



Oregon

Kate Brown, Governor

Department of Environmental Quality

Northwest Region

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Portland, OR 97232

(503) 229-5263

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TTY 711

June 17, 2022

Lauren Mancuso
Union Pacific Railroad
1408 Middle Harbor Road
Oakland, CA 94607

Re: PCB Areawide – N Bradford St. ROW, Portland, Oregon
ECSI ID# 6480

Dear Lauren Mancuso:

The Oregon Department of Environmental Quality (DEQ) reviewed the May 6, 2022, revised *Investigation Work Plan (work plan)*, prepared by Jacobs on behalf of Union Pacific Railroad (UPRR). The work plan describes sampling along the UPRR right-of-way (ROW) along North Bradford Street in Portland, Oregon (ECSI #6480) to delineate the extent of polychlorinated biphenyls (PCBs) detected along the ROW and responds to DEQ's comments sent on April 14, 2022.

In the interest of conducting sampling in an expedient manner, DEQ is accepting the work plan and requesting that the additional requested information be submitted via email and documented in site investigation report.

DEQ accepts the work plan, with following comments.

1. ISM surface samples should be collected from the 0 to 2 inch interval to be most directly comparable to the ISM sampling conducted in Cathedral Park by DEQ in May 2022.
2. Aroclors 1262 and 1268 should be added to the standard Aroclors included in EPA 8082A. Please refer to this document for more information:
<https://www.epa.gov/risk/regional-issues-paper-pcb-characterization>
3. Given the complexity of the site and coordination with multiple adjacent sampling activities, please submit figures of the increment locations for each DU for DEQ review prior to field work. Since the ISM DUs represent long rectangles, please refer to Section 4.2.5.1 in Hawaii's DU guidance for locating increments in long, narrow DUs (<https://health.hawaii.gov/heer/tgm/section-04/#4.2.5>).

In addition, DEQ has the following comments and recommendations.

General Comments

4. The response to comments indicates that "discrete sampling locations will also be revised." These revised sampling locations should be included in the final Investigation Work Plan. Page 4 of the document notes 20 discrete boring locations while Figure 2 shows 16.

5. As discussed with Jacobs on the phone, please expand the ISM increment locations to 25 ft from the railroad center line.
6. The workplan proposes to collect discrete samples from soil borings in the 0.0 to 0.5 ft and 2.5 to 3.0 ft intervals. To better delineate vertical extent of contamination and address the data gap between 0.5 and 2.5 ft, DEQ recommends adding a sample in the 1.0 to 1.5 ft interval or collecting samples in the 0.0 to 0.5 ft, 1.0 to 1.5 ft, and 2.0 to 2.5 ft intervals.
7. DEQ continues to recommend that all samples, including discrete samples, be sieved in the lab to < 2 mm due to the presence of larger rocks in and around the railroad tracks. Sieving samples will also increase comparability between discrete and ISM samples by using the same defined soil fraction. DEQ notes that the different lab processing of ISM and discrete samples, in particular sieving means that concentrations will not be directly comparable and interpretation more difficult.
8. DEQ acknowledges UPRR is only going to sample for PCBs; however please be aware that DEQ may not be able to issue an NFA based solely on these contaminants. Please archive all collected samples to allow for potential future follow-up analyses.

Incremental Soil Sampling

DEQ appreciates the addition of the incremental soil sampling to the workplan. The following comments are based on DEQ's September 2020 Decision Unit (DU) Characterization internal management directive (<https://www.oregon.gov/deq/FilterDocs/DUIMD.pdf>) .

9. Please identify the site-specific methods that will be used from the Standard Operating Procedure in Attachment 2 (SOP) for both sample collection and lab processing.
10. Attachment 2 Incremental Sampling Methodology SOP.
 - a. DEQ recommends collecting 50 increments instead of 30, and notes that for the 0 to 2 inch interval, 50 samples also facilitates collecting an adequate sample mass of a minimum of 1000 g.
 - b. DEQ recommends a systematic random sampling grid.
 - c. DEQ recommends the use of cylindrical corers, augers, ISM tools, or drills and discourages trowels or shovels to ensure no sample bias in particle size.
 - d. The increments used for replicate samples should be placed far enough away from the primary sample (and each other) to be independent measures of the mean.
11. Pace Analytical, Multi-increment Laboratory Sampling SOP.
 - e. The containers mentioned include 4 or 8 oz containers. The total mass collected per DU should be at least 1000 grams – please ensure the laboratory can process this sample volume.
 - f. Please add a section detailing the site-specific steps that will be used to process the field samples in the laboratory. The attached SOP provides options and not site-specific details. Specifically, outline the steps including if the samples will be air dried, sieved, disaggregated, if grinding or milling will occur and any reduction in mass beforehand.
 - g. For reduction in mass, DEQ recommends using a sectoral splitter to collect the smaller representative sample (e.g. 100-200 grams) of the sieved sample (<2mm),

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Page 3

then grinding this smaller sample to powder (approx 60-70 μm), and taking aliquots from this sample. If grinding is not possible, DEQ recommends using a sectoral splitter used to get a smaller sample of the entire sieved sample (<2mm) and using a slab cake approach to get aliquot samples. The last recommended approach is using the 30 increments from a slab cake of entire sieved sample (<2mm) to get aliquot samples, but doing it this way will introduce more variability.

Please let DEQ know within two weeks of the planned sampling. Please contact me at (503) 229-5538 or franziska.landes@deq.oregon.gov if you have any questions.

Sincerely,

A handwritten signature in blue ink that reads "F. Landes".

Franziska Landes
Project Manager and Data Analyst
Northwest Region Cleanup Section

Ec: David Hodson, Jacobs
Katie Daugherty, DEQ
Mike Poulsen, DEQ
Kevin Parrett, DEQ
Todd Hudson, Oregon Health Authority

May 6, 2022

Franziska Landes
Oregon Department of Environmental Quality
Northwest Region
700 NE Multnomah Street, Suite 600
Portland, OR 97232

Subject: Revised Investigation Work Plan, PCB Areawide – N. Bradford Street Right-of-Way, Portland, Oregon (ECSI No. 6480)

Dear Franziska,

On behalf of Union Pacific Railroad (UPRR), Jacobs Engineering Group Inc. (Jacobs) prepared this revised work plan in response to the Oregon Department of Environmental Quality (ODEQ) October 11, 2021 letter outlining options related to polychlorinated biphenyls (PCBs) contamination in the N. Bradford Street right-of-way (ROW) in Portland, Oregon (site), (Figure 1). This revised work plan presents the methodology to conduct soil sampling within the UPRR ROW on N. Bradford Street to evaluate the current concentrations of PCBs in surface and shallow subsurface soil at the site. This revised work plan was modified in response to comments¹ from ODEQ on the original version of the work plan.² Responses to comments are included in Attachment 1.

Site Background

The site is located along the ROW of N. Bradford Street in north Portland, Oregon, and is bound to the north by N. Baltimore Avenue, and to the south by N. Philadelphia Avenue. The UPRR ROW, including active railroad tracks, is approximately 65 feet wide and located along N. Bradford Street, which separates Peninsula Iron Works, Inc. to the east from Cathedral Park to the west (Figure 2). Peninsula Iron Works has operated as a foundry and machining shop at this location for over 100 years.³

The City of Portland investigated PCB contamination at the site in 2011 as part of its source tracing activities for outfall basin 52. During the 2011 investigation, composite and discrete surface soil samples were collected along the N. Bradford Street/UPRR ROW and areas within Cathedral Park. Subsurface soil samples were collected from soil borings under pavement in a City of Portland-owned parking lot adjacent to the site (Figure 2). Total PCB aroclor concentrations in surface soil ranged from 147 micrograms per kilogram (µg/kg) to 21,700 µg/kg, and concentrations in soil borings ranged from non-detect to 1,050 µg/kg. PCBs were also detected in catch basin, manhole, and inline solids at concentrations ranging from

¹ Oregon Department of Environmental Quality. 2022. PCB Areawide – N Bradford St. ROW, Portland, Oregon, ECSI ID#6480. April 14.

² Jacobs. 2022. Investigation Work Plan, PCB Areawide – N Bradford Street Right-of-Way, Portland, Oregon (ECSI No. 6480). February 25.

³ City of Portland, Environmental Services. 2011. Request for DEQ Site Assessment of Peninsula Iron Works at 6618 N. Alta Avenue. December 13.

11 µg/kg to 8,160 µg/kg. Accumulated solids were subsequently removed from stormwater conveyance lines along the ROW between N. Baltimore Avenue and N. Alta Avenue.

On October 11, 2021, ODEQ issued a letter to UPRR outlining options related to PCB contamination in soil along the UPRR ROW in the N. Bradford Street ROW and identified UPRR as a potential responsible party for historical and/or ongoing releases of hazardous substances at the site⁴. Options included (1) to conduct a site investigation and remedial action under the ODEQ Voluntary Cleanup Program, or (2) wait for ODEQ to place UPRR under an enforcement order. In the letter, ODEQ assigned a high priority to the site for further investigation and remedial action.

A review of UPRR's available historical records for its operations at the site and in the Portland area in general indicate there was no use of PCBs that could have resulted in releases at the site. In addition, there are no records of derailments, spills, or leaks from UPRR trains in this area, and fueling, maintenance, and repair activities are not conducted at the site.

Although the City of Portland concluded⁵ that offsite migration of contaminants from Peninsula Iron Works operations may have resulted in observed contamination in the ROW and City property, UPRR proposes to conduct investigation activities under the ODEQ Voluntary Cleanup Program.

Investigation Objectives

The objective of this site investigation is to collect soil samples to evaluate the current distribution of PCB concentrations in surface and shallow subsurface soils along the N. Bradford Street ROW (Figure 1). The data may, in combination with previously collected data, be used to support an evaluation of the potential sources of PCBs observed at the site. This revised work plan describes field activities to be conducted at the site to achieve the investigation objective.

Field Methods and Procedures

This section discusses investigation activities and methods proposed to achieve the investigation.

Scope of Work

This site investigation will consist of the following components:

- Site visits will be conducted to observe site conditions and points of access, and to evaluate ground conditions at proposed sampling locations (presence of soil, gravel, and concrete/asphalt).
- Utility locates will be performed at each proposed soil boring location and across the greater site area.
- Incremental sampling methodology (ISM) will be conducted along the ROW from N. Bradford Avenue to N. Pittsburg Avenue (Figure 3). Approximately 30 increments will be collected from each of the eight sample units.

⁴ Oregon Department of Environmental Quality. 2011. PCB Areawide – N Bradford St. ROW, Portland, Oregon. ECSI ID# 6480. October 11.

⁵ City of Portland, Environmental Services. 2011. Request for DEQ Site Assessment of Peninsula Iron Works at 6618 N. Alta Avenue. December 13.

- Hand auger borings will be advanced to 3 feet below ground surface (bgs) at 16 locations along 2 transects (Figure 2). Sampling locations will be spaced on approximately 60-foot centers approximately 10 to 15 feet from the centerline of the tracks.
- Soil samples will be collected from 0.0 to 0.5 feet bgs and 2.5 to 3.0 feet bgs at each boring location. Samples will be analyzed for PCBs by U.S. Environmental Protection Agency (EPA) Method 8082 at Pace Analytical Laboratories on a normal turnaround time. Additionally, a portion of the samples will be analyzed by both EPA Method 1668 (PCB congeners), including at least two samples at the high, medium, and low range of detected PCBs.
- Boreholes will be backfilled with a bentonite chips.
- Investigation-derived waste (soil cuttings and decontamination fluid) will be containerized for staging and characterized for disposal.
- Sample locations will be captured using a tablet and handheld Global Positioning System unit.
- Photographs and lithologic descriptions will be recorded for each sample and boring location. Soil samples will be visually characterized for soil type, color, moisture content, texture, grain size and shape, sheen/odor, consistency, visible evidence of staining, and any other observation. Olfactory observations will be recorded on the soil boring log.
- Field personnel will use a project notebook to record pertinent information and to describe sampling procedures. A bound Rite-in-the-Rain field survey book will be used as a project notebook. Personnel will update the project notebook daily during field activities. Notes will include sketches of boring location, visual and olfactory characteristics of the soil sampled, time of sample collection, and other relevant information. In addition to the investigation data, the following site activity records will be recorded in the project notebooks:
 - Time and arrival and departure from the site
 - Project personnel onsite
 - Health and safety monitoring records
- Photographs will be taken throughout the investigation to document site activities, soil boring locations, and other pertinent information.

Field Preparation

Before advancing hand auger borings, Oregon One Call Utility Coordinating Service and UPRR fiber locating will be contacted a minimum of 48 hours before the start of work to locate public underground utilities and fiber optic cables, respectively. In addition, an independent utility verification survey will be performed to confirm the absence of underground utilities at each boring location.

No additional permits are required to advance borings along the ROW.

Incremental Sampling Methodology

The ISM method creates a well homogenized composite sample at each sample area, and reduces data variability, and provides a reasonably unbiased estimate of mean contaminant concentrations in a volume of soil targeted for sampling. Standard operating procedures for multi-increment sampling procedures will be used for guidance and are included in Attachment 2. The sample units (or sample areas) to be evaluated in this investigation are shown on Figure 3. Each area is approximately 100- feet long and 20- feet wide. A total of eight areas will be investigated. Thirty random incremental samples will be collected

from 0.0 to 0.5 feet bgs using a hand auger, trowel, or shovel from each sampling area (approximately 2,000 square feet coverage per sample). The samples will be sent to the analytical laboratory for additional processing and compositing (Attachment 2).

Coring and Discrete Soil Sampling

Soil sampling will be conducted as follows:

- Hand auger soil sampling locations will generally be spaced at 60-foot intervals along 2 parallel transects on the east and west sides of the UPRR tracks (Figure 2). Target sampling locations are planned to extend along the ROW from N. Baltimore Avenue to N. Pittsburgh Avenue, and be 10 to 15 feet away from the centerline of the UPRR tracks.
- The presence of asphalt or gravel on the ground surface will influence whether hand auger sampling can be accomplished at all proposed locations. Field crews may need to move some small material to access sampling locations. If asphalt is obstructing access to a sampling location, asphalt cutting methods may be used to access sub-asphalt soil.
- At each boring location, surface soil samples will be collected from 0.0 to 0.5 feet bgs, and subsurface soil samples will be collected from 2.5 to 3.0 feet bgs. A total of 20 surface and 20 subsurface soil samples, plus quality assurance (QA)/quality control (QC) samples will be collected.
- If hand auger refusal is met before 3 feet bgs at the target location, sample locations will be relocated. Up to 3 attempts at discrete sample locations within 10 feet of the proposed sampling location and between 10 and 15 feet away from the UPRR tracks will be made if hand auger meets refusal. In the event refusal is met before 3 feet bgs, but after the 0- to 0.5- foot interval has been collected, the shallower sample interval will be retained for analysis. Attempts will be made to collect appropriate material (that is, fine-grained, less than 2-millimeter diameter material) and avoid larger rocks.

Sample Analysis

Soil sample analysis will be conducted as follows:

- The QA/QC samples will consist of field duplicate samples and equipment blanks. Field duplicates will be collected at a rate of 10 percent (1 per every 10 primary samples). One equipment blank will be collected from each reusable piece of field equipment (for example, hand auger).
- Samples will be collected using a single-use stainless-steel spoon from the specified depth interval and placed into the appropriate containers provided by Pace Analytical Laboratories. After sample jars are filled, samples will be placed in an iced chest for storage.
- Sample containers will be properly labeled with the following information:
 - Sample ID – unique identification for each sample
 - Date sampled
 - Time sampled (24-hour clock)
 - Initials of sampler(s)
 - Preservative in the sample container, if any
 - Requested analysis
- Each field sample collected during the investigation will be assigned a unique sample ID. The sample ID will indicate the sampling location and type using the following components:
 - **Sampling matrix:** SB for soil borings; ISM for ISM locations

- **Sampling location:** 01, 02, 03, etc.
- **Sample depth:** 0.0-0.5, 2.5-3.0
- **Month year of sample:** 0422, (two-digit month and two-digit year)
- **Example sample ID:** SB01-0.0-0.5-0422
- For QA/QC samples, a unique identifier will be added after the sample location designation. The following designations will be used.
 - D = field duplicate sample
 - EB = equipment blank
- Soil and all associated QA/QC samples will be shipped to Pace Analytical Laboratory under chain-of-custody control. All samples will be analyzed for PCBs by EPA Method 8082, expressed as concentrations as aroclors (1016, 1221, 1232, 1242, 1248, 1254, and 1260). Laboratory chromatograms will be required from the laboratory. Additionally, samples will be archived to analyze a portion of the samples by EPA Method 1668 (PCB congeners), including at least two samples at the high, medium, and low range of detected PCBs.
- To minimize the potential for sample degradation and to maintain a temperature from 0 to 6 degrees Celsius, soil samples will be shipped in a chilled cooler with ice or gel packs in resealable plastic bags. The chain-of-custody form and a QA sample form will be filled out in indelible ink, placed in a resealable bag, and taped to the inside lid of the shipping cooler. Samples will be shipped to the following address:

**Pace National
Sample Receiving
12605 Lebanon Road
Mt. Juliet, TN 37122
615-758-5858**

Field Health and Safety Procedures

All field activities will be conducted according to a site-specific health and safety plan (HSP) (which includes an emergency response plan) that will be developed prior to the field event.

Decontamination Procedures

Decontamination of sampling equipment will be conducted consistently to minimize the potential for cross-contamination. At a minimum, a deionized water rinse will be performed between each sample location. If there is field evidence that indicates contamination is present, additional decontamination of sampling equipment will be performed using the following decontamination procedure:

- Physically remove visible debris, to the extent practicable
- Nonionic detergent wash
- Potable water rinse
- Triple rinse with distilled/deionized water
- Air dry

This decontamination procedure will be used between sampling locations. Disposable sampling equipment will be used as much as practical including nitrile gloves. Decontamination liquids and solids will be collected and staged in drums for disposal.

Waste Management

Waste generated during this investigation will consist primarily of decontamination water; PPE; disposable materials used for sample collection and processing; and soil, asphalt, and/or concrete from boreholes. Containerized solid wastes will be placed in appropriately sized and rated containers, characterized, and disposed of at an appropriate waste facility. PPE and disposable sampling materials will be placed into containers with soil, asphalt, and concrete for management and disposal.

The sole identified contaminant at this location is PCBs and analytical results from previous in-situ samples indicate PCB concentrations in soil are less than 50 milligrams per kilogram. Existing in situ analytical results indicate soil generated as a part of this investigation is classified as nonhazardous. A nonhazardous label will be used on waste containers. However, waste characterization samples will be collected and analyzed to make a final waste determination per ODEQ 340-101-0001.

Following completion of the investigation, waste containers will be placed in the waste staging area of the UPRR Albina railyard.

Solid and liquid wastes (decontamination water) will be containerized in drums at the waste staging area. PPE will be disposed of in drums with soil and disposable materials associated with nonhazardous wastes will be put into black trash bags and disposed of in a municipal waste bin onsite.

Waste sampling will be performed and managed in accordance with applicable local, state, and federal regulations. Representative waste samples will be submitted to a certified laboratory for analysis using U.S. EPA Method 8082 for PCBs.

The waste disposal subcontractor will provide services including, but not limited to, preparation of profiles and manifests and transportation and waste disposal.

Field Variances

Any deviation from this revised work plan will be documented in the field logbook with an explanation for the deviation.

Schedule and Reporting

Soil sampling is anticipated to be conducted within 60 days following the approval of this revised work plan. It is currently anticipated to be conducted within 1 month of approval of this revised work plan.

A site investigation report documenting the results of the soil sampling will be written following receipt of analytical results and will be provided to ODEQ. The report will, at a minimum, include the following components:

- A discussion of the field activities completed, including any modifications made to the revised work plan
- Soil analytical results

- Recommendations, if applicable
- Figures showing sampling locations and results
- Tables summarizing the analytical results
- Appendices, including analytical laboratory results, data validation reports, and field documents

If there are any questions or comments regarding the content of this revised work plan, please contact me at (510) 316-2323 or via email at david.hodson@jacobs.com.

Sincerely,
Jacobs Engineering Group Inc.



EXPIRES: 6/30/2023

David Hodson, P.E.
Project Manager

Enclosures:

Figures

- 1 Project Area
- 2 2011 Sample Results and Proposed Discrete Sample Locations
- 3 Proposed Incremental Sampling Methodology Locations

Attachments

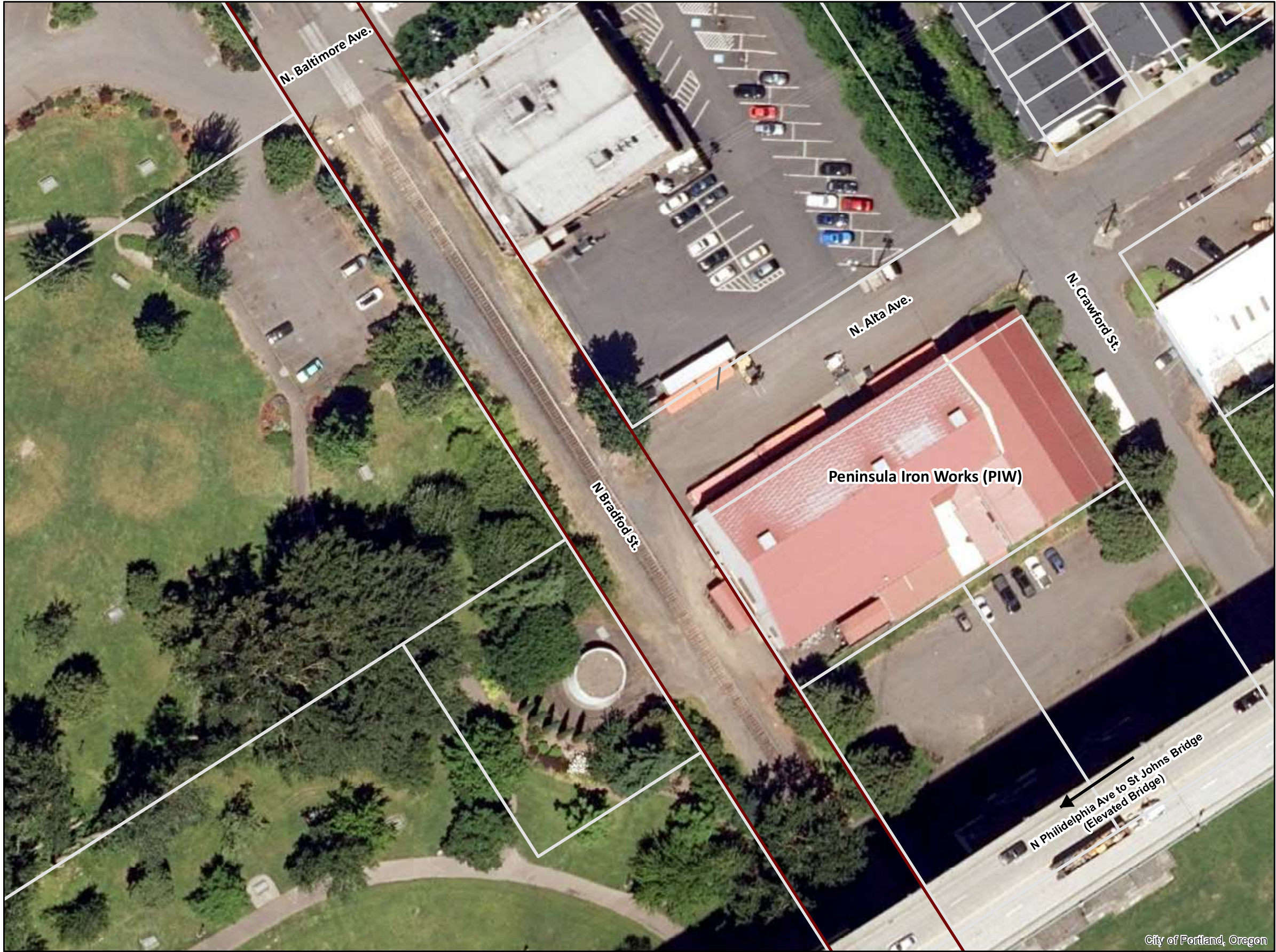
- 1 Reponse to Comments
- 2 Incremental Sampling Methodology Standard Operating Procedures

Copy to (with enclosure):

Electronic copy only:

Lauren Mancuso/UPRR

Figures



- Legend**
- Taxlot
 - Railroad Right-of-Way

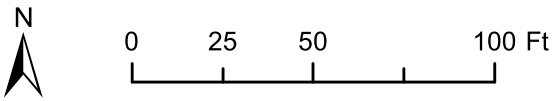
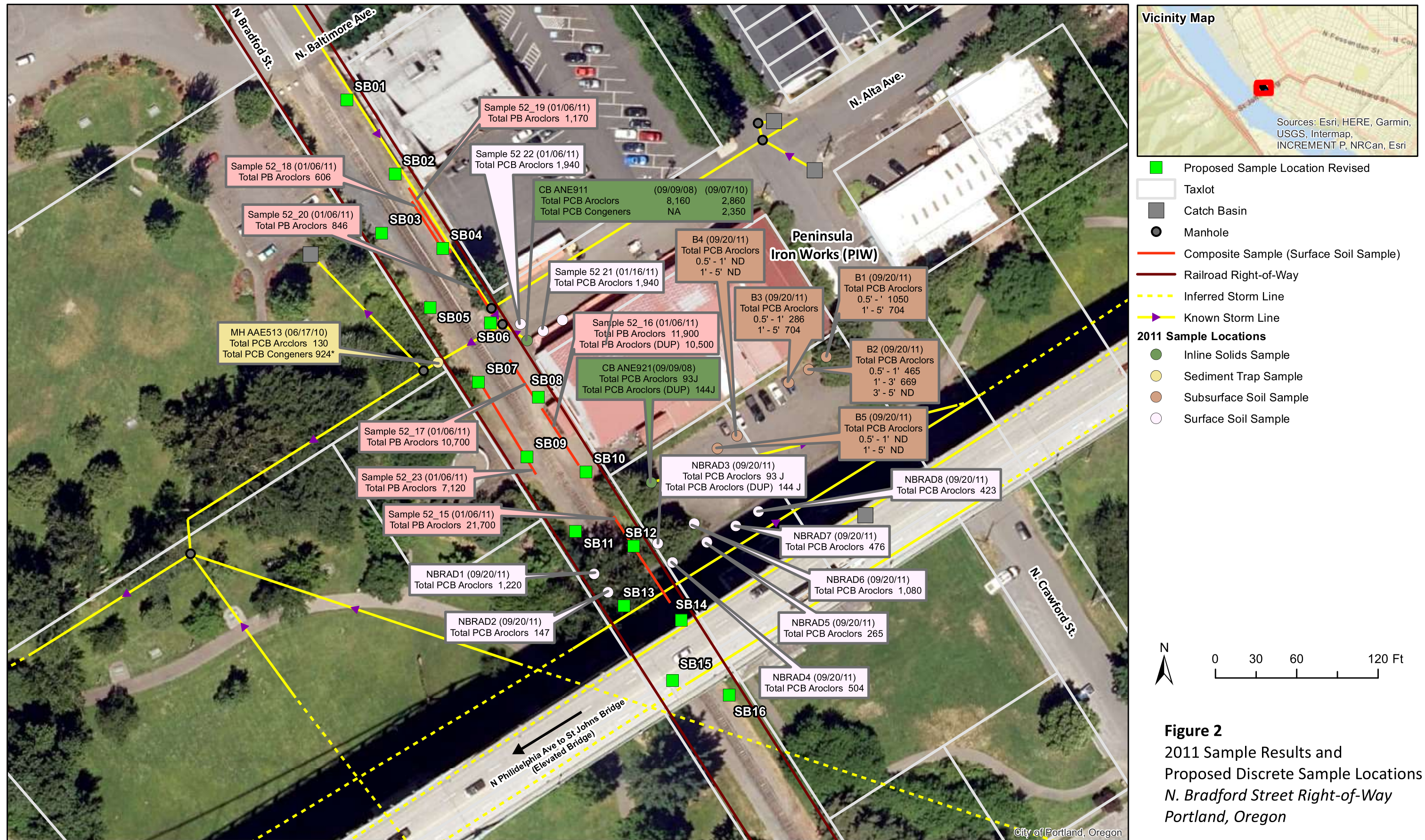
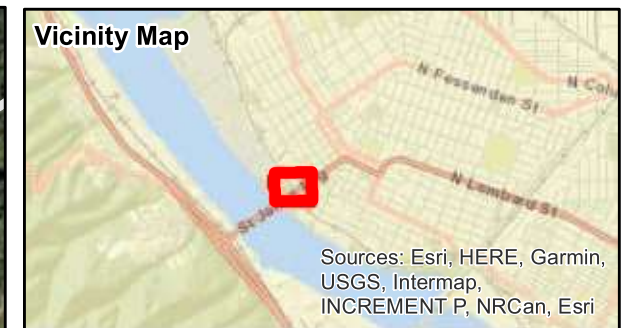
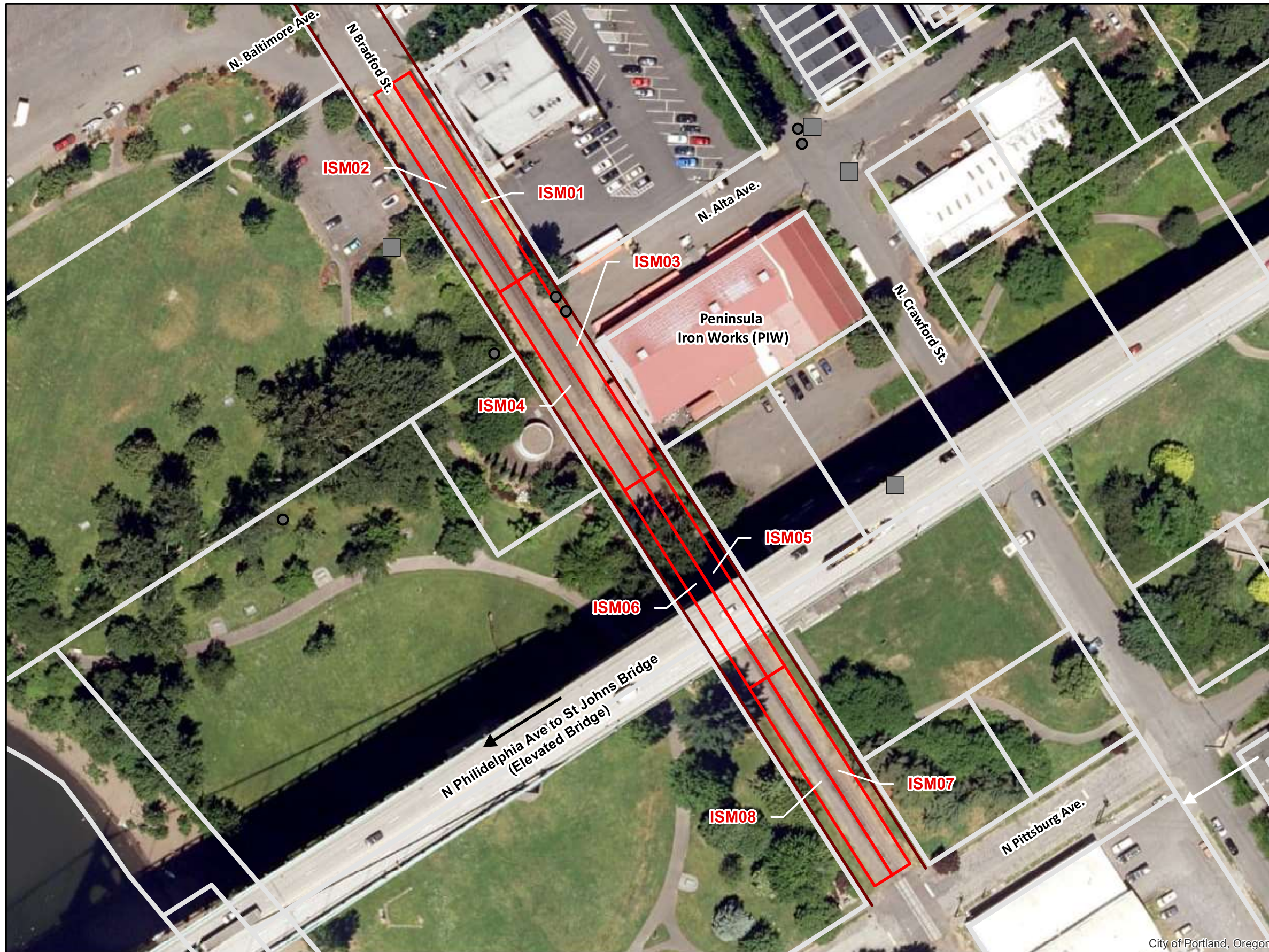


Figure 1
 Project Area
N. Bradford Street Right-of-Way
 Portland, Oregon

Jacobs





- Legend**
- Taxlot
 - Catch Basin
 - Manhole
 - Railroad Right-of-Way
 - Proposed Incremental Sampling Methodology Locations

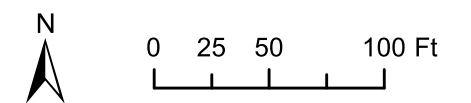


Figure 3
Proposed Incremental Sampling
Methodology Locations
N. Bradford Street Right-of-Way
Portland, Oregon

City of Portland, Oregon

Attachment 1
Response to Comments

Response to Comments Regarding the
Investigation Work Plan, PCB Areawide – N Bradford Street Right-of-Way, Portland, Oregon (ECSI No. 6480)

Dated April 14, 2022

from

Oregon Department of Environmental Quality

No.	Topic or Section	Comments	Responses
1.	Coring and Soil Sampling	Expand the sampling effort north along the ROW to N Baltimore Avenue, and south to N Pittsburg Avenue (see attachment 1). The expanded sampling area will further determine the extent of PCB contamination present in the railroad ROW and assist with determining the location(s) of potential source area(s) of PCBs.	Although there is no current information that PCBs will be detected in shallow soil beyond sampling locations from the City's work in 2010-2011, in response to this comment and comment 2, the work plan has been revised to propose incremental soil sampling methodology (ISM) for surface soil extending from N. Baltimore Avenue to N. Pittsburgh Avenue. Discrete sampling locations will also be revised.
2.	Coring and Soil Sampling	DEQ will be assessing risk to park users walking along the ROW. To assist with that evaluation, please collect surface soil samples using the incremental soil sampling methodology.	The work plan has been revised to include ISM sampling extending from N. Baltimore Avenue to N. Pittsburgh Avenue as indicated on Figure 3.
3.	Coring and Soil Sampling	Please clarify the procedure when hand auguring meets refusal before 3 ft bgs but after the 0 to 0.5 ft interval has been collected. DEQ recommends maintaining the shallow sample interval even if there is refusal at depth.	The work plan has been revised to state that up to 3 attempts at discrete sample locations within 5 feet of the proposed sampling location will be made if hand auger meets refusal. In the event refusal is met before 3 feet bgs but after the 0- to 0.5-foot interval has been collected, the shallower sample interval will be retained for analysis.
4.	Coring and Soil Sampling	Due to the presence of larger rocks in and around the railroad tracks, the workplan should describe how samples will be field screened prior to placement in sample containers. Regardless of the field screening approach, all samples should be sieved in the lab consistent with ISM methodology (<2 mm). Please attach the field sampling and laboratory processing and subsampling procedures.	The work plan has been revised to state that attempts will be made to collect fine-grained material and avoid larger rocks for discrete sample analysis. Additionally, ISM samples will be sieved in the lab consistent with ISM (<2 mm). A standard operating procedure for collecting and processing ISM samples has been included as an attachment to the work plan.
5.	Coring and Soil Sampling	Given the historical activities along the railroad, DEQ recommends expanding the analyte list to include total petroleum hydrocarbons (TPH), metals, and polycyclic aromatic hydrocarbons (PAHs), in addition to PCB Aroclors (EPA method 8082). Please archive all collected samples to allow for potential future follow-up analyses.	There is no evidence of impacted shallow soil caused by railroad activities involving TPH, metals, and PAHs along the railroad right-of-way (ROW). Therefore, analysis for TPH, metals, and PAHs is not warranted and is beyond what was previously discussed with DEQ. Furthermore, in the DEQ October 11, 2021 letter to UPRR, DEQ only required actions related to PCBs previously detected at the site.
6.	Coring and Soil Sampling	In order to determine if chemical weathering has impacted the accuracy of EPA method 8082, DEQ requests that laboratory chromatograms be provided for all samples and a portion of the samples be analyzed by both EPA method 8082 and EPA method 1668 (PCB congeners). This should include at least two samples at the high, medium, and low range of detected PCBs.	The work plan has been revised to state that laboratory chromatograms will be provided. Additionally, a portion of the samples will be analyzed by both EPA Method 8082 and EPA Method 1668 (PCB congeners), including at least two samples at the high, medium, and low range of detected PCBs.

Attachment 2
Incremental Sampling Methodology Standard
Operating Procedure

Incremental Sampling Method for Surface Soil

1 Purpose and scope

The purpose of this standard operating procedure (SOP) is to summarize requirements for the effective field implementation of increment sampling method (ISM) for soil undertaken as part of site characterization at contaminated sites. The ISM soil sampling process provides a view of mean contaminant concentrations over the area of a DU.

This SOP applies to the performance of ISM activities, and is limited to describing methods for obtaining surface soil samples (considered less than 1-foot below ground surface) for non-volatile, semi-volatile and inorganic analyses using ISM techniques. ISM techniques have been developed for volatile organic compounds and subsurface soil, however, they are not included in this SOP. This SOP was developed according to the following reference documents:

- *American Society for Testing and Materials (ASTM) D-6323-98. 2003 (re-approved). Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities.*
- *Hawaii State Department of Health (HDOH). Interim Final 2021. Technical Guidance Manual for the Implementation of the Hawaii State Contingency Plan. Office of Hazard Evaluation and Emergency Response. Sections 3.4 and 4.2.*
- *ITRC. 2020. Technical and Regulatory Guidance, Incremental Sampling Methodology. The Interstate Technology & Regulatory Council Incremental Sampling Methodology Team.*
- *Alaska Department of Environmental Conservation (ADEC). March 2009. Draft Guidance on Multi-Increment Soil Sampling. State of Alaska Department of Environmental Conservation Division of Spill Prevention and Response Contaminated Sites Program.*
- *Ramsey, C. and A. Hewitt (Ramsey, et. al.). 2005. A Methodology for Assessing Sample Representativeness, Environmental Forensics. 6:71-75.*
- *Pitard, Francis F. Pierre Gy's. Sampling Theory and Sampling Practice. 1993. 2nd edition. CRC Press.*
- *U.S. Environmental Protection Agency (USEPA). November 2003. Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples. R.W. Gerlach and J.M. Nocerino, EPA/600/R-03/027. http://www.cluin.org/download/char/epa_subsampling_guidance.pdf.*

This SOP focuses on the most commonly used ISM soil sampling tasks and applications anticipated at a field site and should be used in conjunction with other applicable project SOPs.

2 General

The objective of ISM is to reduce the variability created by taking discrete samples, and improve the reliability and representativeness of environmental data by obtaining multiple sub-samples (sample increments) over a decision unit (DU) (defined as the area or volume in question). These “increments” are combined into one bulk ISM sample, which is submitted to the laboratory, resulting in a better representation of actual mean concentrations in a DU.

The DU encompasses the area or volume about which a decision is necessary (e.g., deciding whether risks are acceptable or not). Appropriate decision units must be identified for ISM to be valid. Therefore, the identification of decision units is one of the most important factors when using ISM. Identification and delineation of the decision units should be conducted during project planning and identified in a client and regulatory approved

Workplan prior to obtaining ISM samples. Since ISM sampling provides an “average” concentration of a DU, agreement on the DU boundaries is extremely important prior to collecting the “bulk increment sample”.

The number of increments incorporated into the bulk ISM, and the overall size of the ISM collected are not dependent on the size of the decision unit. The sampling theory is based on an assumption (and empirical observations) that 30 to 100 increments from a given decision unit of any size will result in a sample that is adequately representative of the average contaminant level in the decision unit as a whole. If the decision unit is the size of a small backyard garden, then 30 to 100 increments are collected. If the decision unit is a 10-acre, neighborhood-size area in a former agricultural field, then 30 to 100 increments of a similar mass are likewise collected.

If the contaminant distribution is expected to be very heterogeneous, it may be preferable to increase the number of increments collected to the recommended maximum of 100 for larger DUs. This may help to reduce field sampling error and minimize the variation between replicate samples used to evaluate the precision of the data collected. It has been reported that increasing the number of increments from 30 up to 100 may improve the reproducibility of data collected, and since the ISM sample is submitted as one sample, the number of increments collected does not typically increase analytical costs except that a small fee may be added for the excess sample mass management in the laboratory.

This SOP describes procedures for selecting sampling locations, marking field sampling locations, collecting incremental soil samples, and submitting these samples for laboratory analyses. This SOP assumes that the DU, and method for selecting increment locations within the DU has already been determined in the project work plan or project Quality Assurance Project Plan (QAPP), and that analyses and the laboratory conducting the analyses have been identified in the QAPP.

3 Responsibilities

This section describes the responsibilities of key project personnel including the PM, SM, DM, HSM, FTL, and field sampler.

3.1 Project Manager

The PM provides adequate resources and engages field staff with adequate experience and training to successfully comply with and execute project-specific SOPs and implement the project HS&E program. The PM will solicit the appropriate technical expertise to adequately identify the best methods and technology for the job given the current understanding of the site and project goals. In addition, the PM should be consulted if complications arise in following sample handling and custody procedures.

3.2 Site Manager

The SM coordinates and schedules daily field activities. In addition, the SM trains field staff engaged in this activity and ensures compliance with this SOP.

3.3 Data Manager

The DM maintains and manages the sample tracking and scheduling program used to track field MI samples. The DM should consult the SM, FTL, and Project Chemist regarding MI soil sampling field sample processing.

3.4 Health and Safety Manager

The HSM is responsible for site-specific HS&E oversight and overall compliance with project HS&E requirements. The HSM conducts HS&E evaluations, selects the appropriate safety procedures for the project, lists the requirements in the project-specific HSP, and coordinates with the SM to complete and certify the HS&E program.

3.5 Field Team Leader

The FTL maintains compliance with MI sample techniques and methods, particularly the procedures to be used. The FTL, or their designee, should know the requirements of MI soil sampling and maintain adequate documentation of sample collection activities. The FTL should take responsibility for collecting MI samples

accurately and correctly and for coordinating with the SM and data manager to successfully conduct any MI field sample processing before laboratory analysis.

3.6 Field Sampler

The Field Sampler, under the supervision of the FTL (who may be the same person), should confirm that samples are correctly collected, labeled, tracked by chain of custody, and stored until they are delivered to the FTL or data manager. The Field Sampler should maintain custody of the samples until they are relinquished to the FTL or data manager. The Field Sampler informs the FTL and/or data manager of sampling conditions and potential deviations in sample collection.

3.7 Project Chemist

The project chemist is responsible for ensuring that the laboratory selected to analyze the ISM samples is qualified to do the work and meet the project data quality objectives (DQOs).

4 Procedures

ISM samples are prepared by typically collecting 30 to 50 small increments (samples) (up to 100 may be needed if a soil at the DU is determined to be very heterogeneous) of soil from systematic random locations within a specified decision unit and combining these increments into a single sample, referred to as the “bulk multi-increment sample.” Individual soil increments typically weigh between 30 and 50 grams, with bulk ISM typically weighing between 900 and 2,500 grams. The mass of the final bulk ISM depends on the number of increments collected, the size of the sample collection tool utilized. However a minimum final sample size should not be less than 1 kilogram as a general guideline.

4.1 Reconnaissance, Planning and Field Staging

During the DU planning process (Work Plan stage of project), a historical records search and site walk should be conducted to determine if there are areas that may have elevated levels of contamination as it may be desirable to break these “hotspots” into separate decision units. It is also useful for planning sample collection locations if access to some areas will be difficult. Logistics of sampling around buildings or sampling in areas of heavy vegetation should be considered when setting up the random grid (discussed in the following section).

4.2 Setting up a Stratified or Systematic Random Grid

A systematic random or stratified random sample collection scheme is developed from a random starting point in the DU. Typically a systematic random grid is the preferred sampling method. However, both strategies result in sample collection points spread out approximately equally across the DU. For example, a square-shaped decision unit could be divided into six rows and five columns with six increments collected from each of the five rows in a systematic, random fashion to obtain 30 increments for one bulk ISM.

The following are the definitions for these two sampling collection options:

1. **Stratified Random Sampling Mode:** DU into representative strata, sample at random within each strata, with number of samples proportional to relationship of each strata to the entire DU. This is used when there is high heterogeneity expected within the DU.
2. **Systematic Random Sampling:** DU into equal subunits, select starting location in first subunit, and sample all other subunits in the same location (grid sampling). This is the most reproducible sampling mode.

The project planning documents should generate a map, showing the DU(s) and approximate proposed sample locations (increments) within the DU(s). Random locations for incremental sample collection points can be pre-determined in the office using a random number generating program, or in the field. For more rectangular-shaped decision units, a fewer number of rows might be used with more increments per row collected. Row lengths and increments per row may be modified as needed for odd-shaped decision units.

4.3 Field Delineation of DU

Corners of the DU and some other strategic locations should be entered into a Global Positioning Device (GPS) in the office prior to going to the field. Using the GPS device, it is useful to mark the ends of each row with flags to help establish approximate lines for the collection of increments. Flags may also be placed along the edges of the decision unit parallel to the rows to help ensure approximate spacing. Placing flags at every increment collection point is usually not necessary. Often, just the four corners of the DU (or enough points to delineate the DU shape, if irregular) are located via GPS to document the DU location and to create maps for the soil investigation report.

Once the corners and rows of the DU have been marked using the GPS, the increment samples may be collected by pacing the same number of steps within each subunit or row of the DU where the sample increments are to be collected.

4.4 Tools for Collection of ISM

Using the wrong tools, or collecting a sample that contains more soil particles from the top of the sample than the bottom (or vice versa) could lead to biased sample results due to the heterogeneous distribution of contaminated particles in the soil. Care should be taken to collect increments in a manner that produces a cylindrical or core-shaped sample. This can be accomplished using a soil coring sampler (preferred), a trowel (if used to collect a “core-shaped” sample), or even a large drill in some soils. The most appropriate type of sampling device is dependent in part on the hardness of the soil, or how rocky it is. For soft soils, a soil core barrel that can be advanced by hand/foot is quick and efficient. Battery-operated drills with large bits may also be an option. For harder or rocky soils, a coring device with slide hammer, a mattock (large pick), hydraulic, or electric-assisted device, may be needed to advance the core barrel or access the soil column for sampling. Whatever tool(s) is used, the objective should focus on collecting core-shaped sample increments. It is important to understand field conditions and test proposed sampling tools at the site before selecting a particular type or combination of tools. If the site cannot be visited ahead of time, then a mix of sampling tools should be taken to help ensure that adequate soil samples can be collected in as efficient a manner as possible.

4.5 Sample Collection

Once the DU has been delineated with flags in the field collection of sample increments may begin.

Use flags or survey twine to define the edges of each grid cell and complete construction of the ISM sample grid as depicted in Figure 1 below.



Figure 1. Example Completed ISM Sample Grid

Sketch the ISM sample grid design, orientation (compass bearing), overall dimensions, cell dimensions, nearby features, and any other valuable information in the field notebook. Photograph the completed sample grid for future reference. Survey the center and corner stake locations of the DU or record them with a GPS unit.

If using stratified random sampling mode, a grid is set up over the DU making each part of the grid equal size, and one increment is collected at random from each subunit of the grid. If using systematic random sampling mode, select a random starting point in one subunit, then collect an increment sample at this location, and the same location at each subsequent subunit of the DU.

In either mode the following procedures should be followed:

- Sampling tools shall be new or decontaminated prior to use according to the project planning documents.
- Sampling tools need not be decontaminated between each sample increment, but shall be decontaminated or discarded prior to sampling a new DU.
- Test the proposed sampling tool(s), and determine what tool(s) will provide the best sample increments.
- Samples should be collected from the same depth at all incremental sampling locations.
- Larger sized particles (rocks, cobbles, and coral) and roots should be avoided or discarded prior to transferring the sample into the bulk ISM container.
- The laboratory is going to sieve out anything >2 millimeters (mm), so collect enough sample at each increment such that there will still be sample for analysis after the portion > 2mm has been sieved out. This may require collection of multiple aliquots per increment if MI samples are collected using a small diameter coring device.
- The sample collector will describe and classify soils collected according to Universal Soil Classification System (USCS) nomenclature. At a minimum, this will be done for the final bulk ISM sample after all the increments have been collected. Additionally, during collection of increments, the soil will be described at each significant change in lithology type encountered across the DU. Soil descriptions and classifications will be recorded in the field logbook.
- Individual increments collected are placed into a single sample container to produce the bulk ISM.
- If replicates and triplicates are being collected (strongly recommended), replicate increments may be collected from near the normal sample location by pacing off a few feet from the normal sample collection and obtaining a replicate increment. The triplicate increment may be collected by pacing another few feet from the duplicate increment sampling location (see Section 4.6).
- Store bulk ISM samples as required by the project planning documents.
- Pack and ship samples to the laboratory in accordance with the project planning documents.

4.6 Collection of Field Replicate MI Samples

To statistically evaluate sampling precision for each DU, replicate ISM samples are collected from selected decision units. Typically two replicate increments are collected from the same depth as the normal sample in different locations. A different random starting location is determined for each replicate collected in the selected DU(s). Replicate sample increments are generally collected along the same approximate directional lines established through the DU for the initial ISM samples, though at different systematic random locations than initially used. This is accomplished by pacing off the replicate increments from a different random starting location on the first line/row of the DU, and continuing to sample at this different random interval throughout the DU.

Replicate samples may be collected by establishing rows for increment collection that run perpendicular to or at a 45 degree angle to the direction used to collect the initial ISM. Another option is to use the same rows but collect increments in between the locations used for the initial sample. Replicate samples should be sent to the laboratory as “blind” samples, meaning the laboratory does not know they represent replicate samples of the initial ISM.

The replicate samples are prepared and analyzed in the same manner as carried out for the initial sample. Triplicate samples (i.e., initial ISM plus two replicates) are preferred and more useful than just duplicates for

statistical analysis. If only one DU is being investigated, triplicate samples are recommended. If multiple DUs are being investigated, it may not be necessary to collect triplicates at all DUs.

4.7 Laboratory Processing of ISM Samples

The bulk ISM is submitted to the laboratory for analysis. Careful planning with the laboratory for processing of ISM samples by the Project Chemist prior to sample collection is essential to obtain meaningful results. Details of project requirements will be described in the project planning documents.

It is important to note that, while the laboratory is receiving a bulk sample of up to 2,500g, it will only analyze a subset of this sample. One issue discussed in both the Environmental Protection Agency (EPA) and American Society of Testing Materials (ASTM) guidance documents is the choice of a minimum sub-sample mass for extraction/analysis of soil samples in order to reduce “Fundamental Error” of the lab analyses to approximately 15% or less. The minimum appropriate mass is based on the maximum particle size in the soil samples. For samples with a maximum particle size of <2mm, the minimum analysis mass is 10 grams. If the analytical method to be used typically calls for sample extraction/analysis mass of less than 10 grams, the method should be modified to increase extraction/analysis mass to at least 10 grams for samples with maximum particle sizes of <2mm (larger mass could be beneficial for some analyses). For analyses of fine particulates (e.g., <250 µm), a one-gram sub-sample may be adequate to reduce Fundamental Error below 15%; however a larger mass may be reliably run by the method (e.g., 2-10 grams).

4.8 Investigation Derived Waste

Any IDW generated during sample collection (such as used PPE and soil collection apparatus) should be disposed of properly in accordance with the project planning documents.

5 Records

Record all ISM soil sampling activities, including field bulk sample collection in a field notebook, in accordance with the Work Plan and applicable project SOPs. Chain-of-custody forms, photographs, and any other sampling documentation should comply with the project planning documents.

6 References

American Society for Testing and Materials (ASTM) D-6323-98. 2003 (re-approved). Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities.

Alaska Department of Environmental Conservation (ADEC). March 2009. Draft Guidance on Multi-Increment Soil Sampling. State of Alaska Department of Environmental Conservation Division of Spill Prevention and Response Contaminated Sites Program.

Hawaii State Department of Health (HDOH). 2020. Technical Guidance Manual for the Implementation of the Hawaii State Contingency Plan. Office of Hazard Evaluation and Emergency Response. Sections 3.4 and 4.2.

HDOH. May 11, 2007. Pesticides in Former Agricultural Lands and Related Areas – Updates on Investigation and Assessment (arsenic, technical chlordane, and dioxin test methodologies and action levels and field sampling strategies). Office of Hazard Evaluation and Emergency Response. 07-241 RB.

ITRC. February 2012. Technical and Regulatory Guidance, Incremental Sampling Methodology. The Interstate Technology & Regulatory Council Incremental Sampling Methodology Team.

Ramsey, C. and A. Hewitt (Ramsey, et. al.). 2005. A Methodology for Assessing Sample Representativeness, Environmental Forensics. 6:71-75.

Pitard, Francis F. Pierre Gy's. Sampling Theory and Sampling Practice. 1993. 2nd edition. CRC Press.

U.S. Environmental Protection Agency (USEPA). November 2003. Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples. R.W. Gerlach and J.M. Nocerino, EPA/600/R-03/027. http://www.cluin.org/download/char/epa_subsampling_guidance.pdf.

7 Definitions

Bulk MI Sample: The compilation of all MI soil sampling increments collected from a DU.

Compositional Heterogeneity: The variability of contaminant concentrations between the particles that make up the population. This type of heterogeneity results in fundamental error (FE).

Decision Unit (DU): The area or volume in which a decision must be made (for example, deciding whether risks are acceptable or not). The DU may be as small as a 55-gallon drum or as large as acres in size.

Distributional Heterogeneity: The non-random distribution across a population due to slight spatial variations. This type of heterogeneity results in grouping and segregation error (GSE).

Fundamental Error (FE): A result of not representing proportional concentrations of all of the particles in a population.

Increment: A group of particles collected from a population with a single operation of the sampling device.

Sieving: Pouring material (for example, the bulk MI sample) through a sieve.

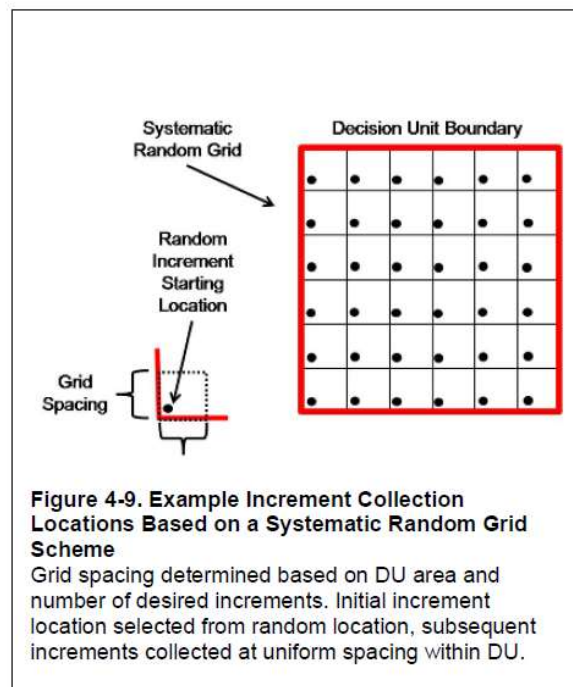
Stratified Random Sampling: A statistical sampling method that divides the sample population (DU) into representative strata (grid cells), then randomly sampling within each stratum with the number of samples proportional to relationship of each stratum to the entire population.

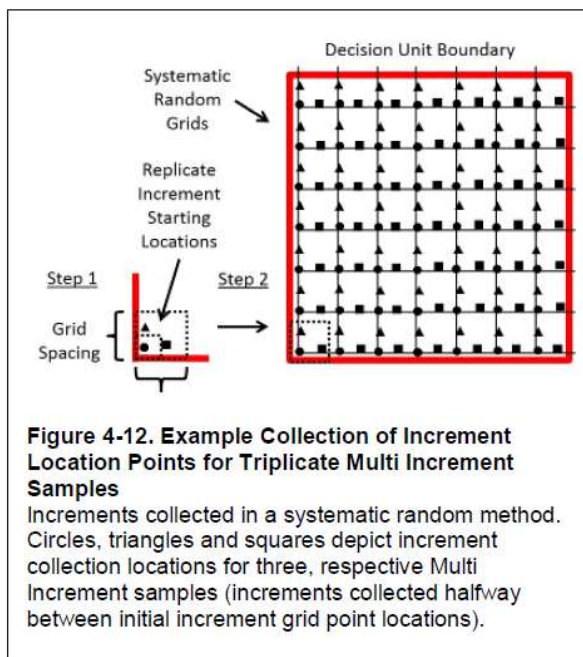
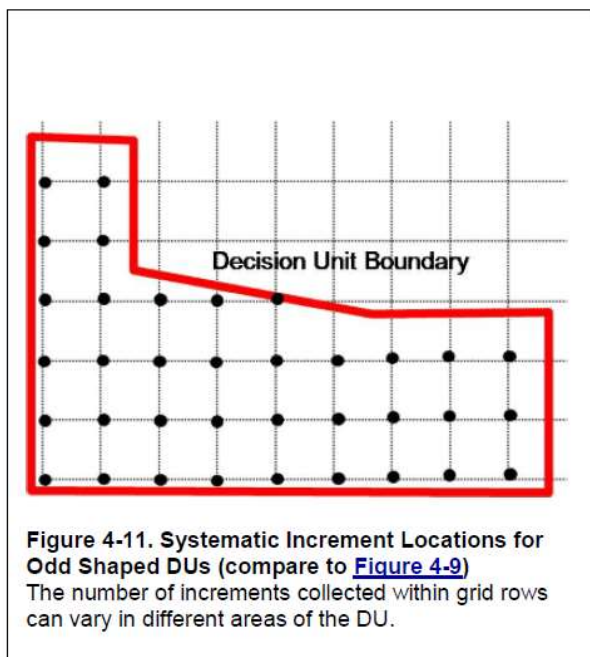
Systematic Random Sampling: divide population (DU) into equal subunits, select starting location in first subunit, and sample all other subunits in the same location (grid sampling). This is the most reproducible sampling mode.

Sub-sampling: Dividing the sieved bulk MI sample to create a final laboratory sample.

Attachment 1 - Figures Illustrating Systematic Random Sampling Method

(Source: HDOH *Technical Guidance Manual for the Implementation of the Hawaii State Contingency Plan*, 2016 and updates)







Document Information

Document Number: ENV-SOP-GBAY-0134	Revision: 02
Document Title: Multiincrement Soil Sampling	
Department(s): Other	

Date Information

Effective Date: 15 Oct 2020

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest**Document Number:** ENV-SOP-GBAY-0134**Revision:** 02**Title:** Multiincrement Soil Sampling

All dates and times are in Central Time Zone.

ENV-SOP-GBAY-0134-Rev.02 Multiincrement Soil Sampling**QM Approval**

Name/Signature	Title	Date	Meaning/Reason
Kate Verbeten (007119)	Manager - Quality	14 Oct 2020, 04:27:27 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Christopher Haase (007121)	Manager	15 Oct 2020, 09:32:16 AM	Approved
Nils Melberg (007142)	General Manager 2	15 Oct 2020, 10:08:36 AM	Approved



TEST METHOD STANDARD OPERATING PROCEDURE

TITLE: Multi-Increment Soil Sampling

TEST METHOD Pace MIS

ISSUER: Pace ENV – Green Bay Quality – GBAY

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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for performing multi increment soil sampling using the guidance given in documents: ITRC Incremental Sampling Methodology; State of Alaska Draft Guidance on Multi Increment Soil Sampling; State of Hawaii Multi Increment Sample Collection, and is applicable to soil to be analyzed for metals and/or SVOC type methods (8270, PCBs etc.). Additionally, this method may be used for volatiles sub sampling when following the appropriate procedure as outlined in the SOP.

1.1 Applicable Matrices: This SOP is applicable to soil only.

1.2 Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

2.0 SUMMARY OF METHOD

The soil is sieved, or air dried and sieved through a #10 sieve. The soil is spread on a large tray. A determined number of sample aliquots are taken from designated areas on the tray and combined to form the sample to be tested. Samples preserved in the field for Volatile analysis will be composited by taking equal portions of the methanol and combining in the laboratory or compositing all methanol preserved field samples into an adequately sized container(s) prior to taking a sample aliquot(s) for analysis. The number of aliquots taken is determined by the client or project requirements.

3.0 INTERFERENCES

Metallic Devices – Samples to be analyzed for metal constituents should not be sampled using any metallic mixing devices or containers as it may result in contamination of the sample with a variety of metals. Use only glass, plastic or ceramic materials when working with these sample types.

Plastic Devices – Samples to be analyzed for organic constituents should not be sampled using any plastic mixing devices or containers as it may result in both positive and negative interferences. Use only glass and ceramic devices when working with these sample types. Metal instruments may also be used if analysis for metals is not required from the same sample.

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of the analytical results. All of these materials must be demonstrated free from interferences under the conditions of the analysis by performing method blanks.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

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5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handling these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Soil samples that are collected in regulated domestic areas or that are of foreign origin must be handled in accordance with the Pace SOP: ENV-SOP-GBAY-0121, *Regulated Soil Handling* (current revision or replacement).

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory SOP ENV-SOP-GBAY-0007 *Bottle Preparation* (most recent version or replacement).

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General Requirements:

Matrix	Routine Container	Minimum sample amount ¹	Preservation	Holding time
Soil/Solids	4 or 8oz Amber Glass or plastic (Dependent on analysis method required))	Dependent on number of sampling sites and analysis method required.	Thermal: ≤ 6°C Chemical: None	Metals Analysis: 6 months; excluding Mercury 28 Days if Mercury requested. Pesticide/BNA or PAH Analysis: 14 Days PCB Analysis: 365 Days.
MeOH soil for VOA	VOA MeOH preserved vial	Dependent on number of sampling sites and analysis method required.	Thermal: ≤ 6°C Chemical: MeOH	14 days

¹ Minimum amount needed for each discrete analysis.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0006 *Sample Management* (most recent revision or replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation. Shipments of soil samples to the laboratory require thermal preservation in the form of cubed or block ice. At the time of laboratory receipt, proper thermal preservation is checked by measuring the temperature of melt water or when provided the temperature blank. The Pace Analytical acceptable temperature range is 0 to 6°C. All QAPjP and regulatory authority requirements become priority over this requirement.

After receipt, samples are stored at ≤6°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at the temperature designated in the analytical method.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES**7.1 Equipment**

Equipment	Vendor*	Model/Version*	Laboratory Identification	Description/Comments
Analytical Balance	A&D	GH200	40BALL	Electronic with RS-232 output, capable of weighing 0.1g
Drying Rooms	In-house	Custom Made	40OVNK	Equipped with exhaust or blower fans for air circulation
Sieve Shakers	Various	Various	NA	To be used for disaggregation.
Computer for Electronic Prep Log	NA	NA	NA	Automated sample weight upload into LIMS

* Or Equivalent

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7.2 Supplies

Supply*	Manufacturer*	Vendor*	Catalog#*	Description
Drying trays	NA	Local Vendor	NA	Restaurant Grade
#10 sieves	Gilson	Gilson	V8SF#10	Stainless steel or brass
Catch pans	Gilson	Gilson	V8SFXPN	
Separator pans	Gilson	Gilson	V8SHXPE	
Lids	Gilson	Gilson	V8SFXCV	
Blunt end scoop	Fisher	Fisher or Local Vendor	14-241-202	2 oz Bent Handle Scoop or; Adjustable scoop used for baking
Mallet	NA	Local Vendor	NA	Rubber or Hard Plastic
Rolling pin	NA	Local Vendor	NA	Marble
Zip top plastic bags	NA	Local Vendor	Various sizes	NA
Sampling Template	In-house	In-house	Custom Made	Lab made template
Paper towels	NA	Local Vendor	NA	NA
Gastight Syringe	Hamilton	Fisher	50 µL – 1mL	Gastight for MeOH samples

*Or Equivalent

8.0 REAGENTS AND STANDARDS

Reagent	Concentration/Description	Requirements/Vendor/Item#	Expiration Date
Deionized water	Type II ASTM	US Filter 18Ω	NA
Methanol	Pesticide Grade	Burdick & Jackson / Cat: PP230-19	Manufacturer's recommended expiration date or 2 years from receipt, whichever is sooner.
Alconox	Cleaning Solution	Fisher / 50-212-165	NA

*Or Equivalent

9.0 PROCEDURE
9.1 Calibration
9.1.1 Analytical Balance Calibration.

9.1.1.1 Annual Calibration - The balance must be calibrated at least annually by an outside agency and checked daily before each use using Class 1 or 2 weights. Refer to Pace ENV-SOP-GBAY-0115, *Support Equipment* (current revision or replacement).

9.1.2 Daily Calibration Check.

9.1.2.1 Clean the balance and surrounding area prior to starting the daily calibration check.

9.1.2.2 Check the sight level on the balance. If it needs adjusting, level the balance.

9.1.2.3 The weight set ID indicated in the logbook is used as the primary set. If an alternate weight set ID is used, that ID must be recorded in the comment section of the balance calibration logbook for that day.

9.1.2.4 Tare the balance before weighing the NIST certified weights.

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9.1.2.5 Use forceps or other means to lift each weight (Do not touch the weights with fingertips as the residue may artificially adjust the true value of the weights). Record the date of the calibration check, the true value of the weight, and the actual measured weight in the logbook. Repeat this procedure for the other certified weights. If calibration weights differ from the certified weights by more than specified in the balance calibration logbook, corrective action must be taken (see 12.2).

9.2 Procedure for Metals and SVOC Samples:

- 9.2.1 The project Quality and Assurance Plan (QAPP) or Sampling and Analysis Plan (SAP) should be consulted to determine if the sample should be dried prior to sieving. The entire sample submitted must be sieved or dried and sieved. Subsampling/splitting prior to sieving to reduce sample volume is not allowed unless addressed in the QAPP or SAP. If drying is not required go to 9.1.5.
- 9.2.2 If the client requires an equipment blank to be processed with the samples, the same equipment which is used to process the samples must be Deionized Water (DI) rinsed prior to sample processing. One equipment blank per day may be processed and must be completed with sufficient volume for all tests to be performed. The equipment blank must be logged into the LIMs system to report with the sample data.
- 9.2.3 If the sample is to be dried, spread the entire sample volume out in the drying tray. Break up any clumps of soils to about ¼" to ½" diameter. This will speed the drying process and ease the disaggregation process prior to sieving. Change gloves between samples. Place in the drying room (≤100°F) overnight or longer until dry. Samples should be dried to moisture content of 15% or less for soils and 30% or less for sediments (may be determined by QAPP or client requirements).
- 9.2.4 Once the samples have been dried, they should be removed from the drying room and the process of disaggregation should begin. Disaggregation is the process of loosening clumped soil. It is not meant to crush or reduce the natural particle size of the soil. If necessary, place the dried sample in zip top like plastic bag and break down the sample by rolling a rolling pin over the dried soil for 1 to 2 minutes. Alternatively, a mallet can be use also to break up the soil. Change gloves between samples. Dispose of the sample drying tray and rinse equipment used in the disaggregation after each use with methanol.
- 9.2.5 Record the lab sample number on the catch pan. Then pour the entire sample onto the #10 sieve. If there is too much sample to fit on one sieve, the lab will pour the sample through the sieve and manually shake multiple times. Alternatively, if the sample does not pass through the sieve easily, stack up to three sets of #10 sieves and catch pans, cover with a lid and place on the sieve shaker. Tighten the adjustments so that the sieves fit tightly and securely. Set the time for 10 minutes and begin the sieve shaking.
- 9.2.6 After the sample passes through the sieve, carefully separate the sieve from the catch pan. Pour the contents of sieved material onto a tray. If more than one set of sieves was used, pour all the catch pan contents onto the same tray. Pour the remaining contents retained on the #10 sieve into a zip top style bag. Label the bag with the lab number and the comment ">10 coarse fragments". Wash and dry the catch pan and sieve, then rinse with methanol, between each sample.

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TEST METHOD Pace MIS

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- 9.2.7 Spread the sieved material evenly on a tray to a depth of ½" to 1". Place the sampling template or determine the measuring grid with the correct number of sample aliquots portioned out on top of the tray and gently make an indentation into the soil sample. This will mark the spot where each sample aliquot will be sampled. If using the template, remove it.
- 9.2.8 Before beginning the sampling process, the total mass needed for the requested analysis must be determined. Obtain guidance from the lab manager or project manager. For instance, if 30 grams total is desired then obtain 30 1-gram portions. If 90 grams required, obtain 30 3-gram portions, etc. Before beginning, sample a few trial scoops to calibrate the amount needed for each scoop. Take the trial scoops from areas not designated as sample areas based on the template indentions.
- 9.2.8.1 If a volume required for each increment is less than 0.1g, the laboratory will sub-sample a larger volume than necessary for the individual parameter and will subsample the necessary amount for the analysis.
- 9.2.9 Using a blunt end scoop (or adjustable scoop) transfer a portion from each of the designated areas to a sample jar. Be sure to sample the entire depth of the soil. Transfer the remaining soil from the sampling tray into a zip top style bag and store at the designated method temperature ($\leq 6^{\circ}\text{C}$).
- 9.2.10 If more sample mass is required than can be obtained from one round of incremental sampling, re-smooth the sample on the tray and repeat the entire sampling process until a sufficient volume of sample is achieved. Wash and dry the trays then rinse with methanol between samples.
- 9.2.11 The QAPP or SAP may call for further particle size reduction prior to metals analysis being performed. If this is required, the sample may be sent to a sub-lab for pulverization.

9.3 Procedure for Methanol preserved soils:

- 9.3.1 The project Quality and Assurance Plan (QAPP) or Sampling and Analysis Plan (SAP) should be consulted to determine the number of samples collected to determine the number of aliquots required.
- 9.3.2 Equal portions of methanol will be removed from each container. If the samples do not have a 1:1 soil:methanol ratio, multiple containers can be combined to have a total weight that will exceed the volume of methanol in the container to achieve a 1:1 soil:methanol ratio.

10.0 DATA ANALYSIS AND CALCULATIONS

See Section 9.1.8 on the determination of total mass required for analysis.



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11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control: A client requested equipment blank may be created for a sample batch. See Section 9.2.2.

11.2 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. Refer to laboratory SOP ENV-SOP-GBAY-0094 *Orientation and Training Procedures* (most recent revision or replacement) for more information.

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employees complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Draw a single-line strikethrough for any unacceptable or changed data, then DATE and INITIAL and provide a written explanation of the reason for the change

Refer to laboratory SOP ENV-SOP-GBAY-0120 *Data Review and Final Report Processes* (most recent revision or replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

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Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

12.2.1 Balance Corrective Action:

12.2.1.1 Clean the balance and balance pan. Check the sight level on the balance and adjust if necessary. Re-tare and reweigh all the certified weights.

12.2.1.2 The internal calibration function (if available) of the balance may be used as a means of corrective action.

12.2.1.3 Utilize the internal calibration function and diagnostics. Refer to instrument manual.

12.2.1.4 Utilize the internal calibration function and diagnostics. Refer to instrument manual.

12.2.1.5 If the above action does not correct the problem, the balance should be taken out of service and appropriately labeled to avoid improper usage. A service technician will be contacted by the Supervisor or Quality Assurance Department

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

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15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Not applicable to this SOP.

17.0 REFERENCES

17.1 Pace Analytical Services, LLC – Green Bay, WI Quality Assurance Manual- current version.

17.2 Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-VI-2016-Rev.2.1.

17.3 The Interstate Technology and Regulatory Council (ITRC) "Technical and Regulatory Guidance, Incremental Sampling Methodology" Feb. 2012

17.4 State of Alaska Department of Environmental Conservation "Draft Guidance on Multi Increment Soil Sampling" March 2009

17.5 State of Hawaii "Soil Sample Collection Approaches" Section 4.2 "Multi-Increment Sample Collection"

18.0 REVISION HISTORY

This Version: ENV-SOP-GBAY-0134-Rev.02

Section	Description of Change
All	Transferred to new format

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-GBAY-0134	Multi-Increment Soil Sampling	Rev.01

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Document Information

Document Number: ENV-SOP-GBAY-0164	Revision: 00
Document Title: Soil Sieve	
Department(s): Wet Chemistry	

Date Information

Effective Date: 12 Apr 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest**Document Number:** ENV-SOP-GBAY-0164**Revision:** 00**Title:** Soil Sieve

All dates and times are in Central Time Zone.

ENV-SOP-GBAY-0164-Rev.00 Soil Sieve**QM Approval**

Name/Signature	Title	Date	Meaning/Reason
Elizabeth Turner (007857)	Manager - Quality Program	09 Apr 2021, 02:09:58 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Chad Rusch (007163)	General Manager 2	08 Apr 2021, 09:50:26 AM	Approved



TEST METHOD STANDARD OPERATING PROCEDURE

TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for drying and sieving soil samples to obtain a portion of soil for analysis.

- 1.1 Target Analyte List and Limits of Quantitation (LOQ) - Not applicable to this SOP.
- 1.2 Applicable Matrices: Soils and sediments.
- 1.3 Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

2.0 SUMMARY OF METHOD

A sample is homogenized and air dried. After air-drying, the sample is then sieved through a selected sieve size. The portion that passes the sieve is then ready for analysis.

3.0 INTERFERENCES

Not applicable to this SOP.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handling these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of

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solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount ¹	Preservation	Holding Time
Hg Samples	Ziplock Bag	2 cups	Thermal: ≤ 6°C Chemical: None	28 Days
All Other Metals	Ziplock Bag	2 cups	Thermal: ≤ 6°C Chemical: None	6 Months
Organic Parameters	16 oz glass jar	2 cups	Thermal: ≤ 6°C Chemical: None	VOA 14 Days SVOA 7 Days

¹Minimum amount needed for each discrete analysis.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0006 *Sample Management* (current revision or replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at ambient temperature until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ambient temperature until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

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7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment*	Manufacturer / Vendor*	Catalog #*
Sieve Shaker	RO-TAP®	RX-29
Sieve Shaker	Gilson	SS-15
Sieve Shaker	Endecotts	Minor 200
Sieve Shaker	Endecotts	Octagon 200
Sieve	Gilson or equivalent	Stainless steel, #10, #60, or other as needed
Sieve catch pans and lids	Gilson or equivalent	Stainless steel
Bakers' racks	Restaurant Supply	To hold 18" x 26" trays
Drying fan	Various	Local Store
Mortar ceramic/porcelain	Cole-Parmer	60322
Pestle ceramic/porcelain	Cole-Parmer	60323

*Or Equivalent

7.2 Supplies

Supplies	Vendor	Model/Version
Aluminum Foil Cake Pan	Durable Packaging / Webstaurant	612604245
8x8 Ziploc Bags	Fisher Scientific	23700218
12x12 Ziploc Bags	Uline	S-14416
Freezer Paper	Fisher Scientific or equivalent	50-200-5215
Wooden Rolling Pin	Restaurant Supply	Local Store
Rubber Mallet	Various	Local Store
Scissors	Various	Local Store

*Or Equivalent

8.0 REAGENTS AND STANDARDS

Not applicable to this SOP.

9.0 PROCEDURE

- 9.1 Balance calibration must be verified daily prior to use. Refer to SOP ENV-SOP-GBAY-0115 *Support Equipment* (current revision or replacement).
- 9.2 For any USDA marked samples, refer to SOP ENV-SOP-GBAY-0121 *Regulated Soil Handling* (current revision or replacement). Containers will be labeled with a pink Regulated Soil sticker.
- 9.3 Pulling Samples
 - 9.3.1 Batch the samples in the LIMS.

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- 9.3.2 Pull the samples from either the soil Walk-In Cooler or from the ambient storage area in the Physical Testing Lab and organize them in the order to be processed. Their location will be dependent on the analytical work, if any, that will be done after the sieving.
- 9.4 Create a new Dry Sieve Worksheet File.
 - 9.4.1 Use the Dry Sieve Template in the Dry Sieve folder, and make sure to “Save As”, using the Horizon Batch Number (HBN).
 - 9.4.2 Fill in the drying information for each sample on the Worksheet.
- 9.5 Air Dry Samples
 - 9.5.1 Wearing gloves, line a tray with freezer paper wax side down. Fold the sides of freezer paper up about 1- 1 1/2” on each side to form a “boat”.
 - 9.5.2 Label the freezer paper with the sample number. Place the entire sample on the freezer paper. Multiple trays may be used for drying if a large sample volume was received.
 - 9.5.3 Entire sample does not need to be dried if excess volume was received. Sample must be homogenized before splitting. Return undried portion to original container.
 - 9.5.4 Weigh and document remaining sample mass. Some projects may require this to be labeled as “Archive”.
 - 9.5.5 Spread the soil evenly. Break up all clumps into about 1/2” or less size pieces. This will speed the drying process and ease the disaggregation process prior to sieving. Continue this process for all samples in the set. Change gloves between each sample.
 - 9.5.6 In the drying logbook record the sample numbers, date, time, temperature, and humidity when the samples are placed in the drying cabinet. Place the entire set in a drying cabinet to air dry overnight. Longer drying may be required for wetter samples.
- 9.6 Soil Disaggregation
 - 9.6.1 After the samples are dried remove them from the drying cabinets. Record the date, time, temperature, and humidity in the drying logbook.
 - 9.6.2 Place a tray on the counter. Pick any rocks, twigs or other foreign matter and set to the side of the freezer paper boat.
 - 9.6.3 Disaggregate the soil. Disaggregation is the process of loosening the clumped soil. It is not meant to crush or reduce the natural particle size of the soil. Place a sheet of paper, wax side up, over the sample. Using a rolling pin, roll over the dried soil for 1-2 minutes. A rubber mallet or pestle may be used to disaggregate soil clumps. Take care that the sample remains on the freezer paper. If sample is hard clay, a porcelain pestle may be used to break up chunks, being careful not to crush rocks.
- 9.7 Soil Sieve Procedure Using #10 Sieve
 - 9.7.1 Place sieve on catch pan or clean freezer paper, wax side down. Pour sample into #10 sieve and sift. Gently rub the sample remaining on the sieve to break up clumps. When no more sample passes through sieve, dump all remaining sample on top of sieve onto

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- a separate sheet of freezer paper. If large clumps are still present, repeat disaggregation and sieve until no clumps remain.
- 9.7.2 The sample portion remaining in the #10 sieve is then weighed, documented, and bagged with the sample number and a “Coarse Fragments” label on it.
- 9.7.3 Weigh, document, and place all the sample passing through the #10 sieve into a labeled Ziploc bag with the sample number and a “Fines” label on it. Add any organic matter that had been removed previously. This Organic matter may need to be cut up into smaller pieces using clean scissors.
- 9.7.4 Change gloves between samples.
- 9.8 Soil Sieving Procedure using sieves other than #10
- 9.8.1 Determine the sieve sizes and process to be used to meet project specifications.
- 9.8.1.1 Check with the project manager or lab manager for project specifications.
- 9.8.1.2 If multiple sieve portions are to be obtained, stack the set of sieves in the with the largest size openings on top to the smallest on the bottom, with a catch pan at the base.
- 9.8.1.3 If sieve sizes smaller than a #10 sieve are being used, the #10 sieve can be used to not plug up the smaller sieve. Anything retained by the #10 sieve must be considered part of the biggest sieve portion.
- 9.8.2 Pour the dried and disaggregated soil onto top sieve.
- 9.8.3 Record the sample number on the side of the catch pan. An abbreviated number may be used such as 407-1.
- 9.8.4 All dried contents are poured onto the sieve including the rocks and foreign matter that had been set to the side. The organic foreign matter may need to be cut up into smaller pieces using clean scissors.
- 9.8.5 Place the set of sieves on a mechanical shaker. Tighten the mechanical shaker adjustments so that the sieves fit tightly and securely in the mechanical shaker. Set the timer for 10 minutes and begin the sieve shaking.
- 9.8.6 After 10 minutes remove the sieves from the mechanical shaker.
- 9.8.7 Weigh, document, and place all the sample contents in the catch pan into a labeled Ziploc bag with the sample number and a “Fines” label on it.
- 9.8.8 Great care should be taken in matching the sample number written on the catch pans to the sample numbers on the labeled container.
- 9.8.9 Certain projects may require that the portion of sample above the sieve be retained. If this is required pour the sample remaining on top of the sieve(s) into a second bag, label with the lab number and mark “Coarse Fragments”. Zero the balance with the same bags used, then weigh and document the mass of this portion.
- 9.8.10 Record the sieve date, analyst, shaker ID, and sieve size used on the Soil Sieve Prep Log. Note if coarse fragments were retained.

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9.9 Pulverization - Some projects or methods may require that the sieved sample be further pulverized prior to analysis. The sample may be pulverized with a motor and pestle or other method.

9.10 Cleaning Sieves – the sieves must be washed and dried between each use.

9.10.1 Place the sieves in the sink and scrub with a brush or green scrubbie and running hot water to remove any soil particles embedded in the mesh. Rinse well with tap water then rinse with deionized water. Soap is not used as it is very difficult to rinse from the sieves.

9.10.2 Place the sieves and catch pans in an oven to dry. Alternatively allow to air dry overnight on the counter.

10.0 DATA ANALYSIS AND CALCULATIONS

Not applicable to this SOP.

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control - Not applicable to this SOP.

11.2 Instrument QC - Not applicable to this SOP.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits - Not applicable to this SOP.

12.0 ANALYST QUALIFICATIONS AND TRAINING

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-GBAY-0094 *Training and Employee Orientation* (current revision or replacement) for more information.

13.0 DATA REVIEW AND CORRECTIVE ACTION

Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

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The review steps and checks that occur as employees complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-GBAY-0120 *Data Review and Final Report Processes* (current revision or replacement) for specific instructions and requirements for each step of the data review process.

13.1 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

There is no QC performed with this analysis.

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TITLE: Soil Sieve

TEST METHOD ENV-SOP-GBAY-0164

ISSUER: Pace ENV – Green Bay Quality – GBAY

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14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

15.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

16.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

17.0 ATTACHMENTS

Attachment I: Sieve prep log (Example)

Attachment II: Dry Sieve Flow Chart

18.0 REFERENCES

18.1 Pace Quality Assurance Manual - most current version.

18.2 The NELAC Institute (TNI); Volume 1, "Management and Technical Requirement for Laboratories Performing Environmental Analysis" - most current version.

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19.0 REVISION HISTORY

This Version: ENV-SOP-GBAY-0164-Rev.00

Section	Description of Change
All	First Issue of SOP.

This document supersedes the following document(s):

Document Number	Title	Version

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Attachment I: Sieve Prep Log

Work Order		Date/Time In		Humidity In (%)		Temp In (°C)		Reviewed by
Batch		Date/Time Out		Humidity Out (%)		Temp Out (°C)		
Samples	Sieve Date	Shaker ID	Analyst	Archive Weight (g)	Analyst	Weight of >60 Mesh (g)	Analyst	Weight of <60 Mesh (g)
				Balance ID: 40BALW		Balance ID: 40BALX		Balance ID: 40BALX
-001		40SKR3						
-002		40SKR4						
-003		40SKR5						
-004		40SKR3						
-005		40SKR4						
-006		40SKR5						
-007		40SKR4						
-008		40SKR6						
-009		40SKR3						
-010		40SKR4						
-011		40SKR6						
-012		40SKR7						
-013		40SKR8						
-014		40SKR3						
-015		40SKR4						
-016		40SKR5						
-017		40SKR6						
-018		40SKR7						
-019		40SKR8						
-020		40SKR4						

A similar version including the same information may be used.

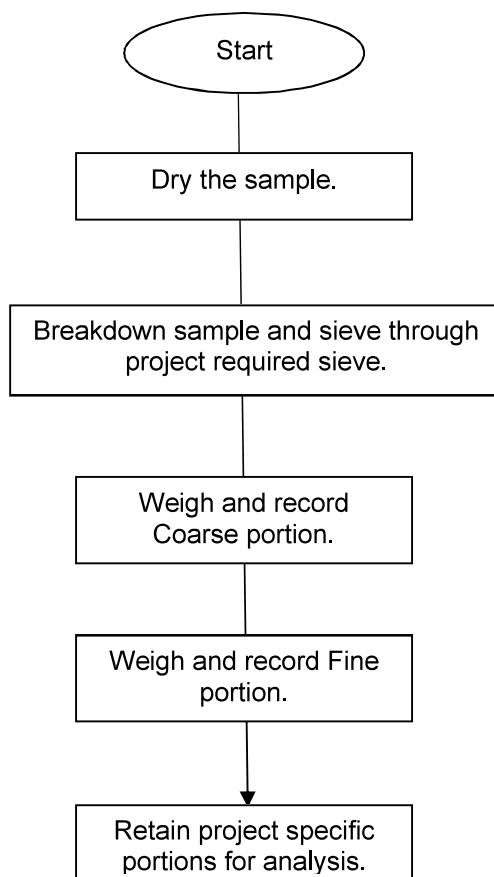
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Attachment II: Dry Sieve Flow Chart



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