CARTY LAKE SEDIMENT MONITORING SAMPLING AND ANALYSIS PLAN

FORMER PACIFIC WOOD TREATING CO. SITE
FACILITY ID 1019, CLEANUP SITE ID 3020

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The material and data in this plan were prepared under the supervision and direction of the undersigned.

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ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
CAP	cleanup action plan
CFR	Code of Federal Regulations
cm	centimeter(s)
COC	chain of custody
COMP	comprehensive operations and maintenance plan
CUL	cleanup level
DGPS	differential global positioning system
dioxins	polychlorinated dibenzo dioxins and furans
DMMP	Dredged Material Management Program
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management
ISM	incremental sampling methodology
LCS	laboratory control sample
MFA	Maul Foster & Alongi, Inc.
Port	Port of Ridgefield
PSEP	Puget Sound Estuary Program
PWT	Pacific Wood Treating Co.
QA/QC	quality assurance
QC	quality control
REL	remediation level
RNWR	Ridgefield National Wildlife Refuge
RSD	relative standard deviation
SAP	sampling and analysis plan
TEQ	toxicity equivalent
TOC	total organic carbon
UCL	upper confidence limit
USEPA	U.S. Environmental Protection Agency
WAC	Washington Administrative Code

1 INTRODUCTION

On behalf of the Port of Ridgefield (Port), Maul Foster & Alongi, Inc. (MFA) has prepared this sampling and analysis plan (SAP) for post-remedy monitoring to be conducted in Carty Lake. Carty Lake is located in the Ridgefield National Wildlife Refuge (RNWR), adjacent to the former Pacific Wood Treating Co. (PWT) site in Ridgefield, Washington (see Figure 1-1). PWT operated a wood-treating facility from 1964 to 1993 at the Port's Lake River Industrial Site, now known as Miller's Landing.

On November 5, 2013, the Port entered into a Consent Decree with the State of Washington requiring remedial action to address contaminated sediments in Carty Lake. The selected remedial action was substantively completed in 2014 and consisted of sediment excavation, placement of a clean sand cap layer, and stabilization of a treated-wood bulkhead as described in the cleanup action plan (CAP) (Washington State Department of Ecology [Ecology], 2013). In addition, the CAP specifies institutional controls to limit fishing in the lake. The remedy includes post-remedial monitoring, which will assess the efficacy of the remedial action and quantify the reduction in concentrations relative to the cleanup level (CUL) (Ecology, 2013).

The Consent Decree requires a comprehensive operations and maintenance plan (COMP) that summarizes requirements for inspection and maintenance of former PWT site cleanup actions; includes actions required to operate and maintain equipment, structures, or other remedial systems (including management and maintenance of soil caps); and describes compliance monitoring plans. This SAP addresses the compliance monitoring plan for cleanup actions in Carty Lake and will be an appendix to the forthcoming COMP.

This SAP describes sampling objectives and methods that will be used to meet compliance monitoring requirements. This SAP is generally consistent with current Puget Sound Estuary Program (PSEP) and U.S. Environmental Protection Agency (USEPA) protocols for sampling and analysis (PSEP, 1986, 1997a,b; USEPA, 1993) and standard USEPA methods based on USEPA test methods for evaluating solid waste, physical/chemical methods (also known as SW-846) requirements, as amended (USEPA, 1986). This SAP meets the requirements of Washington Administrative Code (WAC) 173-340-820, and its contents are consistent with the Sediment Management Standards (WAC 173-204) and guidance provided in Ecology's Sediment Cleanup Users Manual II (Ecology, 2015).

1.1 Background

The CAP identifies remediation levels (RELs) based on risk-based ecological factors and a CUL for polychlorinated dibenzo-p-dioxins and furans (collectively referred to as dioxins) in Carty Lake sediments (see Table 1-1).[1] As described in the Carty Lake Engineering Design Report (MFA, 2014), areas in the southern end of Carty Lake that exceeded RELs were excavated and treated with a clean

[1] RELs protective of ecological resources are congener-specific; the CUL is based on human health considerations and is evaluated as a dioxin toxicity equivalent (TEQ).

sand layer. The planned post-excavation surface was well-characterized prior to finalizing the project design, and the excavation prism was conservatively designed to remove contaminants (MFA, 2014). Confirmation monitoring will be conducted in surface sediments of the active remedy area five years after remedy completion to quantify the long-term effectiveness of the cleanup action, i.e., the reductions in dioxin concentrations relative to RELs and the CUL of 5 nanograms per kilogram dioxin TEQ.

1.2 Investigation Objectives

The objective of this SAP is to provide procedures for collection of data of sufficient quality to characterize the long-term effectiveness of the cleanup action in the remedy area (see Figure 1-2). The average concentration and variability of surface sediment (0 to 10 centimeters [cm] deep) dioxins in the remedy area will be quantified. Sampling will be conducted in a way that ensures that results are reproducible, to the extent practicable, and that results are representative.

This SAP specifies field and analytical methods, including quality assurance (QA) and quality control (QC) requirements.

1.3 Sampling Schedule

The CAP calls for surface sediment dioxin monitoring in the remedy area five years after cleanup. The remedy was substantively completed in 2014, and thus long-term effectiveness monitoring will be conducted in 2019. Additional Carty Lake sediment sampling after 2019 could be conducted in consideration of eliminating institutional controls on fishing and to further evaluate long-term concentration trends.

2 SITE CONDITIONS

Carty Lake is a 52-acre lake in the RNWR Carty Unit. The National Wetlands Inventory classifies much of Carty Lake as a lacustrine, limnetic, unconsolidated bottom, permanently tidal. The remedy area is in the southern end; this area is a shallow, open water body with a fringe of emergent wetland (Category II lake-fringe) (MFA, 2013). During the rainy season, Gee Creek and Carty Lake can be hydraulically connected at the lake's northern end. During most of the year, Carty Lake has no outlet. Water depths range from 3 to 10 feet, varying seasonally, and are generally greater during winter and spring and lower during summer and fall. Water fluctuations are generally muted relative to Lake River, with increases and decreases occurring more gradually because there is no direct connection with the Columbia River.

Hydrodynamics and grain size distribution indicate that Carty Lake features a low-energy, depositional environment. Percent fines in Carty Lake are uniformly high, generally over 75 percent fines. Carty Lake's hydraulic exchange with other surface water bodies is limited to unusually high water events.

Further, given that human access to Carty Lake is limited and boat access is restricted, anthropogenic high-velocity events are not expected.

Predicted post-excavation (i.e., prior to clean sand placement) sediment concentrations are shown in Figure 2-1. Predicted post-remedy (i.e., following excavation and clean sand placement but prior to long-term recovery) surface sediment dioxin concentrations are shown in Figure 2-2. The estimated sediment concentrations were calculated as described in the Carty Lake Engineering Design Report (MFA, 2014).

3 SEDIMENT SAMPLING

The incremental sampling methodology (ISM) will be used to characterize the average concentration of dioxins in sediments (HDOH, 2009, 2011; ITRC, 2012). ISM characterizes the average concentration of contaminants in a predefined area termed the decision unit. Samples (called increments) are collected from multiple locations within a decision unit under evaluation. The increments are combined into one sample (called an ISM sample) and analyzed to obtain a representative average contaminant concentration for the entire decision unit. Replicates are collected to define variability due to sampling error or spatial heterogeneity. ISM obtains data that are more representative of average concentrations than areawide concentrations derived from discrete or composite samples (HDOH, 2009; ITRC, 2012).

3.1 ISM Design

ISM requires selection of a decision unit(s). A decision unit is the area and depth of sediment to be represented by the sampling process. The sampling objective is to characterize the average concentration of dioxins in surface sediments in the remedy area. As specified in the CAP (Ecology, 2013), surface sediments in Carty Lake are defined as the top 10 cm of sediment. The proposed decision unit therefore spans the remedy area and extends from surface to 10 cm below mudline (see Figure 3-1).

ISM sampling theory demonstrates that 30 increments of an adequate mass from a given decision unit of any size will generally result in a sample that is adequately representative of the average contaminant level in the decision unit (HDOH, 2009; ITRC, 2012). Additional increments may reduce error in estimating the true mean, and more than 30 increments are typically recommended when spatial heterogeneity is expected to be high. Since dioxin spatial heterogeneity is expected to be low following remedy implementation, 30 increments will be collected during the monitoring event.

Three field replicates (called a triplicate) will be used to assess sample variability (i.e., relative standard deviation [RSD]) and to assign confidence levels to results. If it is determined that additional monitoring samples are necessary and the initial ISM sample RSD is high, i.e., above 30 percent, triplicates will also be collected during subsequent monitoring events (ADEC, 2009). If RSD is low but it is determined that average concentrations in subsequent monitoring samples have changed relative to the initial ISM sample, triplicates may be collected to confirm acceptable sample variability.

Increment locations were selected based on a systematic random approach using a triangular grid (using ArcGIS 10 and Visual Sample Plan 6). Using a systematic random grid, as opposed to a simple random sampling approach, reduces the probability of missing areas with significantly elevated concentrations. Three ISM samples from 30 locations each (A, B, and C) are assigned for collection of the triplicate composite increment samples A, B, and C. Increment locations are shown on Figure 3-1. Subsequent monitoring events, if necessary, will be collected at location set A.

All ISM samples will be analyzed for dioxins and total organic carbon (TOC).

3.2 Sampling Methods

Surface sediment samples will be retrieved by a 1-inch-diameter, thin-walled, stainless steel sampling tube. The sampling tubes will be manually advanced to a depth greater than 10 cm. The sampling tube will be withdrawn and the increment extruded, using a plunger, onto a clean work surface. The increment will be measured and trimmed to 10 cm. If increment recovery is poor, the increment will be discarded and resampled within a few feet of the original location. Approximately 100 grams per increment, for a total of 3 kilograms per ISM sample, will be collected to provide the overall mass required by the analytical laboratory.

If it is determined that sampling tubes do not achieve sufficient recovery, a grab sampler (e.g., clamshell-style petite ponar or clamshell-style petite Van Veen) will be deployed from a vessel or land, depending on the water level. The speed of the grab sampler's descent will be controlled to minimize disturbance of the sediment. The speed of ascent will also be controlled to minimize loss of sediment from washout. The sediment sample will be inspected upon retrieval to ensure that the grab sampler was completely closed and retained all sediment, including any surficial fines. Upon retrieval of an acceptable sediment sample, an approximately 100-gram increment that extends from 0 to 10 cm will be collected from the retrieved material. Sediment that is in contact with the sides of the sampler will not be sampled.

Procedures for handling and analyzing sediment are as follows:

- Samplers will wear clean, disposable gloves while collecting samples. Gloves will be changed after collection of each ISM replicate.
- Field activities and conditions and sampling data (e.g., sample description) will be recorded in a field notebook. Any deviations from the sampling protocol will be noted on field records and will be brought to the attention of the project manager. General sediment observations, such as description of surface materials, soil type and variability within decision units, and any staining or discoloration, will be recorded.
- Increment composites will be placed in glass jars. Samples will be labeled, stored in iced shipping containers with chain-of-custody (COC) documentation, and transported to the contract laboratory.
- Each increment composite will be analyzed for dioxins and TOC, using USEPA Method 1613B and PSEP/SM Method 5310B, respectively. Laboratory test methods, QA/QC procedures, and data validation and reporting procedures are described in Section 4.

3.3 Positioning

A differential global positioning system (DGPS) will be used to locate the sampling position for each proposed location shown on Figure 3-1. Sample locations will be determined to an accuracy of ±3 meters. Horizontal coordinates will be referenced to the Washington South State Plane HARN (NAD83). Reasonable effort will be made to collect sediment from each location; however, some locations may remain inaccessible. Sample locations may be field adjusted and will be collected as close as possible to the intended sample location. The DGPS will be used to record the location of each location that has been field adjusted. Locations may be accessed by boat or on foot (e.g., locations adjacent to the shoreline).

3.4 Equipment Decontamination Procedures

Nondisposable sampling equipment that comes in direct contact with the sample (e.g., scoops, bowls) will be decontaminated before use for each ISM replicate, according to the following procedure:

- Distilled-water rinse.
- Wash with scrub brush and Alconox™ soap and distilled water solution.
- Distilled-water rinse.
- Methanol solution rinse (1:1 solution with distilled water).
- Final distilled-water rinse.

The sampling tube or grab sampler will be decontaminated before use for each ISM replicate according to the following procedure:

- Rinse with site water.
- Wash with scrub brush and Alconox soap and distilled water solution.
- Rinse with distilled water.

The thoroughness of equipment decontamination will be verified by collection and analysis of equipment rinsate samples. Liquid generated by decontamination will be properly handled, according to procedures specified in Section 3.5.

3.5 Management of Investigation-Derived Waste

Decontamination fluids will be collected and stored in sealed plastic buckets and disposed of through a permitted service provider. Personal protective equipment will be disposed of in a sanitary landfill.

3.6 Field QA/QC Samples

QC samples will be collected to ensure that field samples and quantitative field measurements are representative of the media collected. Field QA/QC samples and collection frequency are as follows:

- **Equipment Rinsate Blanks—**To ensure that decontamination procedures are sufficient, an equipment rinsate blank will be collected when nondedicated equipment is used. One equipment rinsate blank will be collected for each monitoring event. Equipment rinsate blanks will be collected by passing laboratory-provided deionized/distilled water through or over sampling equipment and will be submitted for analysis of dioxins by USEPA Method 1613B. The rinsate blank results will be evaluated during data quality review.
- **Field Replicates—**Field replicates are collected to measure sampling and laboratory precision. Samples will be collected in triplicate (three sets of 30 increment samples) (see Section 3.1). The field replicate results will be evaluated during data quality review (see Section 4.3).

3.7 Work Documentation

Accurate recordkeeping will be maintained throughout the field sampling effort. A field notebook will be prepared documenting the following information:

- Name(s) of the person(s) collecting samples
- Sampling vessel and field staff
- A record of site health and safety meetings and updates
- Weather conditions
- Date and time of collection of each sample
- Representative photographs with sample location ID
- Gross characteristics of the sample, such as organic matter, biota, debris, and sheen
- Physical description of the sample soil, consistent with the Unified Soil Classification System (includes soil type, density/consistency of soil, color)
- Description of material selectively removed from the sample before filling of containers for chemical analysis (e.g., gravel, wood debris)
- Any deviation from this Ecology-approved SAP

3.8 Sample Containers, Preservation, and Transport

Sample container, preservations, and holding-time requirements are summarized in Table 3-1. All sediment samples will be collected in glass jars. Each sample will have an adhesive plastic or waterproof paper label affixed to the container and will be labeled at the time of collection. Samples will be uniquely identified with a sample identification that, at a minimum, specifies sample name, sample location, and sample date/time. Sample containers, sample coolers, and packing materials will be supplied by the laboratory. The laboratory will maintain documentation certifying the cleanliness of containers provided. The samples will be stored in iced coolers at 4 ($\pm$ 2) degrees Celsius (°C).

Individual sample containers, along with COC forms, will be placed in a sealed plastic bag. Glass jars will be packed to prevent breakage and will be separated in the shipping container by a shock-absorbent material, such as bubble wrap. Ice in sealed plastic bags will be placed in the cooler to maintain a temperature of approximately 4°C.

When the cooler is full, the COC form will be placed in a zip-locked bag inside the cooler and a temperature blank will be placed in the cooler. Coolers will be taped and then sealed with two COC seals. The temperature blanks are prepared by the laboratory, using analyte-free (reagent) water. Temperature blanks are used by the laboratory to record the temperature of each cooler used to transport samples from the field to the laboratory. The laboratory will verify that the temperature blank measurement is 4 (±2)°C.

Coolers will be transported to the laboratory by courier or overnight shipping service. Packing and shipping procedures are consistent with U.S. Department of Transportation regulations as specified in 49 Code of Federal Regulations (CFR) 173.6 and 49 CFR 173.24.

3.9 Sample Custody, Packaging, and Shipping

Sample custody will be tracked from point of origin through final analysis and disposal, using a COC form, which will be filled out with the appropriate sample and analytical information as soon as possible after samples are collected. For purposes of this work, custody will be defined as follows:

- In plain view of MFA field representatives
- Inside a cooler that is in plain view of MFA field representatives
- Inside any locked space such as a cooler, locker, car, or truck to which the MFA field representatives have the only available key(s)

After sample containers have been filled, they will be packed on ice in coolers and then transported to the laboratory in iced shipping containers (with a custody seal affixed).

COC procedures will begin in the field and will track delivery of the samples to the laboratories. Specific procedures are as follows:

- Samples will be packaged and shipped in accordance with U.S. Department of Transportation regulations as specified in 49 CFR 173.6 and 49 CFR 173.24.
- Individual sample containers will be packed to prevent breakage.
- A sealed envelope containing COC forms will be enclosed in a plastic bag inside the cooler.
- Signed and dated COC seals will be placed on all coolers before shipping.

Upon transfer of samples to the laboratory, the COC form will be signed by the persons transferring custody of the coolers. Upon receipt of samples at the laboratory, the shipping container seal will be broken and the condition of the samples will be recorded by the receiver. Copies of the COC will be included in laboratory reports and data validation memoranda.

3.10 Field Instrumentation

Staff or subcontractors responsible for navigation will confirm proper operation of the navigation equipment daily. This verification may consist of internal diagnostics or visiting a location with known coordinates to confirm the coordinates indicated by the navigation system. No other field equipment requires calibration. Any issues will be noted in the field logbook and corrected before sampling operations continue.

4 LABORATORY MEASUREMENTS AND QA/QC PROCEDURES

4.1 Laboratory Test Methods and Reporting Limits

Chemical testing will be conducted using the analytical methods and detection limits presented in Table 4-1. A laboratory that can achieve detection limits lower than those required by the associated USEPA method will be selected. Samples will be maintained according to the appropriate holding times and temperatures for each analysis.

MFA will submit samples representing the decision unit replicate for chemical ISM analysis. The decision unit will have equal mass collected from its 30 increments (approximately 100 grams wet weight per increment). As discussed above, the approximately equal mass collected from each increment will be field consolidated to generate a sample of approximately 3 kilograms (wet weight).

The laboratory will air dry each decision unit sample at room temperature. The entire volume of each sample will be chopped and sieved to facilitate obtaining a representative subsample and improving analyte extraction efficiency. The sample will be sieved using an American Society for Testing and Materials No. 10 (2-millimeter) sieve.

Once the sample is dried and sieved, the laboratory will perform the "1-dimensional slabcake" subsampling procedure to sub-aliquot sample volume to be used for analysis. The slabcake procedure involves spreading the sample at a consistent depth in a line, using 20 or more passes and using a square scoop to cut across the line as needed to create an aliquot for each analysis. Samples for TOC will be ground prior to analysis.

Each sub-aliquot will be placed in its own, single-sample container, consistent with the volume and preservation requirements indicated in Table 4-1. The final mass of the sample must be sufficient to run the requested analyses and attain the requested reporting limit. Please note that sufficient sample volume must be composited by the laboratory to create a laboratory duplicate sample and matrix spike and matrix spike duplicate, where applicable.

The remaining volume of the composite samples will be archived at the laboratory at -18°C.

An ISM standard operating procedure is included as the appendix.

4.2 Laboratory Instrumentation

Laboratory QA/QC will be maintained through the use of standard USEPA methods, based on USEPA test methods for evaluating solid waste, physical/chemical methods (also known as SW-846) requirements, as amended (USEPA, 1986). Table 4-1 presents the data quality objectives of solid-phase testing for precision, accuracy, and completeness, while Table 4-2 summarizes general laboratory QA/QC procedures. The laboratory will also meet QA/QC requirements specified in the 2010 Dredged Material Management Program (DMMP) clarification paper (Hoffman and Fox, 2010). If the laboratory does not meet QA/QC acceptance limits, particularly if estimated maximum potential concentration qualifiers are anticipated, MFA will be contacted and corrective actions consistent with DMMP requirements will be taken (Hoffman and Fox, 2010).

4.2.1 Preventive Maintenance

Preventive maintenance of laboratory equipment will be the responsibility of the laboratory personnel and analysts. This maintenance includes routine care and cleaning of instruments, and inspection and monitoring of carrier gases, solvents, and glassware used in analyses. The preventive-maintenance approach for specific equipment will follow the manufacturers' specifications and good laboratory practices.

Precision and accuracy data will be examined for trends and excursions beyond control limits to determine evidence of instrument malfunction. Maintenance will be performed when an instrument begins to change, as indicated by the degradation of peak resolution, shift in calibration curves, decrease in sensitivity, or failure to meet any of the QC criteria.

4.2.2 Laboratory QA/QC Checks

QC samples and procedures verify that an instrument is calibrated properly and remains in calibration throughout the analytical sequence, and that the sample preparation procedures have been effective and have not introduced contaminants into the samples. Additional QC samples are used to identify and quantify positive or negative interference caused by the sample matrix. The following laboratory QC procedures are required for most analytical procedures:

- **Calibration Verification—**Initial calibration of instruments will be performed at the start of the project or sample run, as required, and when any ongoing calibration does not meet control criteria. The number of points used in the initial calibration is defined in the analytical method. To track instrument performance, continuing calibration will be performed as specified in the analytical method. If a continuing calibration does not meet control limits, analysis of project samples will be suspended until the source of the control failure is either eliminated or reduced to within control specifications. Any project samples analyzed while the instrument was outside control limits will be reanalyzed.
- **Method Blanks—**Method blanks are used to assess possible laboratory contamination of samples associated with all stages of preparation and analysis of samples and extracts.

The laboratory will not apply blank corrections to the original data. A minimum of one method blank will be analyzed for every sample extraction group, or one for every 20 samples, whichever is more frequent.

- **Laboratory Control Samples (LCSs)—**LCSs are fortified with target analytes to provide information on analysis accuracy. Analyses of LCSs will be performed by the lab at a frequency that satisfies the analytical method requirements.
- **Laboratory Duplicates—**Laboratory duplicates are used to assess laboratory batch precision associated with all stages of preparation and analysis of samples and extracts. Laboratory duplicates will be analyzed according to method frequency requirements.
- **Surrogate Spike Compounds—**Surrogate spikes are used to evaluate the recovery of an analyte from individual samples. All project samples to be analyzed for organic compounds will be spiked with appropriate surrogate compounds as defined in the analysis method, i.e., carbon-13 labeled internal standards for the dioxin method. Recoveries determined using these surrogate compounds will be reported by the laboratory; however, the laboratory will not correct sample results using these recoveries.

4.3 Data Reduction, Validation, and Reporting

The analytical laboratory will submit analytical data packages that include laboratory QA/QC results to permit independent and conclusive determination of data quality. Data quality will be determined by MFA, using the data evaluation procedures described in this section. The results of the MFA evaluation will be used to determine if the project data quality objectives have been met.

4.3.1 Field Data Reduction

Daily internal QC checks will be performed for field activities. Checks will consist of reviewing field notes and field activity memoranda to confirm that the specified measurements and procedures are being used. The need for corrective action will be assessed on an ongoing basis, in consultation with the project manager.

4.3.2 Laboratory Evaluation

Initial data reduction, evaluation, and reporting at the analytical laboratory will be carried out as described in USEPA SW-846 manuals for organic analyses (USEPA, 1986), as appropriate. Additional laboratory data qualifiers may be defined and reported to further explain the laboratory's QC concerns about a particular sample result. All additional data qualifiers will be defined in the laboratory's case narrative report associated with each case.

4.3.3 Data Deliverables

Laboratory data deliverables are listed below. Electronic deliverables will contain the same data that are presented in the hard-copy report.

- Transmittal cover letter
- Case narrative
- Analytical results
- COC documentation
- Surrogate recoveries
- Method blank results
- LCS results
- Laboratory duplicate results

4.3.4 Data QA/QC Review

MFA will evaluate the laboratory data for precision, completeness, accuracy, and compliance with the analytical method. Dioxin data will be reported consistent with recent dioxin data treatment guidance (Ecology, 2015). The data review will include an assessment of laboratory performance criteria and will be consistent with the USEPA national functional guidelines (USEPA, 2011, 2014). Results of the data review will be provided as a memorandum to be included with the data report and lab result sheets. Ecology will be notified before development of the data review memorandum if laboratory results indicate any significant data quality issues.

Data qualifiers, as defined by the USEPA, are used to classify sample data according to their conformance to QC requirements. The most common qualifiers are listed below:

J—Estimate, qualitatively correct but quantitatively suspect.
R—Reject, data not suitable for any purpose.
U—Not detected at a specified reporting limit.

Poor surrogate recovery, blank contamination, or calibration problems, among other things, can cause the sample data to be qualified. Whenever sample data are qualified, the reasons for the qualification will be stated in the data evaluation report.

QC criteria not defined in the guidelines for evaluating analytical data are adopted, where appropriate, from the analytical method.

The following information will be reviewed during data evaluation, as applicable:

- Sampling locations and blind sample numbers
- Sampling dates
- Requested analysis
- COC documentation
- Sample preservation
- Holding times
- Method blanks
- Surrogate recoveries
- Laboratory duplicates (if analyzed)

- Field replicates
- Field blanks
- LCSs
- Method reporting limits above requested levels
- Any additional comments or difficulties reported by the laboratory
- Overall assessment

The results of the data evaluation review will be summarized for each data package. Data qualifiers will be assigned to sample results on the basis of USEPA guidelines, as applicable.

4.3.5 Evaluation of ISM Replicates

Field QC sampling will include the collection of triplicate samples (see Section 3.1). The RSD of the analytical results for triplicate samples will be calculated to measure data precision. The RSD is calculated using the following equation:

$$RSD\% = \frac{100\% * \text{Standard Deviation}}{\text{Average}}$$

Lower RSD values are desirable, as the lower the RSD, the greater confidence there is that the average approximates a normal distribution and that the average contaminant concentrations are adequately representative of the decision unit (HDOH, 2009). It is assumed that data normally distributed have an RSD of 30 percent or less (ADEC, 2009). Acceptability of the calculated RSD percent will be evaluated in the context of such considerations as analytical results at or near the method reporting limit, which may exhibit a greater level of variability and, therefore, an elevated RSD (ADEC, 2009). However, if results are non-detect or less than 5 times the method reporting limit RSDs will not be calculated.

4.3.6 Data Management and Reduction

MFA uses EQuIS environmental data management software to manage all laboratory data. The laboratory will provide the analytical results in electronic EQuIS-deliverable format. Following data evaluation, data qualifiers and analytical results will be entered into MFA's EQuIS database as well as into Ecology's Environmental Information Management (EIM) database. Consistent with WAC 173-340-840(5) and Ecology Toxics Cleanup Program Policy 840 (Data Submittal Requirements), data will be submitted simultaneously in both written and electronic formats.

Data may be reduced to summarize particular data sets and to aid interpretation of the results. Statistical analyses may also be applied to results. Data reduction QC checks will be performed on all hand-entered data, any calculations, and any data graphically displayed. Data may be further reduced and managed using one or more of the following computer software applications:

- Microsoft Excel® (spreadsheet)
- EQuIS (database)

- Ecology's EIM (database)
- AutoCad and/or Arc GIS (graphics)
- USEPA ProUCL (statistical software)

5 REPORTING

Ecology will be notified in writing at least 30 days before monitoring activities begin. A data report will be prepared and submitted to Ecology within 30 days of receipt and review of the validated analytical data. Data will be submitted to Ecology's EIM data system when the final report is submitted. The data report will include a brief summary of data collection procedures (noting, in particular, deviations from the SAP); increment locations; summary of field notes; analytical results; a data validation memorandum; and data interpretation. Data interpretation will focus on the following issues to assess remedy action effectiveness and compliance:

- Whether the dioxin TEQ and congener concentrations are representative of the decision unit.
- Dioxin concentration trends for the decision unit over time, if applicable.
- TOC trends for the decision unit over time may be used to understand dioxin TEQ trends, if applicable.
- Evaluation of ISM concentrations relative to the CUL. The CUL objective will be attained if one of the following is true:
  - The mean of replicate ISM sample results does not exceed the CUL and the RSD does not exceed 30 percent.
  - If the RSD exceeds 30 percent, compliance will be demonstrated if the 95 percent upper confidence limit (UCL) of the replicate sample results or the maximum replicate sample result does not exceed the CUL. The UCL will be calculated using the Student's-t (representing the low range estimate) and Chebyshev (representing the high range estimate) UCL methods (ITRC, 2012). The UCL method accounts for the increased likelihood of underestimating the true mean when sample variability is high (ITRC, 2012).

The CAP calls for confirmation monitoring in the active remedy area five years after remedy completion. Additional confirmation sampling of Carty Lake sediment could be conducted in consideration of eliminating institutional controls on fishing in the lake, and to evaluate long-term concentration trends.

LIMITATIONS

The services undertaken in completing this plan were performed consistent with generally accepted professional consulting principles and practices. No other warranty, express or implied, is made. These services were performed consistent with our agreement with our client. This plan is solely for the use and information of our client unless otherwise noted. Any reliance on this plan by a third party is at such party's sole risk.

Opinions and recommendations contained in this plan apply to conditions existing when services were performed and are intended only for the client, purposes, locations, time frames, and project parameters indicated. We are not responsible for the impacts of any changes in environmental standards, practices, or regulations subsequent to performance of services. We do not warrant the accuracy of information supplied by others, or the use of segregated portions of this plan.

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TABLES

Table 1-1
Sediment Performance Standards
Former PWT Site
Ridgefield, Washington

Analyte	Performance Standards (ng/kg)
Cleanup Level	
Dioxin TEQ	5.0E+00
Remediation Levels	
2,3,7,8-TCDD	3.3E+00
1,2,3,7,8-PeCDD	9.8E+01
1,2,3,4,7,8-HxCDD	2.0E+02
1,2,3,6,7,8-HxCDD	1.2E+03
1,2,3,7,8,9-HxCDD	1.2E+03
1,2,3,4,6,7,8-HpCDD	3.1E+05
OCDD	1.0E+07
2,3,7,8-TCDF	8.6E+01
1,2,3,7,8-PeCDF	5.5E+02
2,3,4,7,8-PeCDF	6.5E+00
1,2,3,4,7,8-HxCDF	9.8E+02
1,2,3,6,7,8-HxCDF	9.8E+02
1,2,3,7,8,9-HxCDF	9.8E+02
2,3,4,6,7,8-HxCDF	9.8E+02
1,2,3,4,6,7,8-HpCDF	2.5E+05
1,2,3,4,7,8,9-HpCDF	2.5E+05
OCDF	1.0E+07

NOTES:
ng/kg = nanograms per kilogram.
TEQ = toxicity equivalent.

Table 3-1
Container Requirements, Holding Times, and Preservation
Former PWT Site
Ridgefield, Washington

Parameter	Sample Size*	Container Size and Type	Hold Time for Analysis	Preservation
Dioxins	3.0 kg	1-gallon jar (protect from light)	30 days	4°C
			1 year	-18°C
Total organic carbon			28 days	4°C
			6 months	-18°C

NOTES:

°C = degrees Celsius.

dioxins = polychlorinated dibenzo-p-dioxins and furans.

kg = kilogram(s).

*Sample size is for each decision unit replicate. Approximately 100 grams will be collected for each sub-aliquot.

Table 4-1
Analytical Methods and Data Quality Objectives
Former PWT Site
Ridgefield, Washington

	Analytical Method	Units	Practical Quantitation Limit	Level of Detection*	Precision	Laboratory Control Sample Accuracy	Internal Standard Accuracy	Completeness
Dioxins								
2,3,7,8-TCDF	USEPA 1613B	ng/kg	0.5	0.10	NA	75-158% R	24-169% R	100%
2,3,7,8-TCDD	USEPA 1613B	ng/kg	0.5	0.10	NA	67-158% R	25-164% R	100%
1,2,3,7,8-PeCDF	USEPA 1613B	ng/kg	2.5	0.50	NA	80-134% R	24-185% R	100%
2,3,4,7,8-PeCDF	USEPA 1613B	ng/kg	2.5	0.50	NA	68-160% R	21-178% R	100%
1,2,3,7,8-PeCDD	USEPA 1613B	ng/kg	2.5	0.50	NA	70-142% R	25-181% R	100%
1,2,3,4,7,8-HxCDF	USEPA 1613B	ng/kg	2.5	0.50	NA	72-134% R	26-152% R	100%
1,2,3,6,7,8-HxCDF	USEPA 1613B	ng/kg	2.5	0.50	NA	84-130% R	26-123% R	100%
2,3,4,6,7,8-HxCDF	USEPA 1613B	ng/kg	2.5	0.50	NA	70-156% R	28-136% R	100%
1,2,3,7,8,9-HxCDF	USEPA 1613B	ng/kg	2.5	0.50	NA	78-130% R	29-147% R	100%
1,2,3,4,7,8-HxCDD	USEPA 1613B	ng/kg	2.5	0.50	NA	70-164% R	32-141% R	100%
1,2,3,6,7,8-HxCDD	USEPA 1613B	ng/kg	2.5	0.50	NA	76-134% R	28-130% R	100%
1,2,3,7,8,9-HxCDD	USEPA 1613B	ng/kg	2.5	0.50	NA	64-162% R	NA	100%
1,2,3,4,6,7,8-HpCDF	USEPA 1613B	ng/kg	2.5	0.50	NA	82-122% R	28-143% R	100%
1,2,3,4,7,8,9-HpCDF	USEPA 1613B	ng/kg	2.5	0.50	NA	78-138% R	26-138% R	100%
1,2,3,4,6,7,8-HpCDD	USEPA 1613B	ng/kg	2.5	0.50	NA	70-140% R	23-140% R	100%
OCDF	USEPA 1613B	ng/kg	5.0	1.00	NA	63-170% R	NA	100%
OCDD	USEPA 1613B	ng/kg	5.0	1.00	NA	78-144% R	17-157% R	100%
Physical Parameters								
Total organic carbon	PSEP/SM 5310B	%	0.02	0.01	+/- 20% RPD	85-115% R	NA	90%

Table 4-1
Analytical Methods and Data Quality Objectives
Former PWT Site
Ridgefield, Washington

NOTES:

dioxins = polychlorinated dibenzo-p-dioxins and furans.

NA = not applicable.

ng/kg = nanograms per kilogram (parts per trillion).

PSEP = Puget Sound Estuary Program.

R = recovery.

RPD = relative percent difference.

USEPA = U.S. Environmental Protection Agency.

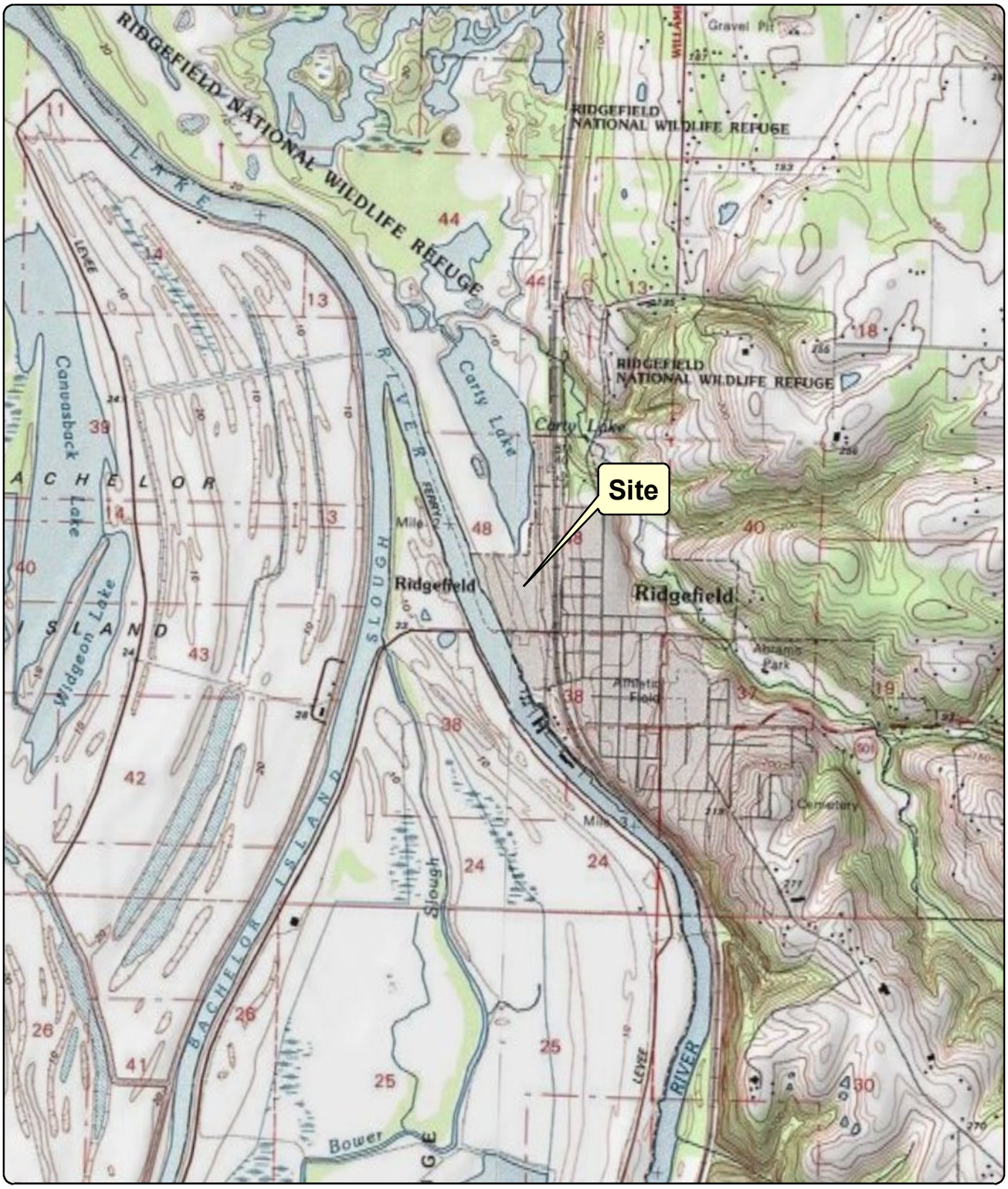
*Level of detection for Method 1613B is based on likely estimated detection limits from Vista Analytical Laboratory. Estimated detection limits may change, depending on matrix conditions and laboratory discretion.

Table 4-2
Analytical Quality Control Requirements
Former PWT Site
Ridgefield, Washington

Analysis Type	Initial Calibration	Ongoing Calibration	Labeled Analogs	Batch Duplicates	Matrix Spikes	LCS/OPR	Method Blanks	Surrogate Spikes	Equipment Rinsate Blank	Field Triplicates
Dioxins	As required by USEPA Method 1613B	Every 12 hours	Every sample	NA	NA	1 per 20 samples	1 per 20 samples	Every sample	1 per sampling event	1
Total organic carbon	As required	1 per 15 samples	NA	1 per 10	NA	1 per 20 samples	1 per 20 samples	NA	NA	1

NOTES:
dioxins = polychlorinated dibenzo-p-dioxins and furans.
LCS = laboratory control sample.
NA = not applicable.
OPR = ongoing precision and recovery sample (used for dioxin analysis).
USEPA = U.S. Environmental Protection Agency.

FIGURES



Source: Topographic Quadrangle obtained from ArcGIS Online Services/NGS-USGS TOPO! US Geological Survey (1999)
7.5-minute topographic quadrangle: Ridgefield
Address: Lake River Industrial Site
111 W. Division Street, Ridgefield, WA 98642
Section: 24 Township: 4N Range: 1W Of Willamette Meridian

Figure 1-1
Site Location

Former PWT Site
Ridgefield, Washington

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0 990 1,980
Feet

Path: X:\9003.01 Port of Ridgefield\401\Projects\06\Lake River Initial Design Report\Fig1-1_Site Location.mxd
Print Date: 4/19/2013
Approved By: J. King
Produced By: D. Coe/ W. Coffey
Project: 9003.01.20

Source: Aerial photograph (2014) obtained from Clark County GIS. Site features and boundaries provided through a survey conducted by Minister & Glaeser Surveying in 2014 and 2015. All features are approximate.

Legend

- Ordinary High Water
- Fish-Mix Rock
- Bank
- Excavation Extent
- Former Berm (Approximate)

Figure 1-2
Remedy Location

Former PWT Site
Ridgefield, Washington

0 50 100
Feet

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Project: 9003.01.40
Produced By: jschane
Approved By: P. Wiescher
Print Date: 6/12/2015

Figure 2-1
Post Excavation Surface Sediment
Former PWT Site
Ridgefield, Washington

LRIS-CL-23	TEQ	PeCDF
	24	2.93

LRIS-CL-22	TEQ	PeCDF
	36	4.21

LRIS-CL-19	TEQ	PeCDF
	26	2.97

LRIS-CL-03	TEQ	PeCDF
	1.1	0.29

LRIS-CL-04	TEQ	PeCDF
	2.1	0.21

LRIS-CL-18	TEQ	PeCDF
	120	**15.2**

LRIS-CL-17	TEQ	PeCDF
	22	2.72

LRIS-CL-02	TEQ	PeCDF
	2.5	0.41

LRIS-CL-01	TEQ	PeCDF
	5.5	1.7

LRIS-CL-16	TEQ	PeCDF
	46	6.49

Legend

- Sediment Sample Location
- Excavation Extent
- Former Berm (Approximate)

Notes:
1. **Bold** value exceeds remediation level.
2. TEQ = Toxicity Equivalent
3. PeCDF = 2,3,4,7,8-Pentachlorodibenzofuran
4. TEQ and 2,3,4,7,8-PeCDF measured in ng/kg (nanograms per kilogram)
5. Conditions shown prior to clean sand placement.

0 20 40
Feet

Source: Aerial photograph (2014) obtained from Clark County GIS.

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Path: X:\9003.01 Port of Ridgefield\40\Projects\06\Long Term Monitoring SAP - Carty Lake\Fig2-1_Post Excavation Surface Sediment.mxd
Print Date: 6/12/2015
Approved By: P. Wiescher
Produced By: jschane
Project: 9003.01.40

Figure 2-2
Modeled Post Remedy Surface Sediment Concentrations

Former PWT Site
Ridgefield, Washington

Legend

- Sediment Sample Location
- Decision Unit
- Former Berm (Approximate)
- Excavation Extent

Notes:
1. **Bold** value exceeds remediation level.
2. TEQ = Toxicity Equivalent
3. PeCDF = 2,3,4,7,8-Pentachlorodibenzofuran
4. TEQ and 2,3,4,7,8-PeCDF measured in ng/kg (nanograms per kilogram)
5. Final conditions assume 100% mixing with clean sand layer.

* The modeled concentration marginally exceeds the remediation level of 6.5 ng/kg. This estimated concentration is based on a number of conservative assumptions and is not expected to result in unacceptable risk for a variety of reasons discussed in the Carty Lake Engineering Design Report (MFA, 2014).

0 20 40
Feet

Source: Aerial photograph (insert date) obtained from Esri ArcGIS Online

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Path: X:\9003.01 Port of Ridgefield\40\Projects\06\Long Term Monitoring SAP - Carty Lake\Fig2-2_Modeled Post Remedy Surface Sediment Concentrations.mxd
Print Date: 5/18/2015
Approved By: P. Wiescher
Produced By: jschane
Project: 9003.01.40

Source: Aerial photograph obtained from ESRI, Inc. ArcGIS Online (2010). Site features and boundaries provided through a survey conducted by Minister & Glaeser Surveying in 2014 and 2015. All features are approximate.

Legend

- Incremental Sample Location A
- Incremental Sample Location B
- Incremental Sample Location C
- Ordinary High Water
- Decision Unit
- Excavation Extent
- Bank
- Fish-Mix Rock

Figure 3-1
Carty Lake
Sample Locations

Former PWT Site
Ridgefield, Washington

0 20 40
Feet

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Path: X:\9003.01 Port of Ridgefield\40\Projects\06\Long Term Monitoring SAP - Carty Lake\Fig3-1_Carty Lake Sample Locations.mxd
Print Date: 5/18/2015
Approved By: P. Wiescher
Produced By: jschane
Project: 9003.01.40

APPENDIX

ISM STANDARD OPERATING PROCEDURE

APEX LABORATORIES, LLC

STANDARD OPERATING PROCEDURE
APPROVAL SIGNATURE PAGE

SOP Title: **Incremental Sampling Methodology (ISM)**

SOP Number: **GS-103 R0 ISM**

Effective Date: June 3rd, 2011

Approval Signatures:

Technical Manager: _____ 6/3/11
David Jack — date

QA Manager: _____ 6-3-11
Evan Holloway (Technical Review) — date

1 INTRODUCTION

This SOP describes the policies and procedures of Apex Laboratories concerning the preparation of soil samples received from Incremental Sampling Methodology (ISM) events. ISM is a sampling procedure that relies on a large number of samples (typically greater than 30) being collected in a certain area and combined into a single sample, rather than a smaller number of discrete samples that are analyzed individually. This procedure involves preparation of the combined sample and differs from normal lab compositing.

2 SCOPE AND APPLICATION

This procedure is typically applicable for analysis of metals and non-volatile organics. Preservation of samples for volatile organic analysis (VOA) is performed in the field. Compositing of preserved ISM VOA samples is not covered by this version of this SOP, which will be revised as necessary. See ITRC guidance for further information on VOA sampling and compositing.

ISM is a very project specific procedure, and should be driven by the client's Sampling Analysis Plan. Contact with the client is essential prior to beginning ISM processing, as the end use of the data may significantly change the procedure used to composite the samples. This SOP is intended as guidance for the steps common for most ISM samples, and is not intended to supersede client instructions as to how their samples should be handled. Modifications will be documented on the ISM request form (example, Appendix A).

3 SUMMARY OF METHOD

The entire volume of each sample is used in this preparation. The samples are air dried, then sieved through a #10 (2 mm mesh) sieve and the material that does not pass through is discarded. The material is either extracted and analyzed as is or further prepared for metals extraction.

4 SAFETY AND ENVIRONMENTAL

4.1 Personal protective equipment (P.P.E.) such as lab coats, nitrile gloves, and safety glasses must be worn while working with samples. Dust masks are optional, but recommended.

4.2 All secondary containers used to store samples or solutions beyond immediate use require proper labeling.

4.3 All waste, rinsate, expired solutions and/or solvents generated by this method should be handled in accordance with Apex's hazardous waste procedures. Care should be taken not to discharge any potentially hazardous or unknown substances into the drains or sinks.

4.4 Any step that creates dust, such as sieving or grinding, must be performed in a fume hood.

5 APPARATUS AND MATERIALS

- #10, #20 or other sieves
- Stainless steel bowls and spoons
- Ceramic mortar and pestle, Automated or Manual
- Dish and Puck Mill
- Aluminum baking sheets
- Heavy duty aluminum foil
- Butcher's paper
- Flat metal spatulas
- Sieve cleaning brushes
- Lab grade acetone or methanol

6 PREPARATION FOR PROCESSING

6.1 CLIENT CONTACT

An ISM coordinator will be designated for each project. This person will be the main client contact at Apex for the duration of the ISM event, and will supervise and review all steps of the process that occur at Apex and any portions of the processing that are subcontracted.

The ISM coordinator MUST contact the client regarding appropriate sample handling procedures and fill out an ISM Request Form. This should be done significantly prior to samples being received at the laboratory to allow for modifications of the method or apparatus as necessary.

The client's Sampling and Analysis Plan (SAP), however named, and DQOs must also be received by the laboratory prior to sample processing.

The ISM coordinator will also generate a project specific ISM Worksheet (example, Appendix B) to use as a template for the ISM process. This spreadsheet will act as a guide for sample login by designating the appropriate log in procedure and will outline the steps required by the client's SAP.

Effective communication between the lab, the samplers, and the project team is essential to a successful ISM project.

6.2 SAMPLE RECEIPT

6.2.1 ISM samples will be received either in multitudes of individual soil jars (at least 30) or in multiple bags containing samples pulled from at least 30 sites. These containers will generally not be logged in to Element as being associated with the sample work order or sample number. The sample referenced by Apex for all analyses will be created by this procedure. Log samples in for the Incremental Sampling Methodology test code, and create empty sample jars with labels in accordance with the ISM worksheet.

6.2.2 Once the ISM procedure is complete, the jars will be returned to sample receiving and

requested analysis can be added to the appropriate samples.

6.3 BLANK SAMPLE

6.3.1 A Blank sample consisting of Ottawa Sand will be processed through most steps of the ISM procedure along with the samples IF metals analysis is requested. It will be analyzed for metals only unless otherwise specified by the ISM worksheet. All references to a sample in the following steps will also include the Blank sample.

6.3.1.1 Due to volume restrictions, some steps of the process are not applicable to the blank. Note any steps not performed on the ISM worksheet. 1-D Japanese Slab Cake Subsampling is performed by default, 2-D Slab Cake is not applicable for the small volume used for the blank.

6.3.2 The Blank sample should be logged in as the last two samples on each work order where ISM will be performed. The first of the two Blanks will be processed as a sample by ISM. It will be provided to Sample Receiving in a 1 gallon plastic bag. The second will be analyzed as is in order to provide a baseline for metals analysis, and will be provided in a 4 oz jar.

6.3.3 Log in jars for the first of the two Blank samples according to the following table:

Jar A	Plastic Bag	Blank	<2mm	NA	No analysis
Jar B	4 oz jar	Metals analysis	Requested final grain size	> 15 g	Requested metals

6.4 EQUIPMENT CLEANING

6.4.1 All equipment and work spaces must be cleaned before and after each sample is processed in order to minimize the potential for cross contamination. The fume hood used for sieving and grinding must have its work surface and inside walls washed with soap and warm water and rinsed with acetone initially and between preparation of each composite batch of samples. All equipment should be washed with warm water and soap before and in between each sample batch, followed by a rinse with acetone.

6.4.2 Trays used for air drying, subsampling, etc. may be lined with new aluminum foil or butcher's paper prior to use instead of the above cleaning procedure.

6.4.3 All references to cleaned equipment indicate that one of these procedures should be followed before use.

7 SAMPLE PROCESSING

In order to reduce potential sources of error, this procedure processes the entire sample received

at the laboratory through as many steps as possible. Unless otherwise specified, references to sample in this document refer to the total amount of sample received, or what is still defined as sample after prior processing steps. See the Quality Control section for a further discussion on sources of error and Data Quality Objectives (DQOs).

Each ISM sample will be different. The following steps are potential parts of any ISM processing, but may not be used for all samples. As such, the processing for each ISM sample will be driven by the SAP and the steps below should not be considered sequential requirements for all ISM projects. Refer to the SAP and the ISM worksheet for which steps are necessary for each sample. Steps not included in this SOP may be necessary. Details of these steps should be included in the ISM worksheet or other documentation.

7.1 SAMPLE IDENTIFICATION

ISM samples may include material that is not considered part of the analytical sample. Vegetation, oversized material, and decantable water are examples of material that may be requested to be removed before sample processing begins. The SAP should include detailed instructions as to what defines the analytical sample, and what to do with materials that are removed. This may include documenting their removal photographically, and potentially by weight.

7.2 PERCENT MOISTURE DETERMINATION

If as received percent moisture determination is requested on samples, it must be performed before samples are air dried. Samples will be homogenized as best as possible with field most samples, and a subsample aliquot taken as using the 2-D Japanese Slab Cake method. This may be done with or without wet sieving.

This result will be reported as the percent moisture. Dry weight analysis and correction will be performed on the prepared samples, but this result does not reflect the percent moisture of the sample as received.

7.3 SAMPLE SPLITTING/MASS REDUCTION

Splitting an ISM sample may be requested prior to other processing in order to have two separate sample processing paths for two different types of analysis, for sample mass reduction, or other reasons. This is not recommended due to potential increases in uncertainty of the data. Duplicate field samples are the preferred method for separate processing steps.

7.3.1 Three simple sample splitting techniques are available for use at Apex:

7.3.1.1 Alternate Shoveling divides the sample into two subsamples by placing alternate subsample scoops of the original sample into two separate sample containers.

7.3.1.2 Fractional Shoveling is similar to alternate shoveling except the sample is divided into three or more subsamples.

7.3.1.3 Cone and Quartering splits the sample into two subsamples by pouring the sample into a large cone, flattening the top and dividing into four sections. Opposite sections of the sample are then combined to form the two subsamples. This requires a flowable sample, and should be performed after samples are air dried and disaggregated. Therefore, this is only an option if both sample splits can be air dried.

7.4 SAMPLE CONDITIONING

Sample conditioning is usually necessary before homogenization or particle size reduction steps, in order to produce a flowable sample. Some sample conditioning steps may not be appropriate for some Chemicals of Concern (COCs), such as low boiling point SVOCs and Mercury. (See ITRC Table 6.1.) The SAP should address acceptable sample conditioning steps and how to process samples if conditioning is not acceptable.

Air drying at room temperature is the default sample conditioning step used by Apex if particle size reduction steps such as sieving are required. Other conditioning steps include drying at elevated temperature, freeze drying, and water addition. If these methods are requested, their procedure should be carefully specified in the SAP.

7.4.1 AIR DRY

7.4.1.1 Air dry the entire volume of all the sample containers by emptying them out on flat aluminum bakers sheets lined with heavy duty aluminum foil or butcher's paper and spread out to a depth of < 1 inch.

NOTE: Aluminum may not be an appropriate choice for samples where aluminum, chromium, or other compounds that may react with aluminum, are COCs. Paper or plastic maybe better choices in these cases. However, plastic must be avoided if phthalates or placsticizers are COCs, and paper cannot be used if organic carbon or other organics that may sorb to paper are COCs.

7.4.1.2 Place trays in bakery rack and allow to dry at ambient temperature in a low traffic area with sufficient air flow to carry away evaporated moisture, such as in or near a fume hood. 1-2 days are normally needed. Turning samples may be necessary to aid the drying process for wet samples, and layers of clay should be broken up in a mortar and pestle halfway through the drying process to avoid formation of bricks that are difficult to break apart after they are fully dried.

7.4.1.3 Samples should not be allowed to dry for more than three days, due to potential loss of more volatile analytes.

7.4.1.4 Record the air drying start and end times on the ISM worksheet.

7.4.1.5 After samples are dry, remove any visible sticks, rocks, vegetation, or other non-soil materials.

NOTE: If samples will be air dried, they do not need to be stored in the refrigerator. However, they most likely will be for ease of sample control. Ask the Sample Control department if questions arise about appropriate sample storage locations.

7.5 PARTICLE SIZE REDUCTION

For many projects, particle size reduction will be required in order to reduce the uncertainty associated with the data. Most samples will require that the particle size is less than 2 mm before analysis. This will ensure that a 10 to 30 gram aliquot will be enough sample volume to meet DQOs. For analyses that cannot use at least 10 grams of sample, (metals, cyanide, and other wet chem tests) grain size of less than 0.25mm must be achieved. Specific projects may require even finer grain sizes for these analyses.

If the ISM worksheet specifies that the sample will be processed to reduce particle size, there are many techniques that may be used. Automated mortar and pestle or dish and puck mill are two that are available to Apex. Depending on the COCs, these may not be appropriate, and SAP should specify which technique to use.

If a particle size reduction step is required, the entire sample should be ground so that it can pass through the sieve corresponding to the final grain size requested by the ISM worksheet. If multiple analyses are to be performed, this may require multiple samples to be taken in the field, or the sample to be split prior to processing.

7.5.1 SAMPLE SIEVING

7.5.1.1 Soil clumps should be broken up to allow them to pass through the sieve, and anything remaining in the sieve (stones, metal, glass) should be discarded and noted. Clay, wet, and/or rocky samples pose significant difficulties during this process. Breaking up dried clumps of dirt/clay and separating them from the material to be removed may be facilitated by grinding, pounding, tumbling or shaking samples by any available means. Record procedure used on ISM worksheet.

7.5.1.1.1 A sieve stack consisting of a lid, #4 and #10 sieves and a sieve pan may be loaded with sample and placed in to a sieve shaker for 5 to 10 minutes to breakup clumps without changing particle sizes.

7.5.1.1.2 A blender or coffee grinder may be used to disaggregate samples, but keep blending times low to reduce wear on blade, contamination of samples with blade material, and loss of analyte due to sample heating.

7.5.1.1.3 A mortar and pestle may be used, though this method can cause more particle size reduction than other methods.

7.5.2 MILLING/GRINDING

This step is often done on the sample that has passed through the #10 sieve. (Everything larger than 2mm is not defined as sample.)

7.5.2.1 Automated Mortar and Pestle:Using a cleaned mortar and pestle, grind the entire sample until it is fine enough to pass through the required sieve, as noted on the ISM

worksheet. See instrument manual or Apex operating procedure for details.

NOTE: This can also me done manually, which is a very laborious process and should only be done for small samples with few particles greater than the required size.

7.5.2.2 Dish and Puck Mill: This may be appropriate for some projects where metals are not COCs. See instrument manual or Apex operating procedure for details.

7.5.3 Enter details of the operation, operator initials and date on the ISM worksheet.

7.6 HOMOGENIZATION

The sample mixing step specified here assumes that the sample has been sieved so that all particles are less than 2mm. If this is not the case, simply stirring the sample will be more likely to increase sample homogeneity than decrease it, due to particle size separation within the bowl. Tumbling the sample in a container with sufficient headspace to allow free movement, or placing the entire sample into a blender or mill are better options in the case of un-sieved samples.

7.6.0.1 Place the entire sample (minus any portions removed during the Air drying and Sieving steps, if performed) into a stainless steel bowl. Stir the sieved sample well (approximately 3 minutes) to homogenize.

7.6.0.2 If it is necessary to complete the compositing procedure at a later time, place the entire homogenized sample into the 1 gallon re-closeable plastic bag labeled A for storage.

7.6.1 Enter operator initials and date on the ISM worksheet.

7.7 SUBSAMPLING

There are many methods available for subsampling, some of which produce less error than others. Apex has available two simple incremental sampling methods. If other methods are required, Apex will procure the appropriate technology or subcontract this portion of the process.

If subsampling for an analytical aliquot, pay close attention to the ISM worksheet. The aliquots taken must be very close to the mass requirements, because the entire aliquot subsampled must be used for analysis.

If specified by the ISM worksheet, repeat this process as needed to provide sample volume for process duplicate or triplicate analyses.

7.7.1 1-D JAPANESE SLAB CAKE

7.7.1.1 Pour the entire sample into a line, using 20 or more passes along the line to distribute the sample. For samples where small analytical masses are required (e.g. metals, cyanide) a long thin line should be created.

7.7.1.2 Using a square scoop, cut across the line to create an aliquot. Combine as many of these aliquots as needed to create the analytical sample or mass reduction required. Repeat until all analytical aliquots have been created.

7.7.1.3 Place the aliquots into their respective containers, according to the ISM worksheet.

7.7.1.4 Place the remainder of the sample into the 1 gallon re-closeable plastic bag labeled A for storage.

7.7.2 2-D JAPANESE SLAB CAKE

7.7.2.1 Pour the entire sample into a cleaned aluminum tray and spread evenly. Use a pre-formed grid with 30 sections to divide the sample. Pull an equally sized aliquot of sample from each section of the grid and combine into the appropriate container for analysis. Be sure to scrape along the bottom of the tray in order to include a representative portion of all grain sizes present in the sample.

7.7.2.2 Pull an aliquot of sample from each section of the grid to ensure that the final sample size is close to the mass requested for analyses, typically 10-30 grams. Place the aliquots into their respective containers, according to the ISM worksheet.

7.7.2.3 Place the remainder of the sample into the 1 gallon re-closeable plastic bag labeled A for storage.

7.7.3 When subsampling is complete, roll the jar(s) for 1 minute to homogenize the sample. Initial and date the ISM worksheet.

7.8 DOCUMENTATION:

7.8.1 Create a batch in Element for the ISM test code, add the samples processed as a batch, and print out the bench sheet. Set sample status to Needs Review, attach the completed ISM worksheet and submit for review and scanning.

7.8.2 Return jars to Sample Receiving for completion of log in.

7.9 LOG IN

7.9.1 After samples are returned from ISM processing, analysis test codes can be added to the samples.

7.9.2 Be sure to add comments indicating the use for each jar in accordance with the ISM worksheet. Because one jar will be created per analysis, duplicate, and MS/MSD, there will be a large number of containers for some samples. The container comments should match the ISM worksheet, and the work order should be reviewed carefully by the person coordinating the ISM project.

7.10 ANALYSIS

Each aliquot for analysis has been pulled during sample processing and placed into a separate container. Use the ISM worksheet and the analysis comments to find which container is designated for your analysis. Be sure to use the entire amount of the aliquot provided, and rinse the container into the extraction vessel. Check the sample comments for sample specific instructions (e.g. MS/MSD, etc).

8 QUALITY CONTROL

8.1 FUNDAMENTAL ERROR

The steps in this procedure are designed to ensure that the fundamental error (FE) associated with the sample is below 15% in the final aliquot used for extraction and analysis. This FE measure has been determined to be the primary lab DQO.

Fundamental Error is calculated using the following equation:

$FE = \text{Square Root}((20 * d^3)/m)$

Where:

20 = sampling constant
d = maximum sample grain size (cm)
m = sample mass used for extraction and analysis (g)

For samples taken from the - #10 sieve fraction, d = 0.2, m = 10 and FE = 12.6%
d = 0.2, m = 20 and FE = 8.9%

For samples taken from the milled fraction, d = 0.0850, m = 1 and FE = 11.1 or
d = 0.0250, m = 1 and FE = 1.8%

8.2 CONVENTIONS

8.2.1 Samples will be reported on a dry weight basis. The reported dry weight result will reflect the moisture left in the sample after air drying.

8.3 QUALITY CONTROL SAMPLES

8.3.1 Blank: A blank using Ottawa sand is processed and analyzed along with samples tested for metals to verify that no contamination is being added by processing the samples. This will be done as requested for other classes of COCs.

8.3.1.1 The Ottawa sand will have to be tested before and after processing to compare levels of metals present, as no known clean matrices for metals exist.

8.3.2 Process Replicates: Whether process replicates will be analyzed should be determined by the client on a project basis. They may request that one or two replicates be performed per

project, per batch, or per sample.

8.3.2.1 Aliquots may be pulled and designated to be analyzed as batch duplicates in the same manner as sample aliquots. This should be specified on the ISM worksheet, as a separate container will have to be created for them.

8.3.3 Matrix Spikes: Apex will not evaluate spike samples through the entire ISM process unless requested. If required to do so by a client, the client should specify or provide a standard reference material suitable for ISM processing.

9 REFERENCES

9.1 Hawai'i Department of Health *Technical Guidance Manual for the Implementation of the Hawai`i State Contingency Plan*, Section 4, November 12, 2008.

9.2 Alaska Department of Environmental Conservation Division of Spill Prevention and Response Contaminated Sites Program *Draft Guidance on Multi-Increment Soil Sampling,* March 2009.

9.3 EPA Method 8330B Appendix A Revision 2 October 2006.

9.4 Interstate Technology Regulatory Council *Technical and Regulatory Guidance: Incremental Sampling Methodology*, March 2011 (Draft)

Appendix A – Example ISM Request Form

Client:

Project:

Client Contact:

# of Decision Units:

# of Increments / Unit:

Notes:

Analysis: Note any that require subcontracting or small sample size (e.g. Metals)

Which ISM guidance document is being used for this project?

Alaska Hawaii EPA 8330A Appendix A ITRC Draft ISM Guidance

When will the Sampling and Analysis Plan be completed?
A copy must be provided to Apex before the project begins.

Project Specific Data Quality Objectives and procedures.
Apex follows the ITRC Draft guidance where possible. The following categories are procedural steps that are likely to have project specific goals. Our standard procedure is listed under the Apex heading, followed by specific requirements from the guidance documents. Each sampling event is unique, and modifications from our default procedure are expected. These differences should be noted.

Sample Storage:

Apex: Store refrigerated until air drying, room temperature thereafter.

Client request?

Air Drying:

Apex/ITRC: Air dry samples to help with sieving and grinding. Consider potential effects on volatile Contaminates of Concern (COCs) such as SVOCs and Mercury.

AK: Air dry only if necessary to sieve to < 2mm. May not be appropriate for Pesticides and PAHs.

HI: Air dry for all non-volatile analytes.

Client request?

Dry Weight:

Apex/ITRC: Samples are air dried, sieved, and then subsampled. That subsample is tested for most analysis and for dry weight. Results are reported on a dry weight basis, corrected to the air dried sample. If field percent moisture is requested, then a separate aliquot must be made prior to air drying.

HI: Air dried = dry weight, no further correction needed.

Client request?

Appendix A – Example ISM Request Form

Fundamental Error / Sample size:

Apex:	Our goal is to have less than 15% Fundamental Error at all steps. Our particle size and sample mass requirements are chosen to meet this goal for each analysis. We use at least 10 grams and generally ~20 for most tests, with a particle size smaller than 2 mm. We try to use at least 1 grams for Metals and other limited volume tests, with a particle size less than 250 µm.
AK:	Requires at least 30 grams of sample, particle size smaller than 2 mm.
HI:	At least 10 grams for most tests, particle size smaller than 2 mm. At least 1 gram for Metals and other limited volume tests, particle size less than 250 µm.
ITRC:	Somewhat contradictory. Generally, 10 grams for <2 mm fraction, 2 grams for < 0.25 mm.
Client request?	

Project Specific Fundamental Error (FE) goal?

Laboratory Replicate Samples:

Apex:	Per client SAP.
ITRC:	Field and lab triplicates are recommended for most projects.
Client request?	

Blank:

Apex/ITRC:	We have a blank sand matrix go through all steps of the analysis to ensure that metals are not added by the ISM process. Other analysis can be performed on the blank at additional cost. Matrix spikes are performed on a batch basis, per analysis.

Matrix Spikes:

ITRC:	Suggests that processing standard reference materials may be appropriate for some projects and COCs.

Notes:

Appendix B – ISM Worksheet

Batch

Sample Log in

Each sample created by the ISM procedure will be logged in with the containers and comments specified below. If samples will be treated differently, multiple sections will need to be created.

Sample IDs:

	Container	Use/Analysis	Particle size	Weight Needed	Comments
Jar A	Plastic Bag	Composite	<2mm	NA	No analysis
Jar B	4 oz jar				
Jar C					
Jar D					
Jar E					
Jar F					
Jar G					

Air Dry

Sample ID	Analyst	# of Containers to Composite	Air Dry Start Time	Air Dry End Time	Comments (Note sticks, rocks, etc removed.)

#10 Sieve

Sample ID	Date	Analyst	Homogenized?	Comments

Appendix B – ISM Worksheet

Splitting or Subsampling

This section may be needed multiple times for each sample. Modify worksheet to include this section for each step.

Method Used: 1-D Japanese Slabcake | 2-D Japanese Slabcake | Alternate Shoveling | Fractional Shoveling | Cone and Quarter | Other:

Sample ID	Date	Analyst	Replicates?*	Weight Obtained**	Homogenized?	Comments

*Indicate use for Replicates (Dry Weight, Duplicate analysis, etc)

**Total weight minus tare. (8 oz jar tare weight is 215g, 4 oz jar tare weight is 130g)

Grinding

This section may be needed for only a portion of each sample. Ensure that the proper container is noted.

Method Used: Automated Mortar and Pestle | Manual Mortar and Pestle | Dish and Puck Mill

Sample ID	Jar	Date	Analyst	Sieve size	Homogenized?

Sieve Size Chart	
#10	2 mm
#20	850 µm
#40	425 µm
#60	250 µm
#100	150 µm
#140	106 µm
#200	75 µm

Comments: