

January 19, 2023

Nicole Yuen Department of Toxic Substances Control 700 Heinz Avenue, Suite 200C Berkeley, California 94710

Sara Ziff U.S. Environmental Protection Agency, Region 9 75 Hawthorne Street San Francisco, California 94105

Via electronic mail

#### Subject: Corporation Yard: East Meadow and Building 120 Area, Revised Richmond Field Station University of California, Berkeley

Dear Ms. Yuen and Ms. Ziff:

This letter offers recommendations as a follow-up to the *Corporation Yard, Data Gaps Sampling Results* letter, dated November 22, 2019, prepared by Tetra Tech, Inc. This letter updates the version dated August 24, 2022 based on our discussion on December 21, 2022, and comments received from DTSC dated November 10, 2022, included as Attachment 1. The November 2019 letter provided incremental sampling methodology (ISM) results from 17 decision units in three Corporation Yard areas, summarized below.

Corporation Yard Area	Description	Decision Units
East Meadow	Follow-up sampling at Excavation 8, north of Building 197, within the East Meadow.	DU1, DU2, DU3
Building 120	Follow-up sampling at Excavation 3A, 3B, and 4, adjacent to Building 120.	DU4 through DU8
South of Building 120	Follow-up sampling in all areas not previously sampled within the fence line from south of Building 120, west of Building 178, and west and south of Building 185, up to Egret Way.	DU9 through DU17

The three areas are shown on Figure 1. This letter offers recommendations for the East Meadow and Building 120 areas. The areas south of Building 120 were addressed through additional triplicate sampling, as conveyed in the *Corporation Yard, Triplicates Sample Results* letter, dated May 23, 2022, prepared by Tetra Tech. The May 2022 letter recommended no further evaluation or soil cleanup activities within DU9 through DU17.

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#### EAST MEADOW AREA

The established cleanup goal for total polychlorinated biphenyls (PCBs) is 1 milligram per kilogram (mg/kg). Sample results from DU1, DU2, and DU3 have total PCBs from 0.97 to 1.51 mg/kg. Confirmation sample results at Excavation 8 have total PCBs from 0.53 to 8.96 mg/kg. Sample results from DU1, DU2, DU3, and Excavation 8 are shown on Figure 2. Sample results from three additional decision units in the East Meadow have total PCB concentrations from 0.052 to 1.46 mg/kg, as shown on Figure 1. The East Meadow decision units were sampled to evaluate surface areas impacted by equipment and trucks within the meadow, and were not intended to provide complete characterization of the meadow.

Additional sampling is recommended to further characterize the East Meadow, including the area adjacent to DU1, DU2, and DU3. Figure 3 shows six proposed decision units (DU18 through DU23). ISM protocols will be consistent with all previous ISM protocols within the Corporation Yard.

#### **Sampling Methodology**

ISM will be applied to collect and analyze soil samples from DU18 through DU23. ISM involves collecting many small soil masses (called "increments") evenly across each decision unit, and then pooling them to form a field sample. ISM was selected to achieve a comprehensive and thorough evaluation of chemical concentrations in a specific volume of soil or within a decision unit. Field quality control (QC), in the form of three independent field samples (i.e., field triplicates), assesses ability of an ISM sample to reliably estimate concentrations within the decision unit and quantify inherent soil and contaminant heterogeneity. A field triplicate will be collected at DU21.

Once received at the laboratory, the ISM sample will be processed to homogenized and then subsampled for analysis. QC to assess adequacy of sample processing, subsampling, and analysis will be conducted on three subsamples taken from one of the field triplicates. The field and laboratory subsampling triplicates form an ISM "nested triplicate" set from which the amount of variability due to field heterogeneity and laboratory procedures will be calculated as a statistic called the relative standard deviation (RSD). An RSD will be calculated for both the field triplicates and laboratory triplicates to measure how much field heterogeneity versus laboratory measurement variability contribute to overall data variability.

While ISM procedures are designed to reduce both field and laboratory contributions to data variability, some variability is inevitable. Measurements provided by a nested triplicate set document whether the procedures sufficiently reduced variability for the site-specific matrix and contaminants. If this QC demonstrates that data variability is too high to support desired decision confidence at the action level, it also indicates which aspect, field sampling, sample processing and subsampling, or the analysis itself needs corrective action to fix the problem. In contrast, sources of data variability are rarely used in this way in discrete sampling programs, which limits options for corrective action if discrete data variability is too high. Soils contaminated with PCB typically have both high field heterogeneity and high subsampling variability, so meticulous procedures must be implemented. ISM was chosen for this work because ISM procedures will produce PCB data with much lower data variability and therefore elicit higher confidence than data from discrete sampling.

A field sample will consist of a minimum of 75 increments collected from each decision unit. In addition to chemical results, field triplicate results from DU21 will measure the effectiveness of the ISM sample in capturing PCB contaminant variability within the decision unit. The field triplicate results will inherently include any laboratory variability because each field triplicate is analyzed separately by the laboratory.

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Specific ISM procedures for field sampling will be as follows:

- 1. Corners and edges of each decision unit will be marked with flags to identify where increments will be collected. Triplicate increments for DU21 will be placed equidistant in a triangle formation at each point, as shown on Figure 3.
- 2. Increments will be collected from the top 2 inches of the native surface with a disposable scoop or other disposable sampling apparatus. In some areas, the native surface is the current surface cover; however, any gravel will be removed prior to increment collection. Where river rock is present, a backhoe will be used to scrape aside the river rock prior to sampling. Each increment will be approximately 20 grams of soil.
- 3. Increments from each decision unit will be placed into freezer-grade, 1-gallon, zip-locking bags. The target weight of each ISM sample is approximately 1.5 kilograms. Each bag will be labeled and packed into an insulated cooler and covered with ice packs. The samples will be transported under chain-of custody procedures to McCampbell Analytical, in Pittsburg, California.

Health and safety measures will conform to the *Final Field Sampling Workplan, Appendix B, Health and Safety Plan*, dated June 2, 2010.

#### Laboratory Processing, Subsampling, and Analyses

Soil samples will be processed according to the laboratory's internal ISM protocol, specifically:

- 1. The 1.5-kilogram sample will be air-dried as necessary, then passed through a 10-mesh sieve to remove non-soil material (i.e., particles larger than a 2-millimeter [mm] diameter).
- 2. The sieved soil will be ground to the consistency of sifted flour and spread into a shallow layer in a pan to form a "slab cake" and divided into 30 equal-sized grid cells.
- 3. A 1-gram increment will be taken from each grid cell, and the 30 increments will be pooled to form an analytical subsample weighing 30 grams.
- 4. Each 30-gram subsample will be analyzed for PCBs via U.S. Environmental Protection Agency (EPA) Method 8082 with 3540C Soxhlet extraction.

One of the field samples within the DU21 field triplicate set will be subsampled and analyzed two additional times (for a total of three subsample analyses) to create the laboratory triplicate set. The second and third independent representative subsamples will be collected in the same way by taking separate increments from the same 30 grid cells. The standard operating procedure for McCampbell Analytical Inc. laboratory processing is included as Attachment 2.

The primary purpose of the laboratory triplicate set is to evaluate effectiveness of processing and subsampling protocols for site-specific contaminants and the soil matrix. If the procedures are effective, the three subsamples should yield numerically close results. The closer the agreement among the results, the lower the data variability and RSD for the triplicate set. Variability in the analytical processes of sample extraction, extract cleanup, and instrumental measurement is an unavoidable inclusion in subsampling variability. If necessary, the contribution by analytical variability to subsampling variability can be determined via various analytical QC checks, such as use of laboratory control samples (LCS) and surrogate recoveries.

Together, the field triplicate set and laboratory triplicates from one of the field triplicates constitute a nested triplicate.

## Laboratory Triplicate Evaluation

Laboratory triplicates will be evaluated quantitatively and qualitatively to determine overall data usability. Quantitative evaluation involves calculating an RSD of results from the three laboratory triplicates as a measure of variability. Qualitative evaluation involves assessing whether concentration ranges of laboratory triplicates agree generally (low, moderate, or elevated), and whether these exceed the action level. Low or high variability can indicate complexity of the matrix. Consistently high variability may indicate a complex matrix with "particle effects" that cannot be fully eliminated even by enhanced laboratory protocols such as milling the sample. A data usability determination will be recommended based on results of the quantitative and qualitative analyses.

High subsampling variability leads to high variability in field sample results. If variability in field samples is too high to meet desired decision confidence, a mathematical determination of relative contributions of field, subsampling, and analytical variability will be performed. If subsampling variability is determined to be a significant contributor to overall data variability, corrective action may be required including modifying procedures for sample processing and subsampling.

## **Field Triplicate Evaluation**

The QC role of a field triplicate set is to provide statistics that, in conjunction with statistics from the laboratory triplicate set, allow determination of the respective contributions of these sets to overall data variability. A project with high data variability will result in inefficient site investigations and cleanup.

High data variability is detrimental when it leads to high rates of false positive decision errors when upper confidence limits (UCL) are the basis for cleanup and other decisions. A UCL is an upper bound (or "limit") on estimated decision unit concentration. The sampled concentration (i.e., the average concentration determined from decision unit samples) is an estimate of the true concentration. In contrast, a UCL estimates an upper bound on the true concentration. A UCL is calculated by adding a safety factor to the average obtained from sample results. The size of this safety factor is increased by high data variability, pushing the UCL farther away (higher) from the average. When variability is high, the distance between the average or mean and the UCL can be so large that the UCL exceeds an action level even if the true decision concentration (as suggested by the sample mean) is well below the action level. Therefore, decisions based on the UCL can lead to false positive decision errors when data variability is high. This scenario is exemplified in the following diagram:



A large distance between the mean and UCL indicates significant uncertainty about the true concentration; however, this large data uncertainty might not cause decision uncertainty or decision errors if the mean is far enough below or above the action level. If a confident decision is possible, corrective action to reduce data variability may not be necessary. The diagram above exemplifies elevated decision uncertainty: the location of the data mean with respect to the action level indicates the true mean should

be below the action level, but a UCL exceedance suggests that high data variability renders that conclusion uncertain.

Field triplicates are three independent measures of the decision unit concentration that provide a measure of data variability in the form of an RSD calculated from the three results. When field triplicate results are close (i.e., precise), data variability and the RSD are low. Low RSDs contribute to a narrow mean-to-UCL width, which gives higher confidence that the true mean is near the data mean. As illustrated in the graphic below, that allows decisions based on the UCL to produce fewer false positive decision errors.



If high data variability is causing large mean-to-UCL widths and excessive decision uncertainty, the QC (i.e., the field and laboratory triplicate sets) can target where corrective action will be most effective. If laboratory subsampling variability is high, that problem must be corrected first via application of options described previously. If subsampling variability is low but field triplicates variability is high, corrective actions need to target ISM field sampling procedures. Options include reassessing the size and layout of DUs; increasing the number of field increments and/or mass of those increments; and collecting triplicates from more, or all, of the DUs. The mathematics of the UCL calculation means that adding additional replicates to DU data sets (e.g., using four replicates rather than three) will lower the UCL even if the mean and RSD remain the same.

One of the laboratory triplicate results is used as the concentration of the parent field sample. By convention and to parallel the data from the other two field triplicate samples, the project will use the first laboratory subsample result as the concentration for the parent field sample. Possibly under some circumstances, the field sample concentration may be better represented by averaging all three laboratory triplicate results. An example might be if the first result, and only that result, is a nondetect. The best way to evaluate results cannot be determined until the data are received. The convention of using the first laboratory triplicate result will avoid temptation to "cherry-pick" laboratory triplicate data to obtain the lowest RSD for the field triplicate set.

#### Field Triplicates and Calculating the Weighted-95UCL

The objective for this proposed data collection is to enable determination of mean and UCL concentrations within the large area encompassed by DU18 through DU23. A non-weighted simple average assumes that areas of all DUs are the same, which is not appropriate because the DUs are of various sizes. Logically, a large area should have more influence on overall concentration than a small area; so an area-weighted mean, not difficult to calculate, better represents the true mean over the large area. Obtaining an area-weighted UCL, however, to accompany the area-weighted mean involves complicated calculations that will occur in a specially designed spreadsheet called the "Combining DUs Calculator." The spreadsheet was first designed by Philip Goodrum, Ph.D., a statistician and toxicologist with GSI Environmental, and contributor to Interstate Technology and Regulatory Council for 2012 and 2020 ISM guidances. The spreadsheet was structured to accept ISM field replicate data as the only inputs. The Calculator would then compute the mean and variability from the raw sample data. Deana Crumbling of Tetra Tech, in collaboration with Mr. Goodrum, modified the Calculator in 2020 into a version that can accept DU means, as determined from one or more ISM samples, and RSDs as the inputs. This calculator

has been discussed previously with DTSC and EPA, and has been utilized to aid the PCB removal action at the EPA North Meadow, located within RFS approximately 975 feet west of the Corporation Yard.

An estimate of within-decision unit (i.e., internal) variability is required for each component decision unit to compute a weighted-UCL over the large area. The field triplicate provides that measure of within-decision unit variability. Variability found in DU21 can be applied to the other decision units with only a single ISM sample (termed "singlet-decision units"). A reasonable initial assumption is that variability at DU21 will be similar to that at the other decision units, as these are equivalent from a conceptual site model (CSM) perspective. "CSM-equivalent" decision units are those subjected to the same contaminant release and transport mechanisms, in reasonably close proximity, and expected to have similar concentrations in relation to the action level.

Note that if all sample results are below 1 mg/kg, UC Berkeley will conclude that existing site conditions meet the cleanup goal and recommend that no further statistical evaluation is necessary in the sampled areas. The calculator tool will be used in the event field sample concentrations exceed 1 mg/kg.

#### Application of RSDs in the Calculator

Following receipt of laboratory data, the DU21 field triplicate results will be reviewed for comparability with results from the other decision units. Assuming the results are comparable, the RSD from DU21 triplicates will be applied to the other singlet-decision unit results. Laboratory results, RSDs, and decision unit areas will be entered into the calculator. The Calculator provides two types of overall weighted-95UCL results: the Student's t UCL is used for normal data distributions, and the Chebyshev UCL is used for nonnormal distributions. The Calculator also recommends which of the two UCL options to use.

Evaluation of all triplicate results and application of RSDs should always proceed case by case, and will be discussed with EPA and DTSC following receipt of results. Following discussion with DTSC and EPA, sample results will be presented in a sampling letter report providing complete details regarding the updated weighted 95UCL. Methods and equations and calculation results will be presented within the sample results summary. Recommendations will be offered for further action within the East Meadow and as a follow-up to Excavation 8 sampling.

Data collected during this investigation ultimately will be presented with the comprehensive data following completion of all Corporation Yard removal action activities.

#### **BUILDING 120 AREA**

The Building 120 Area consists of previous Excavations 3A, 3B, and 4, DU4 through DU8, and surficial decision units surrounding Excavation 3A sampled during excavation activities. Per discussions with DTSC and EPA, secured fencing with PCB warning signage was placed around the area, as shown by the red fence pattern on Figure 2.

UC Berkeley understands that the residual levels of PCB contamination within the Building 120 Area do not meet the 1 mg/kg cleanup objective of the RAW or the TSCA Agreement. Currently, this area is proposed for excavation as a part of the adjacent Campus Bay development, and UC Berkeley proposes consolidation of removal of the residual PCB contamination with the redevelopment excavation activities.

In Spring 2023, UC Berkeley proposes to remove all surficial soil at DU7 to a depth of 1.5 feet below ground surface to remove the highest concentrations of PCB-contaminated soils. All excavation activities

would adhere to confirmation sampling protocols, soil management, and disposal protocols appropriate for PCB-contaminated soils within the RAW and TSCA Agreement for the Corporation Yard.

While a specific schedule has not been established for the redevelopment of the Corporation Yard, representatives from Campus Bay have indicated that a schedule and soil management plan will be issued following discussions with UC Berkeley within the next few years.

The secured area shown on Figure 2 will help ensure no exposure to workers or visitors on site, and RFS staff will not be permitted to enter the secured area. UC Berkeley also has halted use of areas north of Building 120, and has cleared away all equipment and materials from the area to further minimize any exposure to RFS staff. The locked gate west of Building 197 will ensure entry of only RFS staff to the Corporation Yard. All RFS staff permitted to enter the area are Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER)-trained, and aware of elevated PCB levels in the area. Photographs of the secured area and signage are in Attachment 3, Photolog.

If you have any questions or comments regarding this submittal, please reply by email or call me at (415) 497-9060 or Alicia Bihler at (510) 725-2528.

Sincerely,

Jason Brodersen, P.G.

Project Manager

 Attachments:
 Figure 1: Site Map

 Figure 2: Previous Sampling Results
 Figure 3: Decision Units 18 through 23

 Attachment 1: Regulatory Comments
 Attachment 2: McCampbell Analytical Inc. Laboratory Processing Standard Operating

 Procedure
 Attachment 3: Photolog

cc: Alicia Bihler, UC Berkeley EH&S John Edgcomb, Edgcomb Law Group



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1/6/2023 C:\misc\_GIS\Richmond\_Field\_Station\Projects\Corporation Yard\Figure3\_DUs18-23.mxd TtEMI-OAK michelle.handley

## ATTACHMENT 1

# **REGULATORY COMMENTS**

Meredith Williams, Ph.D., Director 700 Heinz Avenue Berkeley, California 94710-2721

**Department of Toxic Substances Control** 

# MEMORANDUM

- TO: Nicole Yuen, Project Manager Senior Environmental Scientist Cleanup Program, Berkeley Office Site Mitigation and Restoration Program
- FROM: Mark Sorensen, PG 7448 Engineering Geologist Geological Services Branch – Berkeley Site Mitigation and Restoration Program

November 10, 2022

SUBJECT: REVIEW OF CORPORATION YARD: EAST MEADOW AND BUILDING 120 AREA, RICHMOND FIELD STATION, UNIVERSITY OF CALIFORNIA, BERKELEY

SITE 201605-00 PCA: 11018 MPC: TECHMEMO WR 20088626

## DOCUMENT REVIEWED

As requested, the Berkeley Geological Services Unit (GSU) has reviewed the *Corporation Yard: East Meadow and Building 120 Area, Richmond Field Station, University of California, Berkeley* (Letter), dated August 24, 2022. The Letter was prepared by Tetra Tech, Inc. The Letter offers recommendations as a follow-up to a November 2019 letter that provided incremental sampling methodology (ISM) results from 17 decision units in three Corporation Yard areas. The Report was reviewed with respect to geologic and hydrogeologic interpretations and technical adequacy.

## BACKGROUND

The established cleanup goal for total polychlorinated biphenyls (PCBs) in site soils is 1 milligram per kilogram (mg/kg). ISM sampling addressed the area referred to as "South of Building 120" through additional triplicate sampling, as conveyed in an earlier letter of May 2022 that recommended no further evaluation or soil cleanup in that area





Gavin Newsom Governor





Yana Garcia

Secretary for

**Environmental Protection** 

DATE:

Nicole Yuen November 10, 2022 Page 2 of 3

(represented by decision units [DUs] 9 through 17). The remaining two areas are the subject of this Letter, with (1) additional ISM sampling proposed to characterize soils in a portion of the East Meadow Area, and (2) excavation proposed for areas around Excavations 3A, 3B, and 4 that exceed the cleanup goal.

## COMMENTS AND RECOMMENDATIONS

- East Meadow Area, Page 2 In the second paragraph, DU2 is listed twice in the first sentence. Please change the second DU2 to DU3.
- 2. Laboratory Processing, Subsampling, and Analyses, Page 3 Please cite the applicable quality assurance project plan (QAPP) or sampling and analysis plan (SAP), or state whether new plan(s) will be created.
- 3. Field Triplicates and Calculating the Weighted-95UCL, Pages 5-6
  - a. At the end of the first paragraph, EPA North Meadow is mentioned. Please indicate its location relative to the areas discussed in the Letter.
  - b. In the third paragraph of this section, please edit the text as follows:

"Note that if all sample results are below 1 mg/kg, UC Berkeley will conclude that existing site conditions meet the cleanup goal and recommend that no further statistical evaluation is necessary **in the sampled areas**."

This clarification appears necessary because we know that laboratory results for certain samples from the East Meadow do not meet the cleanup goal.

## 4. Building 120 Area, Page 6

In the first paragraph of the section, please edit the text to read:

"Per discussions with DTSC and EPA, secured fencing with PCB warning signage was placed around the area, as shown **by the red fence pattern** on Figure 2."

5. Figures 1 and 2

Unneeded confusion is introduced by referring to DUs 1 through 17 as "New Decision Units," as they are currently indicated in the legends of these two figures. These DUs are no longer "New," having been sampled in 2019. Also, the title of Figure 2 refers to results of these DUs as "Previous Sampling Results." Please change the legend designation to "Previously Sampled Decision Units" or simply "Decision Units."

6. Figure 3

Please indicate in the title or elsewhere that the depicted area is within the East Meadow Area.

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If you have any questions or comments regarding this memorandum, please contact Mark Sorensen at (510) 540-3947 or <u>Mark.Sorensen@dtsc.ca.gov</u>, or Jon Buckalew (Buck) King at (510) 540-3955 or <u>Buck.King@dtsc.ca.gov</u>.

Reviewed by:Theodore (Ted) Mazzoli, PGEngineering Geologist, Geological Services Unit<br/>Geological Services Branch<br/>Site Mitigation and Restoration Program

## ATTACHMENT 2

# MCCAMBELL ANALYTICAL INC.

# LABOROTORY PROCESSING STANDARD OPERATING PROCEDURES

McCampbell Analytical Inc.

#### Samp-ISM R02

This documentation has been prepared by McCampbell Analytical Inc. solely for MAI's own use and the use of MAI's customers in evaluating its qualification and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to McCampbell Analytics Inc. upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

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# Incremental Sampling Methodology (ISM)

# Laboratory Processing For PCBs and Semi-Volatiles Compounds

Document No. Samp-ISM	<b>Rev No.</b> 02	Effective Date: 02/15/2021

Reviewed and Approved By: \_

Angela Rydelius

Date

Reviewed and Approved By: <u>Maura Johnson</u>

<u>02-09-2021</u> Date

## Disclaimer

The current TNI Standard, Volume 1, Module 2 Quality Systems General Requirements, Section 4.2.8.5 states with regard to laboratory SOPs.

- a. These documents, for example, may be equipment manuals provided by the manufacturer, or internally written documents with adequate detail to allow someone similarly qualified, other than the analyst, to reproduce the procedures used to generate the test result.
- b. The laboratory shall have and maintain an SOP for each accredited analyte or method.
- c. The documents that contain sufficient information to perform the tests do not need to be supplemented or rewritten as internal procedures, if the documents are written in a way that they can be used as written. Any changes, including the use of a selected option must be documented and included in the laboratory's methods manual.
- d. The test methods may be copies of published methods as long as any changes or selected options in the methods are documented and included in the methods manual.

In accordance with these instructions, this SOP is an internally written document that acts as a supplement to the published method it references. This SOP does not stand alone and is to be used in conjunction with the published method. Instrument specific instructions, quality control summaries, as well as internal MAI policies are referenced in this SOP, including any deviations from the published method, if any such deviations exist. In the absence of a stated deviation, this SOP adheres strictly to all the requirements of the published method, regardless of whether or not those requirements are explicitly stated in this document.

## **1.0** Scope and Application

1.1 This Standard Operating Procedure describes MAI procedure and/or Guidance on handling and processing of whole soil and sediment samples for representative subsampling and analysis using the Incremental Sampling Methodology (ISM, see reference 4). The ISM method is designed to statistically reduce or limit the variability associated with discrete sampling and to generate a single representative sample for a given area (or 'decision unit').

#### 2.0 Method Summary

- 2.1 All field collected incremental subsamples representing a single unit are combined and processed (by mixing/homogenizing) into a single sample. The composited sample is air dried under a hood to constant dryness. The dried sample then undergoes particulate size reduction by grinding using the Retsch grinder/crusher.
- 2.2 Sample particulate size is reduced to the level required to pass through a <2mm sieve. The homogenized sample is either: 1) systematically subsampled from a flat tray in 30 different locations (an additional multi-increment sampling) or 2) is split using a rotary sample splitter/divider.
- 2.3 The resulting composited aliquot is analyzed according to the required method procedure(s).

#### 3.0 Definitions

3.1 Definitions are in the Quality Manual, section 3.3 Glossary and Acronyms.

#### 4.0 Interferences

4.1 Not applicable to this procedure.

#### 5.0 Safety

- 5.1 Proper Personal Protective Equipment (PPE) is used in all instances of laboratory practice to assure safety of laboratory personnel at all times. A laboratory coat, eye protection, and gloves are the minimum requirements.
- 5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available.

### 6.0 Equipment & Supplies

- 6.1 Retsch BB50 grinder.
- 6.2 Sieve (2mm opening: #10 US).
- 6.3 Drying trays.
- 6.4 Dust mask (toxic dust respirator preferred, e.g., MSA Safety #817664 mask).
- 6.5 Sample splitter (or tray method).

## 7.0 Reagents & Standards

7.1 This section is not applicable to the process.

#### 8.0 Sample Collection, Preservation, Shipment & Storage

8.1 Samples can be collected in various containers in a sealed container. Once received the samples are stored between 0-6 °C. There is no specified hold time.

#### 9.0 Quality Control

- 9.1 One Method Blank must be analyzed with each analysis batch. The results must be below the RL before continuing the analysis. If not, corrective action must be taken.
- 9.2 Matrix spike and Matrix spike duplicate may be analyzed with every batch of samples as required by the COC.
- 9.3 A Lab Control Sample (LCS) may be prepared and analyzed with each analysis batch as required by the COC.

#### **10.0** Calibration & Standardization

10.1 This section is not applicable to the ISM process.

#### 11.0 Workflow

11.1 The figure below is the official Incremental Sampling Methodology workflow as depicted in the ISM-1 published method (Interstate Technology & Regulatory Council (ITRC) 2012). This is Figure 6.1 from the ISM-1 method indicating: the state of the sample(s) upon acceptance by the laboratory, the laboratory processing required the subsampling methodology and the various laboratory analysis pathways.





#### 12.0 Procedure

#### 12.1 Compositing

- 12.1.1 An ISM sample is a composite sample made up of 30+ smaller individual samples, typically core samples of uniform size and weight. If we receive a single large sample (1 kg or more) we assume that the 30+ smaller individual samples have already been combined by the client in the field. If the sample arrives at the lab as a collection of small samples (30+ tubes or cores) then the individual cores will be combined into a single sample.
- 12.1.2 The COC must indicate whether vegetation, oversized material, or decantable water are to be included or excluded from the sample. Decantable water can be poured off the top of the settled sample. Vegetation and oversized material can be manually removed with tweezers or spatulas but may be removed more reproducibly by sieving once the sample is dried. The excluded materials can be weighed and documented via photographs; and weight adjusted/removed when appropriate.

#### 12.2 Sample Drying

- 12.2.1 The samples must be dry enough to pass through the grinder without sticking or jamming. Weigh the sample to determine initial weight of the sample. Dry the entire soil sample, including organic material, at room temperature (or less) to a constant weight, being careful not to expose the samples to direct sunlight (final weight = constant weight).
- 12.2.2 Use trays to dry the samples under the hood (see Figure 1). Once the entire sample is air-dried large pebbles and vegetation (sticks) should be removed prior to grinding. The drying process may take several days for wet soils.



Figure 1 - Drying the (composited) sample

## 12.3 Sample Grinding

12.3.1 The entire dried sample is ground using the Retsch BB50 Grinder to a final particulate size of 2 mm or less (passes through a #10 sieve). The most common sieve size for ISM samples is <2 mm (standard #10 sieve), but specific objectives may necessitate a smaller or larger sieve.

## 12.4 Subsampling

- 12.4.1 To obtain a representative subsample the entire sample should be spread out on a clean tray (use aluminum foil if Al content is unimportant) to a thickness of 1 to 2 cm. This work should take place in a fume hood designed to prevent the spread of dust and minimize possible inhalation. Mark out a grid of 30 squares on the top surface of the sample (see Figure 2, below).
- 12.4.2 A small sub-sample is then taken by removing material that represents the entire vertical column of the cake a small plastic corer will work. The sub-sampled material is placed in a receiving container. This process is repeated for every grid of the entire spread-out sample. The resulting subsample is typically 10-30g in size. However, as the entire subsample should be used for an analysis the sample size collected should match the size required for that particular analysis.
  - 12.4.2.1 This will help eliminate inhomogeneity issues arising from using only part of a sampled aliquot. 0.33g collected from 30 grids will yield a 10g sample. To further reduce the uncertainty this sample should be mixed in a bladed mixer prior to analysis – unless the entire 10-30g sample will be used for a given analysis.



Figure 2 - 30 square grid marked on sample; sampling the grids

**Note:** If a rotary sample splitter is available then the entire sample is placed in the splitter hopper and one or more aliquots are collected from the entire dried sample.

#### 12.5 Sample Extraction

12.5.1 The resulting 10-30g soil sample aliquots are extracted according to the particular method extraction procedure.

#### **13.0** Data Analysis & Calculations

13.1 This section is not applicable to the ISM process.

#### 14.0 Method Performance

14.1 True method performance can only be measured by verifying sample homogeneity between subsample aliquots. In general, multi-incremental sample replicates are usually normally distributed with very few outliers.

#### **15.0** Pollution Prevention

- 15.1 This method does not contain any specific modifications that serve to minimize or prevent pollution.
- 15.2 The chemicals used in this method pose little threat to the environment when properly managed.
- 15.3 All standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of disposable waste.
- 15.4 For further information on pollution prevention consult Less is Better: Laboratory Chemical Management for Waste Reduction, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington D.C. 20036, (202) 872-4477.

#### 16.0 Corrective Actions for Out of Control data

16.1 Refer to Nonconformance/Corrective Action Report (NC/CAR/PR) Procedure.

#### 17.0 Contingencies for Handling Out of Control Data or Unacceptable Data

17.1 Contact the laboratory manager or technical manager to assess out of control / unacceptable data.

#### **18.0** Waste Management

18.1 All wastes must be disposed of safely, samples and extracts are disposed of following local, state, and federal regulations along with MAI's internal laboratory procedure, G-Waste Disposal.

#### **19.0** References

- 19.1 EPA Method 8330B. Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography (HPLC)
- 19.2 EPA Contaminated Site Clean-up Information; www.CLU-IN.org; Soil Sampling and Decision Making Using Incremental Sampling Methodology - Part 1; www.clu-in.org/conf/itrc/ISM\_110612/
- 19.3 Test Methods for Evaluating Solid Waste SW846.
- 19.4 Incremental Sampling Methodology; <u>http://www.itrcweb.org/ism-1/</u>
- 19.5 Incremental Sampling Methodology, Section 6. Laboratory Sample Processing and Analysis; www.itrcweb.org/ism-1/6\_LABORATORY\_SAMPLE\_PROCESSING\_AND\_ANALYSIS.html
- 19.6 State of Alaska Department of Environmental Conservation, Division of Spill Prevention and Response, Contaminated Sites Program, Draft Guidance on Multi-Incremental Soil Sampling, March 2009.

## 20.0 Revision History

20.1 Provide justification and explanation of change: *The procedure was reviewed and no changes were needed.* 

ATTACHMENT 3

# PHOTOLOG

# Attachment 3 - Photolog Corporation Yard Building 120 Area





# Attachment 3 - Photolog Corporation Yard Building 120 Area



## Attachment 3 - Photolog Corporation Yard Building 120 Area

