropylbenzene | 2 | 0.12 | | | NE | NA | NA |
| 4-Methyl-2-Pentanone | 20 | 2.6 | | 77400 | 77400 | 0.0003 | 0.0000 |
| Methylene chloride | 2 | 0.1 | | 42667 | 42667 | 0.0000 | 0.0000 |
| Methyl tert-butyl ether | 0.5 | 0.09 | | | NE | NA | NA |
| Naphthalene | 2 | 0.14 | | 620 | 620 | 0.0032 | 0.0002 |
| Styrene | 0.5 | 0.1 | | | NE | NA | NA |
| 1,1,1,2-Tetrachloroethane | 0.5 | 0.1 | | | NE | NA | NA |
| 1,1,2,2-Tetrachloroethane | 0.5 | 0.13 | | 2400 | 2400 | 0.0002 | 0.0001 |
| Tetrachloroethene | 0.5 | 0.11 | | 750 | 750 | 0.0007 | 0.0001 |
| Toluene | 0.5 | 0.1 | | 1269 | 1269 | 0.0004 | 0.0001 |
| 1,2,4-Trichlorobenzene | 2 | 0.09 | | | NE | NA | NA |
| 1,1,1-Trichloroethane | 0.5 | 0.14 | | 3493 | 3493 | 0.0001 | 0.0000 |
| 1,1,2-Trichloroethane | 0.5 | 0.08 | | 9400 | 9400 | 0.0001 | 0.0000 |
| Trichloroethene | 0.5 | 0.11 | | 7257 | 7257 | 0.0001 | 0.0000 |
| Trichlorofluoromethane | 0.5 | 0.13 | | | NE | NA | NA |
| 1,2,3-Trichloropropane | 0.5 | 0.28 | | | NE | NA | NA |
| Vinyl Acetate | TBD | TBD | | 810 | 810 | NA | NA |
| Vinyl Chloride | 0.5 | 0.11 | | | NE | NA | NA |
| m&p-Xylene | 0.5 | 0.24 | | 62308 | 62308 | 0.0000 | 0.0000 |
| o-Xylene | 0.5 | 0.1 | | 62308 | 62308 | 0.0000 | 0.0000 |
| **Polycyclic Aromatic Hydrocarbons, µg/L (ppb)** | | | | | | | |
| Acenaphthene | 0.02 | 0.002 | | 74 | 74 | 0.0003 | 0.0000 |
| Acenaphthylene | 0.02 | 0.002 | | | NE | NA | NA |
| Anthracene | 0.02 | 0.002 | | 0.09 | 0.09 | 0.2222 | 0.0222 |
| Benzo(a)anthracene | 0.02 | 0.003 | | 0.65 | 0.65 | 0.0308 | 0.0046 |
| Benzo(b)fluoranthene | 0.02 | 0.002 | | | NE | NA | NA |
| Benzo(k)fluoranthene | 0.02 | 0.002 | | | NE | NA | NA |
| Benzo(g,h,i)perylene | 0.02 | 0.004 | | | NE | NA | NA |
| Benzo(a)pyrene | 0.02 | 0.002 | | 0.3 | 0.3 | 0.0667 | 0.0067 |
| Chrysene | 0.02 | 0.002 | | | NE | NA | NA |
| Dibenz(a,h)anthracene | 0.02 | 0.002 | | | NE | NA | NA |

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting and Detection Limits for Groundwater and Transition-Zone Water Samples.

| Analyte | MRL | MDL | NRWQC[1] | ORNL[2] | ACG[3] | Ratio of MRL to AGC | Ratio of MDL to ACG |
|---|---|---|---|---|---|---|---|
| Fluoranthene | 0.02 | 0.003 | | 15 | 15 | 0.0013 | 0.0002 |
| Fluorene | 0.02 | 0.003 | | | NE | NA | NA |
| Indeno(1,2,3-cd)pyrene | 0.02 | 0.003 | | | NE | NA | NA |
| 2-Methylnaphthalene | 0.02 | 0.003 | | | NE | NA | NA |
| Naphthalene | 0.02 | 0.004 | | 620 | 620 | 0.0000 | 0.0000 |
| Phenanthrene | 0.02 | 0.004 | | 200 | 200 | 0.0001 | 0.0000 |
| Pyrene | 0.02 | 0.003 | | | NE | NA | NA |

**Notes:**

[1] NRWQC freshwater aquatic life criteria (EPA 2002b).

[2] ORNL values are lowest chronic value for all organisms in Table 1 of Suter and Tsao (1996).

[3] ACGs are the lower of the NRWQC freshwater aquatic life criteria (EPA 2002b) and ORNL values (Suter and Tsao 1996).

[4] Analysis is for total chromium. ACGs are based on hexavalent chromium.

[5] Parameters for calculating freshwater dissolved metals that are hardness-dependent are from NRWQC (EPA 2002b). Hardness-dependent criteria based on average hardness of 25 mg/L ($CaCO_3$) (USGS database from 1974 to 1990).

[6] ACG is based on ORNL value (Suter and Tsao 1996) for p-p'-DDD.

[7] ACG is based on ORNL value (Suter and Tsao 1996) for nonspecific 1,3-Dichloropropene.

MRL = minimum reporting limit
MDL = minimum detection limit
NA = not applicable
NE = not established
NRWQC = National Recommended Water Quality Criteria
ORNL = Oak Ridge National Laboratory
TBD = to be determined

Table A6-3. Method Reporting Limits for Volatile Organic Compounds in Vapor Samples.

| Analyte | MRL ($ug/m^3$) | MRL (ppbv) |
|---|---|---|
| 1,1,1,2-Tetrachloroethane | NA | NA |
| 1,1,1-Trichloroethane | 220 | 39.6 |
| 1,1,2,2-Tetrachloroethane | 220 | 33 |
| 1,1,2-Trichloroethane | 220 | 39.6 |
| 1,1-Dichloroethane | 220 | 55 |
| 1,1-Dichloroethene | 220 | 55 |
| 1,2,3-Trichloropropane | NA | NA |
| 1,2,4-Trichlorobenzene | NA | NA |
| 1,2-Dibromoethane | 220 | 28.6 |
| 1,2-Dichlorobenzene | 220 | 37.4 |
| 1,2-Dichloroethane | 220 | 55 |
| 1,2-Dichloropropane | 220 | 48.4 |
| 1,3-Dichlorobenzene | 220 | 37.4 |
| 1,4-Dichlorobenzene | 220 | 37.4 |
| 2,2'-oxybis (1-chloropropane) | NA | NA |
| 2-Butanone (MEK) | 220 | 74.8 |
| 2-Chloroethyl Vinyl Ether | NA | NA |
| 2-Chloronaphthalene | NA | NA |
| 2-Hexanone | 220 | 52.8 |
| 3,3'-Dichlorbenzidine | NA | NA |
| 4-bromophenyl-phenyl ether | NA | NA |
| 4-Chloroaniline | NA | NA |
| 4-Chlorophenyl-phenyl ether | NA | NA |
| 4-Methyl-2-pentanone | 220 | 52.8 |
| Acetone | 1100 | 462 |
| Acrolein | NA | NA |
| Acrylonitrile | NA | NA |
| Benzene | 220 | 68.2 |
| Bis-(2-chloroethoxy) methane | NA | NA |
| Bis-(2-chloroethyl) ether | NA | NA |
| Bromochloromethane | NA | NA |
| Bromodichloromethane | 220 | 33 |
| Bromoform | 220 | 21.34 |
| Bromomethane | 220 | 57.2 |
| Carbon Disulfide | 220 | 70.4 |
| Carbon Tetrachloride | 220 | 35.2 |
| Chlorobenzene | 220 | 48.4 |
| Chlorodibromomethane | 220 | 26.4 |
| Chloroethane | 220 | 83.6 |
| Chloroform | 220 | 44 |
| Chloromethane | 220 | 105.6 |
| cis-1,3-Dichloropropene | 220 | 48.4 |
| Dibromomethane | NA | NA |
| Dichlorodifluoromethane | NA | NA |
| Ethylbenzene | 220 | 50.6 |

Table A6-3. Method Reporting Limits for Volatile Organic Compounds in Vapor Samples.

| Analyte | MRL ($ug/m^3$) | MRL (ppbv) |
|---|---|---|
| Hexachlorobenzene | NA | NA |
| Hexachlorobutadiene | NA | NA |
| Hexachlorocyclopentadiene | NA | NA |
| Hexachloroethane | NA | NA |
| Iodomethane | NA | NA |
| Isopropylbenzene | NA | NA |
| m&p-Xylene | 220 | 50.6 |
| Methyl tert-butyl ether | 220 | 61.6 |
| Methylene chloride | 220 | 63.8 |
| Naphthalene | NA | NA |
| o-Xylene | 220 | 50.6 |
| Styrene | 220 | 50.6 |
| Tetrachloroethene | 220 | 33 |
| Toluene | 220 | 59.4 |
| trans-1,2-Dichloroethene | 220 | 55 |
| trans-1,3-Dichloropropene | 220 | 48.4 |
| trans-1,4-Dichloro-2-butene | NA | NA |
| Trichloroethene | 220 | 41.8 |
| Trichlorofluoromethane | 220 | 39.6 |
| Vinyl Acetate | 220 | 61.6 |
| Vinyl Chloride | 220 | 85.8 |

**Notes:**
MRL = method reporting limit
NA = No analysis. This VOC will not be analyzed.
ug/m3 = micrograms per cubic meter
ppbv = parts per billion by volume

Table A6-4. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples.

| Analyte | CAS number | ACG[1] | MDL[2] | MRL[3] |
|---|---|---|---|---|
| **Conventional Analyses** | | | | |
| Total solids (percent of whole weight) | -- | * | 0.01 | 0.01 |
| Perchlorate (µg/kg) | 14797-73-0 | * | NA | 20 |
| **Metals** | | | mg/kg dry wt | |
| Aluminum | 7429-90-5 | * | 2.0 | 2.0 |
| Antimony | 7440-36-0 | * | 0.02 | 0.05 |
| Arsenic | 7440-38-2 | * | 0.05 | 0.1 |
| Cadmium | 7440-43-9 | * | 0.006 | 0.02 |
| Chromium | 7440-47-3 | * | 0.04 | 0.2 |
| Copper | 7440-50-8 | * | 0.07 | 0.1 |
| Lead | 7439-92-1 | * | 0.02 | 0.05 |
| Mercury | 7439-97-6 | * | 0.01 | 0.02 |
| Nickel | 7440-02-0 | * | 0.03 | 0.2 |
| Selenium | 7782-49-2 | * | 0.05 | 0.1 |
| Silver | 7440-22-4 | * | 0.02 | 0.02 |
| Zinc | 7440-66-6 | * | 0.1 | 0.5 |
| Calcium | 7440-70-2 | * | 3 | 10 |
| Magnesium | 7439-95-4 | * | 2 | 4 |
| Manganese | 7439-96-5 | * | 2 | 5 |
| Potassium | 7440-09-7 | * | 100 | 400 |
| Sodium | 7440-23-5 | * | 20 | 20 |
| **Organochlorine Pesticides** | | | µg/kg dry wt | |
| 4,4'-DDD | 72-54-8 | 0.083 | 0.093 | 1 |
| 4,4'-DDE | 72-55-9 | 0.0588 | 0.076 | 1 |
| 4,4'-DDT | 50-29-3 | 0.0588 | 0.17 | 1 |
| **Total Petroleum Hydrocarbons** | | | mg/kg dry wt | |
| TPH-gas | NA | * | 3.2 | 10 |
| TPH-diesel | NA | * | 7.1 | 25 |
| **Volatile Organic Compounds** | | | µg/kg dry wt | |
| 1,1,1,2-Tetrachlorethane | 630-20-6 | * | 0.031 | 1 |
| 1,1,1-Trichloroethane (TCA) | 71-55-6 | * | 0.059 | 1 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | * | 0.096 | 1 |
| 1,1,2-Trichloroethane | 79-00-5 | * | 0.059 | 1 |
| 1,1-Dichloroethane | 75-34-3 | * | 0.057 | 1 |
| 1,2,3-Trichloropropane | 96-18-4 | * | 0.11 | 1 |
| 1,2-Dichloroethane (EDC) | 107-06-2 | * | 0.031 | 1 |
| 1,2-Dichloropropane | 78-87-5 | * | 0.035 | 1 |
| 1,4-Dichlorobenzene | 106-46-7 | * | 0.12 | 1 |
| 1,2-Dibromoethane (EDB) | 106-93-4 | * | 0.051 | 1 |
| 1,2-Dichlorobenzene | 95-50-1 | * | 0.075 | 1 |
| 1,3-Dichlorobenzene | 541-73-1 | * | 0.11 | 1 |
| 1,2,4-Trichlorobenzene | 120-82-1 | * | 0.36 | 1 |
| 2-Butanone (MEK) | 78-93-3 | * | 1.1 | 4 |
| 2-Chloroethyl Vinyl Ether | 110-75-8 | * | 0.15 | 2 |
| 2-Hexanone | 591-78-6 | * | 0.64 | 2 |
| 4-Methyl-2-Pentanone (MIBK) | 108-10-1 | * | 0.24 | 2 |

Table A6-4. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples.

| Analyte | CAS number | ACG[1] | MDL[2] | MRL[3] |
|---|---|---|---|---|
| Acetone | 67-64-1 | * | 1.6 | 10 |
| Acrolein | 107-02-8 | * | 0.51 | 2.5 |
| Acrylonitrile | 107-13-1 | * | 0.23 | 2 |
| Bromochloromethane | 74-97-5 | * | 0.06 | 1 |
| Bromodichloromethane | 75-27-4 | * | 0.068 | 1 |
| Bromoform | 75-25-2 | * | 0.046 | 1 |
| Bromomethane | 74-83-9 | * | 0.37 | 1 |
| Carbon Disulfide | 75-15-0 | * | 0.13 | 1 |
| Carbon Tetrachloride | 56-23-5 | * | 0.099 | 1 |
| Chlorobenzene | 108-90-7 | * | 0.07 | 1 |
| Chlorodibromomethane | 124-48-1 | * | 0.068 | 1 |
| Chloroethane | 75-00-3 | * | 0.28 | 1 |
| Chloroform | 67-66-3 | * | 0.056 | 1 |
| Chloromethane | 74-87-3 | * | 0.19 | 1 |
| cis-1,3-Dichloropropene | 10061-01-5 | * | 0.031 | 1 |
| Dibromomethane | 74-95-3 | * | 0.082 | 1 |
| Dichlorodifluoromethane | 75-71-8 | * | 0.11 | 1 |
| Hexachlorobutadiene | 87-68-3 | * | 0.17 | 2 |
| Iodomethane (Methyl Iodide) | 74-88-4 | * | 0.67 | 2 |
| Isopropylbenzene | 98-82-8 | * | 0.044 | 1 |
| Methylene Chloride | 75-09-2 | * | 0.29 | 5 |
| Naphthalene | 91-20-3 | * | 0.35 | 1 |
| Styrene | 100-42-5 | * | 0.074 | 1 |
| trans-1,4-Dichloro-2-butene | 110-57-6 | * | 0.51 | 2 |
| Trichlorofluoromethane | 75-69-4 | * | 0.081 | 1 |
| Vinyl Acetate | 108-05-4 | * | 0.36 | 1 |
| 1,1-Dichloroethene | 75-35-4 | * | 0.082 | 1 |
| Benzene | 71-43-2 | * | 0.039 | 1 |
| Ethyl Benzene | 100-41-4 | * | 0.071 | 1 |
| *m,p* -Xylene | 136777-61-2 | * | 0.13 | 1 |
| Methyl tert-butyl ether | 1634-04-4 | * | 0.039 | 1 |
| *o* -Xylene | 95-47-6 | * | 0.075 | 1 |
| Tetrachloroethene (PCE) | 127-18-4 | * | 0.096 | 1 |
| Toluene | 108-88-3 | * | 0.15 | 1 |
| trans-1,2-Dichloroethene | 156-60-5 | * | 0.19 | 1 |
| trans-1,3-Dichloropropene | 10061-02-6 | * | 0.044 | 1 |
| Trichloroethene (TCE) | 79-01-6 | * | 0.062 | 1 |
| Vinyl Chloride | 75-01-4 | * | 0.085 | 1 |
| Chlorobenzene | 108-90-7 | * | 0.070 | 1 |
| **Semivolatile Organic Compounds** | | | µg/kg dry wt | |
| **Polycyclic Aromatic Hydrocarbons** | | | | |
| Naphthalene | 91-20-3 | 24 | 0.34 | 5 |
| 2-Methylnaphthalene | 91-57-6 | * | 0.34 | 5 |
| Acenaphthylene | 208-96-8 | * | 0.22 | 5 |
| Acenaphthene | 83-32-9 | 72 | 0.16 | 5 |
| Fluorene | 86-73-7 | 48 | 0.19 | 5 |
| Phenanthrene | 85-01-8 | * | 0.33 | 5 |
| Anthracene | 120-12-7 | 360 | 0.22 | 5 |
| Fluoranthene | 206-44-0 | 48 | 0.34 | 5 |
| Pyrene | 129-00-0 | 36 | 0.36 | 5 |

Table A6-4. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples.

| Analyte | CAS number | ACG[1] | MDL[2] | MRL[3] |
|---|---|---|---|---|
| Benz(a)anthracene | 56-55-3 | 0.038 | 0.16 | 5 |
| Chrysene | 218-01-9 | 3.8 | 0.41 | 5 |
| Benzo(b)fluoranthene | 205-99-2 | 0.038 | 0.48 | 5 |
| Benzo(k)fluoranthene | 207-08-9 | 0.38 | 0.33 | 5 |
| Benzo(a)pyrene | 50-32-8 | 0.0038 | 0.22 | 5 |
| Indeno(1,2,3-cd)pyrene | 193-39-5 | 0.038 | 0.24 | 5 |
| Dibenz(a,h)anthracene | 53-70-3 | 0.0038 | 0.26 | 5 |
| Benzo(g,h,i)perylene | 191-24-2 | * | 0.23 | 5 |

**Notes:**

[1] ACG values are as provided in the Round 2 QAPP.

[2] The laboratory's current MDL is provided. MDLs are updated periodically by the laboratory.

[3] The MRL is provided on a dry-weight basis and assumes 50% moisture in the samples.
The MRL for project samples will vary with moisture content in the samples.
The MRL represents the level of lowest calibration standard (i.e., the practical quantitation limit).

ACG = analytical concentration goals; established by EPA during *ad hoc* meeting with LWG May 10, 2002
MDL = method detection limit
MRL = method reporting limit
NA = not applicable
* = A risk-based ACG has not been established.

Table A7-1. Laboratory Control Limits for Surrogate Compounds.

| Surrogate compound | Surrogate recovery (percent) | | |
|---|---|---|---|
| | Water samples | Vapor samples | Sediment samples |
| Organochlorine Pesticides | | | |
| Tetrachloro-*meta*-xylene | 17-128 | -- | 39-129 |
| Decachlorobiphenyl | 10-136 | -- | 29-141 |
| Semivolatile Organic Compounds (SIM) | | | |
| Fluoranthene-d10 | 18-137 | -- | 34-122 |
| Fluorene-d10 | 37-107 | -- | 27-110 |
| Terphenyl-d14 | 18-153 | -- | 50-132 |
| Volatile Organic Compounds | | | |
| Toluene-d8 | 77-131 | 70-140 | 77-131 |
| 4-Bromofluorobenzene | 74-116 | 70-140 | 74-116 |
| Dibromofluoromethane | 81-120 | NA | 81-120 |
| 1,2-Dichloroethane-d4 | 80-118 | 70-140 | 80-118 |
| Peteroleum Hydrocarbons | | | |
| 4-Bromofluorobenzene | 50-150 | -- | 50-150 |
| n-Triacontane | 50-150 | -- | 50-150 |
| o-Terphenyl | 50-150 | -- | 50-150 |

**Notes:**

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

NA = not applicable
SIM = selected ion monitoring

Table A7-2. Laboratory Control Limits for Accuracy and Precision.

| Analyte | Matrix Spike Recovery (percent) | Laboratory Control Sample Recovery (percent) | Type of Duplicate | Control Limit (RPD) |
|---|---|---|---|---|
| **Water Samples** | | | | |
| Conventional Analyses | | | | |
| pH | NA | NA | LD | 20 |
| Chloride | 80-120 | 90-110 | LD | 20 |
| Sulfate | 80-120 | 90-110 | LD | 20 |
| Alkalinity as $CaCO_3$ | NA | NA | LD | 20 |
| Perchlorate | 80-120 | 85-115 | LD | 20 |
| Metals | | | | |
| Arsenic | 70-130 | 85-115[2] | LD | 20 |
| Copper | 70-130 | 85-115[2] | LD | 20 |
| Chromium,total | 70-130 | 85-115[2] | LD | 20 |
| Lead | 70-130 | 85-115[2] | LD | 20 |
| Zinc | 70-130 | 85-115[2] | LD | 20 |
| Calcium | NA | 85-115[2] | LD | 20 |
| Magnesium | NA | 85-115[2] | LD | 20 |
| Manganese | NA | 85-115[2] | LD | 20 |
| Potassium | NA | 85-115[2] | LD | 20 |
| Sodium | NA | 85-115[2] | LD | 20 |
| Organochlorine Pesticides | | | | |
| 4,4'-DDD | 20-178 | 47-151 | MSD | 30 |
| 4,4'-DDE | 35-142 | 66-122 | MSD | 30 |
| 4,4'-DDT | 17-183 | 47-157 | MSD | 30 |
| Semivolatile Organic Compounds (PAHs) | | | | |
| Naphthalene | 10-125 | 27-116 | MSD | 30 |
| 2-Methylnaphthalene | 10-121 | 22-106 | MSD | 30 |
| Acenaphthylene | 18-135 | 33-131 | MSD | 30 |
| Acenaphthene | 10-139 | 31-122 | MSD | 30 |
| Fluorene | 10-147 | 33-120 | MSD | 30 |
| Phenanthrene | 15-146 | 35-127 | MSD | 30 |
| Anthracene | 15-137 | 34-126 | MSD | 30 |
| Fluoranthene | 35-125 | 36-132 | MSD | 30 |
| Pyrene | 15-146 | 38-129 | MSD | 30 |
| Benz(a)anthracene | 23-134 | 39-128 | MSD | 30 |
| Chrysene | 34-122 | 40-128 | MSD | 30 |
| Benzo(b)fluoranthene | 31-125 | 36-135 | MSD | 30 |
| Benzo(k)fluoranthene | 36-121 | 38-133 | MSD | 30 |
| Benzo(a)pyrene | 29-118 | 35-129 | MSD | 30 |
| Indeno(1,2,3-cd)pyrene | 31-126 | 37-133 | MSD | 30 |
| Dibenz(a,h)anthracene | 30-133 | 38-135 | MSD | 30 |
| Benzo(g,h,i)perylene | 20-141 | 39-133 | MSD | 30 |
| Total Petroleum Hydrocarbons | | | | |
| TPH-gas | 65-133 | 65-133 | | 30 |
| TPH-diesel | 71-146 | 71-146 | | 30 |

Table A7-2. Laboratory Control Limits for Accuracy and Precision.

| Analyte | Matrix Spike Recovery (percent) | Laboratory Control Sample Recovery (percent) | Type of Duplicate | Control Limit (RPD) |
|---|---|---|---|---|
| Volatile Organic Compounds | | | | |
| 1,1,1,2-Tetrachloroethane | 17-138 | 76-122 | MSD | 40 |
| 1,1,1-Trichloroethane (TCA) | 41-127 | 68-126 | MSD | 40 |
| 1,1,2,2-Tetrachloroethane | 15-132 | 69-120 | MSD | 40 |
| 1,1,2-Trichloroethane | 31-135 | 77-118 | MSD | 40 |
| 1,1-Dichloroethane | 45-122 | 66-120 | MSD | 40 |
| 1,2,3-Trichloropropane | 20-144 | 73-120 | MSD | 40 |
| 1,2-Dichloroethane (EDC) | 34-134 | 63-129 | MSD | 40 |
| 1,2-Dichloropropane | 36-129 | 69-123 | MSD | 40 |
| 1,4-Dichlorobenzene | 10-129 | 71-127 | MSD | 40 |
| 1,2-Dibromoethane (EDB) | 22-132 | 76-120 | MSD | 40 |
| 1,2-Dichlorobenzene | 10-127 | 73-123 | MSD | 40 |
| 1,3-Dichlorobenzene | 10-128 | 69-129 | MSD | 40 |
| 1,2,4-Trichlorobenzene | 10-128 | 63-139 | MSD | 40 |
| 2-Butanone (MEK) | 10-143 | 37-137 | MSD | 40 |
| 2-Chloroethyl Vinyl Ether | 70-130 | 34-149 | MSD | 40 |
| 2-Hexanone | 22-126 | 60-124 | MSD | 40 |
| 4-Methyl-2-Pentanone (MIBK) | 29-137 | 55-136 | MSD | 40 |
| Acetone | 20-127 | 43-119 | MSD | 40 |
| Acrolein | 70-130 | 18-148 | MSD | 40 |
| Acrylonitrile | 70-130 | 10-197 | MSD | 40 |
| Bromochloromethane | 41-134 | 75-126 | MSD | 40 |
| Bromodichloromethane | 19-138 | 67-129 | MSD | 40 |
| Bromoform | 10-136 | 72-121 | MSD | 40 |
| Bromomethane | 15-140 | 37-145 | MSD | 40 |
| Carbon Disulfide | 19-139 | 63-139 | MSD | 40 |
| Carbon Tetrachloride | 25-131 | 69-128 | MSD | 40 |
| Chlorobenzene | 17-130 | 76-119 | MSD | 40 |
| Chlorodibromomethane | 19-138 | 67-129 | MSD | 40 |
| Chloroethane | 28-149 | 44-143 | MSD | 40 |
| Chloroform | 37-132 | 70-123 | MSD | 40 |
| Chloromethane | 36-148 | 51-147 | MSD | 40 |
| cis-1,3-Dichloropropene | 11-135 | 73-127 | MSD | 40 |
| Dibromomethane | 34-137 | 74-124 | MSD | 40 |
| Dichlorodifluoromethane | 34-154 | 43-163 | MSD | 40 |
| Hexachlorobutadiene | 10-117 | 58-132 | MSD | 40 |
| Iodomethane (Methyl Iodide) | 70-130 | 70-130 | MSD | 40 |
| Isopropylbenzene | 10-129 | 62-125 | MSD | 40 |
| Methylenechloride | 41-131 | 70-127 | MSD | 40 |
| Naphthalene | 10-131 | 69-134 | MSD | 40 |
| Styrene | 10-138 | 75-127 | MSD | 40 |
| trans-1,4-Dichloro-2-butene | 70-130 | 70-130 | MSD | 40 |
| Trichlorofluoromethane | 37-135 | 55-134 | MSD | 40 |
| Vinyl Acetate | 70-130 | 10-144 | MSD | 40 |
| 1,1-Dichloroethene | 46-128 | 71-127 | MSD | 40 |
| Benzene | 45-129 | 78-124 | MSD | 40 |
| EthylBenzene | 15-138 | 75-126 | MSD | 40 |
| m,p-Xylene | 15-138 | 76-131 | MSD | 40 |
| Methyltert-butyl ether | 37-132 | 59-124 | MSD | 40 |
| o-Xylene | 12-143 | 76-128 | MSD | 40 |

Table A7-2. Laboratory Control Limits for Accuracy and Precision.

| Analyte | Matrix Spike Recovery (percent) | Laboratory Control Sample Recovery (percent) | Type of Duplicate | Control Limit (RPD) |
|---|---|---|---|---|
| Tetrachloroethene (PCE) | 10-139 | 70-124 | MSD | 40 |
| Toluene | 31-136 | 75-128 | MSD | 40 |
| trans-1,2-Dichloroethene | 33-128 | 68-122 | MSD | 40 |
| trans-1,3-Dichloropropene | 10-131 | 68-114 | MSD | 40 |
| Trichloroethene (TCE) | 18-153 | 69-128 | MSD | 40 |
| Vinyl Chloride | 34-165 | 55-155 | MSD | 40 |
| **Vapor samples** | | | | |
| Volatile Organic Compounds | | | | |
| 1,1-Dichloroethene | NA | 70-130 | LCSD | 30 |
| Benzene | NA | 70-130 | LCSD | 30 |
| Trichloroethene (TCE) | NA | 70-130 | LCSD | 30 |
| Toluene | NA | 70-130 | LCSD | 30 |
| Chlorobenzene | NA | 70-130 | LCSD | 30 |
| 1,2-Dichlorobenzene | NA | 70-130 | LCSD | 30 |
| Naphthalene | NA | 70-130 | LCSD | 30 |
| **Sediment samples** | | | | |
| Conventional Analyses | | | | |
| Total solids | NA | NA | LD | 20 |
| Perchlorate | 75-125 | 75-125 | LD | 20 |
| Metals | 70-130 | Note-2 | LD | 30 |
| Organochlorine Pesticides | | | | |
| 4,4'-DDD | 32-166 | 67-139 | MSD | 40 |
| 4,4'-DDE | 48-146 | 67-133 | MSD | 40 |
| 4,4'-DDT | 47-157 | 68-144 | MSD | 40 |
| Total Petroleum Hydrocarbons | | | | |
| TPH-gas | 42-125 | 78-128 | LD | 40 |
| TPH-diesel | 66-146 | 74-150 | LD | 40 |
| Volatile Organic Compounds | | | | |
| 1,1,1,2-Tetrachloroethane | 17-138 | 76-122 | MSD | 40 |
| 1,1,1-Trichloroethane(TCA) | 41-127 | 68-126 | MSD | 40 |
| 1,1,2,2-Tetrachloroethane | 15-132 | 69-120 | MSD | 40 |
| 1,1,2-Trichloroethane | 31-135 | 77-118 | MSD | 40 |
| 1,1-Dichloroethane | 45-122 | 66-120 | MSD | 40 |
| 1,2,3-Trichloropropane | 20-144 | 73-120 | MSD | 40 |
| 1,2-Dichloroethane(EDC) | 34-134 | 63-129 | MSD | 40 |
| 1,2-Dichloropropane | 36-129 | 69-123 | MSD | 40 |
| 1,4-Dichlorobenzene | 10-129 | 71-127 | MSD | 40 |
| 1,2-Dibromoethane(EDB) | 22-132 | 76-120 | MSD | 40 |
| 1,2-Dichlorobenzene | 10-127 | 73-123 | MSD | 40 |
| 1,3-Dichlorobenzene | 10-128 | 69-129 | MSD | 40 |
| 1,2,4-Trichlorobenzene | 10-128 | 63-139 | MSD | 40 |
| 2-Butanone (MEK) | 10-143 | 37-137 | MSD | 40 |
| 2-Chloroethyl Vinyl Ether | 70-130 | 34-149 | MSD | 40 |
| 2-Hexanone | 22-126 | 60-124 | MSD | 40 |
| 4-Methyl-2-Pentanone (MIBK) | 29-137 | 55-136 | MSD | 40 |

Table A7-2. Laboratory Control Limits for Accuracy and Precision.

| Analyte | Matrix Spike Recovery (percent) | Laboratory Control Sample Recovery (percent) | Type of Duplicate | Control Limit (RPD) |
|---|---|---|---|---|
| Acetone | 20-127 | 43-119 | MSD | 40 |
| Acrolein | 70-130 | 18-148 | MSD | 40 |
| Acrylonitrile | 70-130 | 10-197 | MSD | 40 |
| Bromochloromethane | 41-134 | 75-126 | MSD | 40 |
| Bromodichloromethane | 19-138 | 67-129 | MSD | 40 |
| Bromoform | 10-136 | 72-121 | MSD | 40 |
| Bromomethane | 15-140 | 37-145 | MSD | 40 |
| Carbon Disulfide | 19-139 | 63-139 | MSD | 40 |
| Carbon Tetrachloride | 25-131 | 69-128 | MSD | 40 |
| Chlorobenzene | 17-130 | 76-119 | MSD | 40 |
| Chlorodibromomethane | 10-137 | 74-119 | MSD | 40 |
| Chloroethane | 28-149 | 44-143 | MSD | 40 |
| Chloroform | 37-132 | 70-123 | MSD | 40 |
| Chloromethane | 36-148 | 51-147 | MSD | 40 |
| cis-1,3-Dichloropropene | 11-135 | 73-127 | MSD | 40 |
| Dibromomethane | 34-137 | 74-124 | MSD | 40 |
| Dichlorodifluoromethane | 34-154 | 43-163 | MSD | 40 |
| Hexachlorobutadiene | 10-117 | 58-132 | MSD | 40 |
| Iodomethane (Methyl Iodide) | 70-130 | 70-130 | MSD | 40 |
| Isopropylbenzene | 10-129 | 62-125 | MSD | 40 |
| Methylenechloride | 41-131 | 70-127 | MSD | 40 |
| Naphthalene | 10-131 | 69-134 | MSD | 40 |
| Styrene | 10-138 | 75-127 | MSD | 40 |
| trans-1,4-Dichloro-2-butene | 70-130 | 70-130 | MSD | 40 |
| Trichlorofluoromethane | 37-135 | 55-134 | MSD | 40 |
| Vinyl Acetate | 70-130 | 10-144 | MSD | 40 |
| 1,1-Dichloroethene | 46-128 | 71-127 | MSD | 40 |
| Benzene | 45-129 | 78-124 | MSD | 40 |
| Ethyl Benzene | 15-138 | 75-126 | MSD | 40 |
| m,p-Xylene | 15-138 | 76-131 | MSD | 40 |
| Methyl tert-butyl ether | 37-132 | 59-124 | MSD | 40 |
| o-Xylene | 12-143 | 76-128 | MSD | 40 |
| Tetrachloroethene(PCE) | 10-139 | 70-124 | MSD | 40 |
| Toluene | 31-136 | 75-128 | MSD | 40 |
| trans-1,2-Dichloroethene | 33-128 | 68-122 | MSD | 40 |
| trans-1,3-Dichloropropene | 10-131 | 68-114 | MSD | 40 |
| Trichloroethene (TCE) | 18-153 | 69-128 | MSD | 40 |
| Vinyl Chloride | 34-165 | 55-155 | MSD | 40 |
| Semivolatile Organic Compounds | | | | |
| Naphthalene | 22-101 | 43-102 | MSD | 40 |
| 2-Methylnaphthalene | 27-106 | 44-105 | MSD | 40 |
| Acenaphthylene | 36-113 | 51-107 | MSD | 40 |
| Acenaphthene | 32-114 | 50-105 | MSD | 40 |
| Fluorene | 39-118 | 54-108 | MSD | 40 |
| Phenanthrene | 29-130 | 58-106 | MSD | 40 |
| Anthracene | 38-133 | 61-113 | MSD | 40 |
| Fluoranthene | 30-143 | 63-117 | MSD | 40 |
| Pyrene | 28-143 | 59-121 | MSD | 40 |
| Benz(a)anthracene | 24-149 | 57-120 | MSD | 40 |
| Chrysene | 38-133 | 64-116 | MSD | 40 |

Table A7-2. Laboratory Control Limits for Accuracy and Precision.

| Analyte | Matrix Spike Recovery (percent) | Laboratory Control Sample Recovery (percent) | Type of Duplicate | Control Limit (RPD) |
|---|---|---|---|---|
| Benzo(b)fluoranthene | 26-144 | 58-126 | MSD | 40 |
| Benzo(k)fluoranthene | 29-136 | 61-122 | MSD | 40 |
| Benzo(a)pyrene | 30-146 | 58-128 | MSD | 40 |
| Indeno(1,2,3-cd)pyrene | 24-147 | 46-133 | MSD | 40 |
| Dibenz(a,h)anthracene | 33-136 | 50-128 | MSD | 40 |
| Benzo(g,h,i)perylene | 23-142 | 52-125 | MSD | 40 |

**Notes:**

Note 1 - Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

Note 2 - Percent recovery control limits are not applicable. Laboratory control limits are established based on the manufacturer's established range of acceptable concentrations.

[1] In-house limits
[2] Method specified control limits

Table B2-1. Standard Sample Containers and Preservation Requirements.

| | Container | | Additional | Preservation | Holding Time | Laboratory |
|---|---|---|---|---|---|---|
| | Type | Size | for Lab QC[1] | | | Sample Size |
| **Water samples** | | | | | | |
| Conventionals | HDPE | 250 ml | none | 4±2°C | 14 days[2] | 100 ml |
| Conventionals + Perchlorate | HDPE | 250 ml | none | 4±2°C | 14 days[2] | 100 ml |
| Metals | HDPE | 500 mL | 1 liter | 5 ml of 1:1& $HNO_3$; 4±2°C | 6 months | 300 ml |
| Chlorinated pesticides | AG | 1 liter | 2 liters | 4±2°C | 7/40 days[3] | 1 liter |
| Semivolatile organic compounds (PAH) | AG | 1 liter | 2 liters | 4±2°C | 7/40 days[3] | 1 liter |
| Volatile organic compounds | VOA | 3x40 mL | 2x40 mL | HCl; 4±2°C; no headspace | 14 days | 5 ml |
| TPH-gas | VOA | 3x40 mL | 2x40 mL | HCl; 4±2°C; no headspace | 14 days | 5 ml |
| TPH-diesel | AG | 1 liter | 2 liters | HCl; 4±2°C | 14 days | 400 ml |
| **Vapor samples** | | | | | | |
| Volatile organic compounds | VOA-V | 40 mL | 2x40 mL | 4±2°C | 14 days | 5ml |
| **Sediment samples** | | | | | | |
| Metals, total solids | WMG | 2 oz | none | 4±2°C | 6 months | 10 g |
| Perchlorate | WMG | | | | 14 days | 2.5 g |
| Volatile organic compounds | WMG | 2 oz | none | No headspace; 4±2°C (do not freeze) | 14 days | 5 g |
| Semivolatile organic compounds (PAH) | WMG | 8 oz | none | Deep Frozen (-20°C) | 1 year | 50 g |
| Chlorinated pesticides | WMG | | none | | 1 year | 25 g |
| TPH-gas | WMG | 2 oz | none | No headspace; 4±2°C | 14 days | 5 g |
| TPH-diesel | WMG | 8 oz | none | No headspace; 4±2°C | 14 days | 20 g |

**Notes:**

[1]Sample container sizes may be modified to meet laboratory requirements.

[2]Extra sample volume will be collected at a frequency of 5% of samples to accommodate requirements for laboratory QC samples .

[3]The holding time is 7 days from collection to extraction and 40 days from extraction to analysis.

HDPE = high density polyethylene bottle
AG = amber glass bottle with Teflon™ -lined lid
VOA = 40-ml glass vial with Teflon™ septum cap
VOA-V = 40-ml glass vial with crimp top for vapor samples
WMG = wide-mouth glass jar with Teflon™ -lined lid

Table B3-1. Summary of Minimum Sample Volume Requirements and Recommended Sample Volumes for Transition Zone Water Samples.

| Study Area | Analysis | CAS Minimum Sample Volume Requirement With QC[1] (mL) | CAS Minimum Sample Volume Requirement Without QC[2] (mL) | Recommended Target Volume if Sample Volume is Limited (mL) | Notes |
|---|---|---|---|---|---|
| ***ARCO*** | | | | | |
| | Metals (As, Cu, Pb, Zn, Ca, Na, K, Mg, Mn) | 250 | 100 | 100 | ACGs for metals will be met with 50% sample volume reduction. |
| | VOCs (BTEX) | 120 | 80 | 80 | If sample volume is limited, only 2 VOA vials will be filled. |
| | PAHs | 1000 | 500 | 300 | ACGs can be met with 66% reduction in sample volume and smaller final extract volume. |
| | TPH-gas | 120 | 80 | 80 | If sample volume is limited, only 2 VOA vials will be filled. |
| | TPH-diesel | 1000 | 500 | 500 | May collected in same sample container as PAH sample. |
| | Conventionals (Cl, SO4, pH, alkalinity) | 250 | 150 | 75 | Conventionals are not COIs and thus are low priority analytes. Elevated MRLs will not have a substantial impact on the pilot study objectives. |
| | *Target Total Sample Volume* | 2740 | 1410 | 1135 | |
| ***Arkema Acid Plant*** | | | | | |
| | VOCs (full project list) | 120 | 80 | 80 | If sample volume is limited, only 1 or 2 VOA vials will be filled. |
| | DDT, DDE, DDD | 1000 | 500 | 500 | ACGs for DDT, DDD, and DDE cannot be met with full sample volumes. |
| | Metals (Ca, K, Mg, Mn, Na) | 250 | 100 | 50 | Major cations are not COIs and thus are low priority analytes. Elevated MRLs will not have a substantial impact on the pilot study objectives. |
| | Conventionals (Cl, SO4, pH, alkalinity) | 250 | 150 | 75 | Conventionals are not COIs and thus are low priority analytes. Elevated MRLs will not have a substantial impact on the pilot study objectives. |
| | *Target Total Sample Volume* | 1620 | 830 | 705 | |
| ***Arkema Chlorate Plant*** | | | | | |
| | Metals (Cr, Ca, K, Mg, Mn, Na) | 250 | 100 | 100 | ACGs for metals can be met with this level of dilution. |
| | Perchlorate and Conventionals (Cl, SO4, pH, alkalinity) | 250 | 150 | 150 | No ACG established for perchlorate; default is the standard MRL/MDL. Perchlorate is the priority for this sample; sample volume for conventionals may be reduced if needed. |
| | *Target Total Sample Volume* | 500 | 250 | 250 | |

***Notes:***

[1]CAS-provided minimum sample volumes to meet standard MRLs/MDLs and standard laboratory QC.

[2]CAS-provided minimum sample volumes to meet standard MRLs/MDLs, but with limited laboratory QC.

[3]Approximate amount of dilution of minimum sample volume allowable to meet ACGs.

Table B3-2. Sample Device Quantity Requirements to Meet Sample Volume Requirements.

| Study Area | | Sample Device Type | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Ideal Minimum Sample Volume Requirement (mL) | Small Volume Peeper | Large Volume Peeper | Vapor Diffusion | Trident Probe | UltraSeep | Push-Point | Power Grab & Centrifuge Extraction |
| **ARCO** | | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) |
| *Metals* | | | | | | | | |
| Unfiltered (As, Cu, Mn, Pb, Zn) | 250[a] | 100 | 250 | NS | 250 | 250 | 250 | 250 |
| Filtered (As, Cu, Pb, Zn, Ca, Na, K, Mg, Mn) | 250[a] | NS | NS | NS | 250 | 250 | 250 | 250 |
| *VOCs (BTEX)* | | | | | | | | |
| Unfiltered | 120[b] | 80 | 120 | 120 | 120 | 120 | 120 | 0 |
| *TPH-G* | | | | | | | | |
| Unfiltered | 120[b] | 80[f] | 120 | NS | NS | NS | 120 | NS |
| *PAHs, TPH-D* | | | | | | | | |
| Unfiltered | 1000[c] | 500[f] | 1000 | NS | 1000 | 1000 | 1000 | 1000 |
| Filtered | 1000[c] | NS | NS | NS | 1000 | 1000 | 1000 | 1000 |
| *Cl, SO4, pH, alkalinity* | | | | | | | | |
| Unfiltered | 250[d] | 40[g] | 250 | NS | NS | NS | NS | NS |
| Filtered | 250[d] | NS | NS | NS | 250 | 250 | 250 | 125 |
| | *Total Target Volume (mL)* | 800[f] | 1740 | 120 | 2870 | 2870 | 2990 | 2625 |
| | *Volume per Sampler (mL)* | 200[f] | 1000 | 40 | NA | NA | NA | 2500 |
| | *No. Samplers per Location* | 4[f] | 2 | 3 | NA | NA | NA | 2 |
| **Arkema -- Acid Plant Area** | | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) |
| *VOCs (full project list)* | | | | | | | | |
| Unfiltered | 120[b] | 80 | 120 | 120 | 120 | 120 | NS | NS |
| *DDT, DDE, DDD* | | | | | | | | |
| Unfiltered | 1000[c] | NS | 600 | NS | 1000 | 1000 | NS | 1000 |
| Filtered | 1000[c] | 500 | NS | NS | 1000 | 1000 | NS | 1000 |
| *Ca, K, Mg, Mn, Na* | | | | | | | | |
| Unfiltered | 250[e] | NS[h] | 125 | NS | 250 | 250 | NS | 125 |
| Filtered | 250[e] | NS[h] | NS | NS | 250 | 250 | NS | 125 |
| *Cl, SO4, pH, alkalinity* | | | | | | | | |
| Unfiltered | 250[d] | NS[h] | 125 | NS | NS | NS | NS | NS |
| Filtered | 250[d] | NS[h] | NS | NS | 250 | 250 | NS | 125 |
| | *Total Target Volume (mL)* | 580 | 970 | 120 | 2870 | 2870 | 0 | 2375 |
| | *Volume per Sampler (mL)* | 200 | 1000 | 40 | NA | NA | NA | 2500 |
| | *No. Samplers per Location* | 3 | 1 | 3 | NA | NA | 0 | 1 |

Table B3-2. Sample Device Quantity Requirements to Meet Sample Volume Requirements.

| Study Area | | Sample Device Type | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Ideal Minimum Sample Volume Requirement (mL) | Small Volume Peeper | Large Volume Peeper | Vapor Diffusion | Trident Probe | UltraSeep | Push-Point | Power Grab & Centrifuge Extraction |
| **Arkema -- Chlorate Plant Area** | | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) | Target Volume (mL) |
| *Metals* | | | | | | | | |
| Unfiltered (*Cr*) | 250[a] | 100 | 250 | NS | 250 | 250 | NS | 250 |
| Filtered (*Cr, Ca, K, Mg, Mn, Na*) | 250[a] | NS | NS | NS | 250 | 250 | NS | 250 |
| *Perchlorate, Cl, SO4, pH, alkalinity* | | | | | | | | |
| Unfiltered (*Perchlorate*) | 250[d] | 100 | 250 | NS | 250 | 250 | NS | 250 |
| Filtered (*Perchlorate, Cl, SO4, pH, alkalinity*) | 250[d] | NS | SN | NS | 250 | 250 | NS | 250 |
| | *Total Target Volume (mL)* | 200 | 500 | 0 | 1000 | 1000 | 0 | 1000 |
| | *Volume per Sampler (mL)* | 200 | 1000 | 40 | NA | NA | NA | 2500 |
| | *No. Samplers per Location* | 1 | 1 | 0 | NA | NA | 0 | 1 |

**Notes:**

NS = No sample collected for analysis of subject constituent.

NA = Not Applicable.

Notes

[a]Can reduce to 100 mL if QC level reduced.

[b]Can reduce to 2x40mL vials if QC level reduced.

[c]Target volume for peepers is 1/2 the minimum sample volume. CAS will reduce the final extract volume to maintain MRLs and MDLs.

[d]Minimum volume of 150 mL without QC. 250 mL required for QC and analysis of perchlorate.

[e]Minimum volume of 100 mL without QC.

[f]Due to limited number of available small volume peeper devices. One location will not include analysis of TPH-G and TPH-D, and only 300 mL will be collected for PAH analysis. The replicate sample will be collected from this location and will have the same constraints.

[g]It is anticipated that there will be limited sample volume available for analysis of Cl, SO4, pH, alkalinity. Analysis of these constituents will be included only if sufficient volume is available after other sample volume requirements are met.

[h]Insufficient sample volume will be available for analysis of these parameters.

Table B5-1. Summary of Transition Zone Water, Groundwater, Bulk Sediment, and Field Quality Control Samples for the Pilot Study.

| | In-Situ Diffusion Samplers | | | | | | | | | In-Situ Direct Samplers | | | | | | | | | Ex-Situ Extraction | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Small Volume Peeper | | | Large Volume Peeper | | | Vapor Diffusion | | | Trident Probe | | | UltraSeep | | | Push-Point[b] | | | Power Grab & Centrifuge Extraction | | | **Power Grab Bulk Sediment Samples** | | |
| | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC |
| **ARCO** | | | | | | | | | | | | | | | | | | | | | | | | |
| Metals | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered (*As, Cu, Mn, Pb, Zn*) | 3 | 1 | 1D | 3 | 1 | 1D | 0 | 0 | 0 | 3 | 1 | 1E | 1 | 0 | 1E | 6 | 1 | 1E | 3 | 1 | 1D | 3 | 1 | 0 |
| Filtered (*As, Cu, Pb, Zn, Ca, Na, K, Mg, Mn*) | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 1E | 1 | 0 | 0 | 6 | 1 | 1E | 3 | 1 | 1D | 0 | 0 | 0 |
| VOCs (BTEX) | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered | 3 | 1 | 0 | 3 | 1 | 0 | 3 | 1 | 0 | 3 | 1 | 1E | 1 | 0 | 1E | 6 | 1 | 1E | 0 | 0 | 0 | 3 | 1 | 0 |
| TPH-gas | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered | 3 | 1 | 0 | 3 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 6 | 1 | 1E | 0 | 0 | 0 | 3 | 1 | 0 |
| TPH-diesel | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered | 3 | 1 | 1D | 3 | 1 | 1D | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 6 | 1 | 1E | 3 | 1 | 1D | 3 | 1 | 0 |
| Filtered | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 6 | 1 | 1E | 3 | 1 | 1D | 0 | 0 | 0 |
| PAHs | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered | 3 | 1 | 1D | 3 | 1 | 1D | 0 | 0 | 0 | 3 | 1 | 1E | 1 | 0 | 1E | 6 | 1 | 1E | 3 | 1 | 1D | 3 | 1 | 0 |
| Filtered | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 1E | 1 | 0 | 0 | 6 | 1 | 1E | 3 | 1 | 1D | 0 | 0 | 0 |
| Cl, SO4, pH, alkalinity | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered | 3 | 1 | 0 | 3 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Filtered | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 0 | 1 | 0 | 0 | 6 | 1 | 0 | 3 | 1 | 0 | 0 | 0 | 0 |
| **Arkema -- Acid Plant Area** | | | | | | | | | | | | | | | | | | | | | | | | |
| VOCs (full project list) | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered | 3 | 1 | 0 | 3 | 1 | 0 | 3 | 1 | 0 | 3 | 1 | 1E | 1 | 0 | 1E | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 0 |
| DDT, DDE, DDD | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered | 3 | 1 | 1D | 3 | 1 | 1D | 0 | 0 | 0 | 3 | 1 | 1E | 1 | 0 | 1E | 0 | 0 | 0 | 3 | 1 | 1D | 3 | 1 | 0 |
| Filtered | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 1E | 1 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 1D | 0 | 0 | 0 |
| Ca, K, Mg, Mn, Na | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered | 3 | 1 | 0 | 3 | 1 | 0 | 0 | 0 | 0 | 3 | 1 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 0 | 0 | 0 | 0 |
| Cl, SO4, pH, alkalinity | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered | 3 | 1 | 0 | 3 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Filtered | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 0 | 0 | 0 | 0 |

Table B5-1. Summary of Transition Zone Water, Groundwater, Bulk Sediment, and Field Quality Control Samples for the Pilot Study.

| | In-Situ Diffusion Samplers | | | | | | | | | In-Situ Direct Samplers | | | | | | | | | Ex-Situ Extraction | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Small Volume Peeper | | | Large Volume Peeper | | | Vapor Diffusion | | | Trident Probe | | | UltraSeep | | | Push-Point[b] | | | Power Grab & Centrifuge Extraction | | | **Power Grab Bulk Sediment Samples** | | |
| | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC | Nat | Rep[a] | FQC |
| **Arkema -- Chlorate Plant Area** | | | | | | | | | | | | | | | | | | | | | | | | |
| Metals | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered (*Cr*) | 3 | 1 | 1D | 3 | 1 | 1D | 0 | 0 | 0 | 3 | 1 | 1E | 1 | 0 | 1E | 0 | 0 | 0 | 3 | 1 | 1D | 0 | 0 | 0 |
| Filtered (*Cr, Ca, K, Mg, Mn, Na*) | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 1E | 1 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 1D | 0 | 0 | 0 |
| Perchlorate, Cl, SO4, pH, alkalinity | | | | | | | | | | | | | | | | | | | | | | | | |
| Unfiltered (*Perchlorate*) | 3 | 1 | 1D | 3 | 1 | 1D | 0 | 0 | 0 | 3 | 1 | 1E | 1 | 0 | 1E | 0 | 0 | 0 | 3 | 1 | 1D | 3 | 1 | 0 |
| Filtered (*Perchlorate, Cl, SO4, pH, alkalinity*) | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 3 | 1 | 0 | 0 | 0 | 0 |

**Notes:**

[a]A replicate sample will be collected by each method (except the UltraSeep) at a single location associated with the ARCO and two Arkema study areas.

[b]Push-point sampling will target collection of deep transition zone water and groundwater.

NAT = natural sample
Rep = replicate sample
D = decon blank
E = equipment rinsate blank (will include filtered and unfiltered, as appropriate)
FQC = field quality control sample

# ATTACHMENT 1

# ATTACHMENT 1

# COLUMBIA ANALYTICAL SERVICES

# PROCEDURE FOR EXTRACTION OF INTERSTITIAL / PORE WATER FROM SEDIMENT

# PROCEDURE FOR EXTRACTION OF INTERSTITIAL / PORE WATER FROM SEDIMENT

1. LABWARE RINSING: **Polycarbonate glassware must not be allowed to come into contact with acetone or methylene chloride!** All labware used for extraction must be rinsed with dilute HCl followed by DI water. All labware must be dried prior to further rinsing (air drying works best for polycarbonate labware; methanol may also be used if time is limited. Acetone may be used for all non-polycarbonate labware). All polycarbonate labware must then be rinsed with 0.1% w/v tropolone in hexane, followed by hexane; all other labware must be rinsed with 0.1% w/v tropolone in hexane or methylene chloride, followed by hexane or methylene chloride. Inspect all centrifuge bottles to be employed; if any appear to be in danger of breakage, they must be discarded.

2. All transferring of samples must be carried out in an isolated environment under at least 2 atmospheres positive $N_2$ pressure. Sample centrifugation must be carried out at or below 10° Celsius.

3. Note and record the nature of the samples.

4. Transfer sediment with all standing water to centrifuge bottles.

5. If the client requested a method blank, use 1000 mL reagent water and polycarbonate labware previously employed in an interstitial water from sediment extraction. If the client requested matrix spikes, use 1000 mL reagent water spiked with analytes at the same concentration as in the routine organotins in water extraction; do not add surrogates.

6. Balance bottles to be spun opposite each other to within 1 gram and centrifuge for 30 minutes at 1000 - 3000 G (1100 - 3250 RPM for a 10 inch rotor radius).

7. Very sandy sediments often do not yield sufficient volumes of water. For these samples, it will be necessary to employ at least two water-from-sand recovery apparatuses; make a note of this on the benchsheet as these samples will be exposed to the atmosphere during their interstitial water extraction.

8. Decant water into (a) clean polycarbonate centrifuge bottle(s) and balance as above. It is expected that some sediment will be transferred in this step. If necessary, balance the bottles with centrifuge bottles filled with water.

9. Centrifuge a second time at 3000 G.

10. If the client requested that the samples be filtered, use 0.4 µm polycarbonate filters, polycarbonate Erlenmeyer flasks and porcelain Büchner funnels.

11. Very carefully decant water from the centrifuge bottles (if the water was not filtered) or Erlenmeyer flasks (if the water was was filtered) into polycarbonate sample bottles. If sediment is resuspended during decantation, the samples must be recentrifuged before any more water is decanted.

12. Add 0.5-1 mL concentrated HCl to each sample bottle to preserve (i.e., prevent growth of algae or other life) and store at or below 4° Celsius.

13. Record any difficulties, oddities or deviations from procedure.

Approved / Date

5-8-00

Extraction Manager

5-9-00

Instrument Operator

Quality Assurance