SHANNON & WILSON
GEOTECHNICAL AND ENVIRONMENTAL CONSULTANTS

May 18, 2021

Ms. Sammy Cummings and Mr. Marcus Zimmerman
Alaska Department of Transportation & Public Facilities
Southcoast Region
P.O. Box 110218
Juneau, Alaska 99811

RE: GUSTAVUS AIRPORT STOCKPILE SAMPLING AND ANALYSIS PLAN, GUSTAVUS, ALASKA

We understand the Alaska Department of Environmental Conservation (DEC) has requested sampling of the stockpile adjacent to the long runway at the Gustavus Airport (GST) via incremental sampling methodology (ISM). The purpose of an ISM sample is to report statistically defensible mean analyte concentrations within a given area or bulk quantity of material, known as a decision unit (DU). To meet the strict criteria of a representative and reproducible ISM sample result, the sample collection process must adhere to Interstate Technology and Regulatory Council (ITRC) guidance.

An ISM sample is a composite of a representative number of subsamples referred to as increments. ISM samples are more robust and representative than a typical composite sample because the entire DU is subdivided into units of equivalent surface area and/or volume. An increment of equivalent mass is collected from each of these subunits, such that every portion of the entire DU is represented equally within the final composite. The location from which the increments are collected within the subunits is determined through some form of random selection to remove procedural bias. Replicate ISM samples are collected at a rate of 20 percent of the overall project ISM samples, or at minimum of one set per area of concern. Replicates are collected to analyze the precision of the method and to calculate 95 percent upper confidence limits (95% UCLs) for the target analytes.

Prior to any ISM sampling effort, the number of DUs must be determined. For stockpiles, maximum DU volume is typically capped at 500 to 750 cubic yards (cy). We understand that the quantity of material at the Gustavus Airport is estimated at roughly 1,400 cy, necessitating a minimum of two DUs. With replicates collected on one of these two DUs, we would be required to submit a total of four ISM samples.

2355 Hill Road ▪ Fairbanks, Alaska 99709-5326 ▪ 907 479-0600 ▪ Fax 907 479-5691
▪ www.shannonwilson.com ▪

Two potential processes are proposed to perform ISM sampling on the existing stockpile in such a way as to conform to ITRC guidance. These two methodologies are detailed in the following sections.

METHOD 1: IN-SITU

The in-situ method can be performed with hand tools but requires the stockpile be molded into a shape having defined dimensions and uniform depth. This is done so that the sampler can divide the surface area of the stockpile into a grid of cells having equivalent area and volume. Typically, the stockpile would be spread out, such that the soil fills a rectangular profile with a flattened surface. A grid is physically staked out on the surface of the stockpile and random selection is used to determine increment sample locations within the grid cells. The depth of the stockpile should be roughly one to two feet above ground surface if a single layer sample is desired. A depth of two to four feet is manageable if the DU is divided into two vertical layers and sampled at two depth intervals. This requires the use of an auger.

Grid Creation and Sample Collection

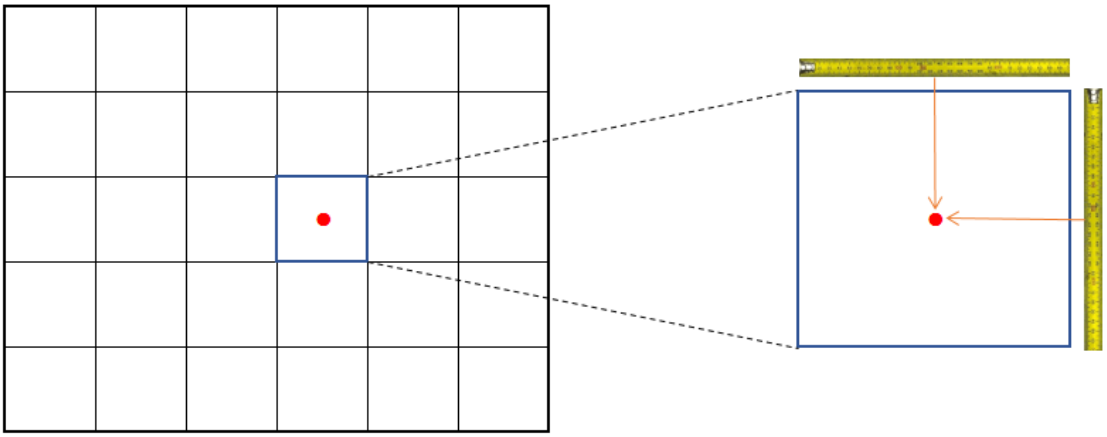
Before sampling can commence, the DU must be subdivided into a grid of equally sized cells. The number of these cells is variable, but DEC and ITRC guidance state that 30 grid cells should be considered a minimum for a representative sample. The total area encapsulated by each grid cell is at the discretion of the environmental professional, but should provide sufficient resolution to capture the spatial variability of the DU. The following formula (Equation 1) may be used to estimate the dimensions of each grid cell:

Equation 1: Grid cell dimension estimation

Equation	Variable and Definition	
$X = \sqrt{\frac{A}{N}}$	X	Length and width dimensions of the resulting grid cell
	A	Total area of the DU
	N	Total number of desired grid cells

When performing the sampling, the environmental professional must systematically collect a quantity of soil of equivalent volume from each grid cell within the DU. The location of these sample points within the grid cells must be determined randomly. The recommended method by which to accomplish this is to lay a tape measure along the X and Y axis of a grid cell. A die or random number generator can then be used to ascertain random values which correspond to units on the tape measures (Exhibit 1).

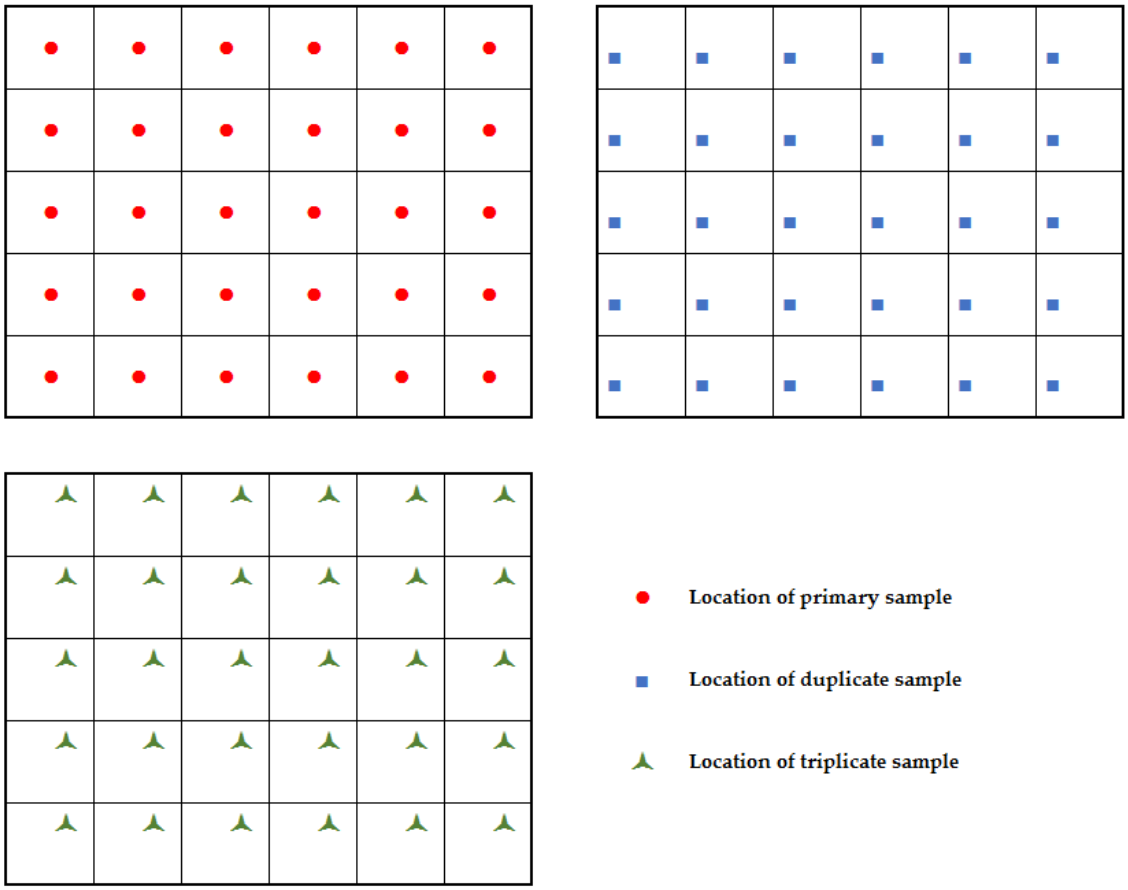
Exhibit 1: Random sample location determination



The sample location within the grid cell must be determined randomly. A die or random number generator is recommended for determining the X, Y, and Z coordinates of the sample location.

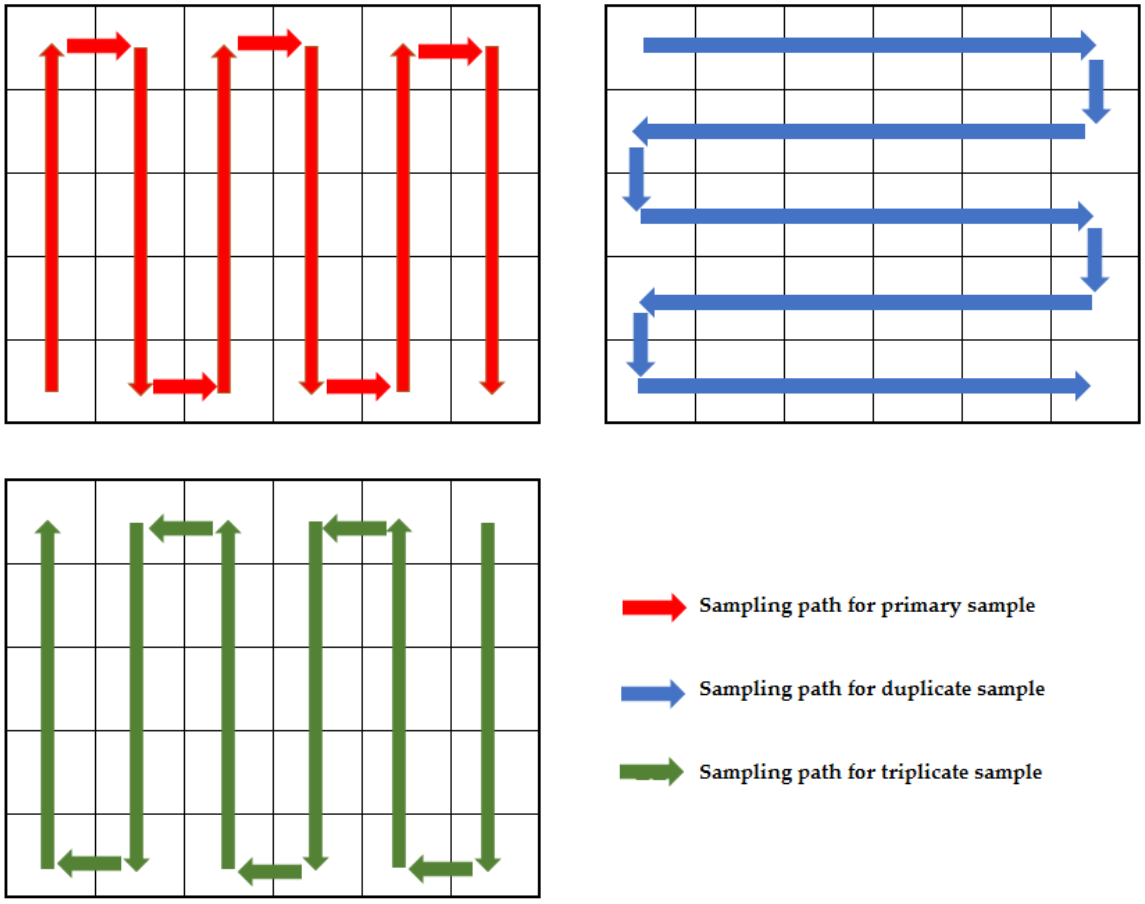
Once the random sample location has been determined within the grid cell, that same location is used when sampling all remaining grid cells within the DU. This process may be repeated for replicate samples, such that a new random location is determined for each of the three replicates (Exhibit 2).

Exhibit 2: Random sampling locations for replicate samples



The systematic order in which the grid cells are sampled should be different for each of the replicate ISM samples (Exhibit 3). While all grid cells within the DU will be sampled during each process, changing the order in which they are sampled reduces procedural bias.

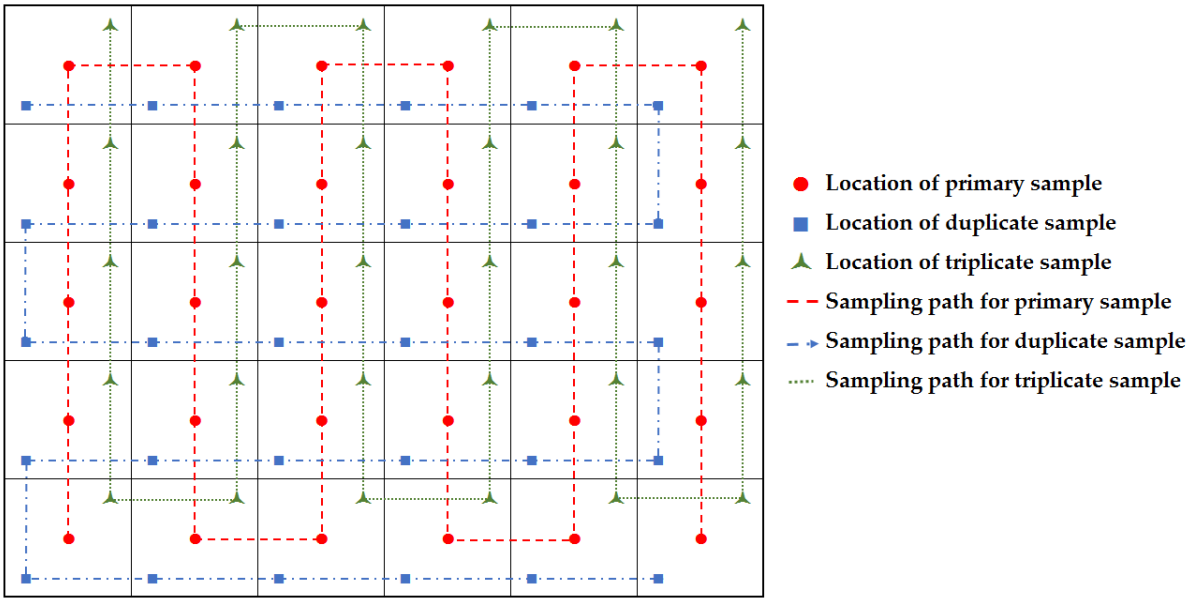
Exhibit 3: Systematic approach to sampling grid cells



The increments collected from each grid cell should be composited with the increments from all other grid cells for that replicate sample for that DU. The end result should be a single unique container for each replicate sample.

By collecting soil from unique locations within the grid cells for each of the replicate samples, the total amount of DU surface area coverage is enhanced and the ability of the method to account for spatial variability can be assessed (Exhibit 4).

Exhibit 4: DU coverage after triplicate samples are collected



The frequency of replicate sample collection is determined by the number of DUs being sampled, and through consultation with the DEC.

This method of sampling requires additional time for the environmental professional(s) to design an appropriate grid and physically establish it in the field. It is beneficial for there to be at least two personnel onsite to measure and mark out the grid and to aid with collection of the replicate samples. The benefit of this method is a more systematic and reproducible approach to sample collection.

For a stockpile with a depth greater than 2 feet, the ISM process would need to be completed for each layer. We note the ITRC document requires 1-foot layers; however, we are proposing 2 foot layers for this project.

METHOD 2: EX-SITU

The ex-situ method has the benefit of expediency but requires an excavator/backhoe operator to be onsite to facilitate sampling. The method involves shifting the stockpile horizontally and collecting ISM increments at regular intervals based on the known volume of the excavator/backhoe bucket.

To perform this method, an accurate estimate of stockpile volume is needed. The total volume of the DU is divided by the number of required increments. The result is then

divided by the bucket size of the excavator/backhoe to determine the frequency of increment collection. Randomization is not needed via this method because the material is already randomized through the scooping action of the excavator/backhoe. An example of sampling frequency is provided below.

Assumptions:

- Maximum DU volume is 750-cy
- 30 increments are required per DU and an ISM sample
- Excavator bucket size is 0.5-cy

Calculations:

$$\frac{750\ cubic\ yards}{30\ increments} = 1\ increment\ per\ 25\ cubic\ yards$$

$$\frac{25\ cubic\ yards}{0.5\ cubic\ yard\ per\ scoop} = 1\ increment\ collected\ per\ 50\ scoops$$

There are fundamental sources of error in this method due to inconsistency in scoop sizes and/or poor estimations of overall DU volume. However, when care is taken to be consistent throughout the process the analytical results can still be representative.

INCREMENT COLLECTION

When collecting increment samples, it is important to collect roughly the same quantity from each subunit of the total area/volume of the DU. Because the analytical results will represent the mean analyte concentrations for the entire DU, each grid cell must be represented equally in the composite sample. To ensure an equal volume of soil is collected from each subunit, the environmental professional should use a standardized tool, such as a Terra Core Sampler™ when collecting increments. Collecting inequal soil from one or more subunits will bias the results, giving more weight to those portions of the DU. Once collected, the increments should be deposited and homogenized in a clean bucket, large sealable bag, or other PFAS-free container for transport. We will target a bulk sample mass of one to two kilograms per ISM sample.

Analytical samples will be submitted to Eurofins/TestAmerica in West Sacramento, California for the analysis of 18 PFAS via Environmental Protection Agency Method 537M. Samples will also be submitted for analysis via synthetic precipitation leaching procedure

(SPLP). The laboratory will perform backend sample processing including passive drying, sieving, and random subsampling of the bulk sample mass.

SAMPLE REPRESENTATIVENESS AND REPORTING

Replicate samples are collected so a relative standard deviation (RSD) and 95% UCL may be calculated upon receipt of the analytical results. The RSD, represented as a percentage, is used to determine the amount of agreement between replicate results. The RSD is calculated via the following formula (Equations 2-4):

Equation 2: Relative standard deviation

Equation	Variable and Definition	
$RSD(\%) = \frac{SD}{\mu} \times 100$	RSD	Relative standard deviation
	SD	Standard deviation
	μ	Arithmetic mean of sample results for a target analyte

Where standard deviation (SD) is defined as:

Equation 3: Standard Deviation

Equation	Variable and Definition	
$SD = \sqrt{\frac{\sum \lvert x - \mu \rvert^2}{N-1}}$	SD	Standard deviation
	x	Result for a target analyte for which the SD is to be calculated
	μ	Arithmetic mean of the data set for the target analyte
	N	Number of results in the data set for the target analyte

And the arithmetic mean (μ) is defined as:

Equation 4: Arithmetic mean

Equation	Variable and Definition	
$\mu = \frac{1}{N}\sum_{i=1}^{N} x_i$	μ	Arithmetic mean of the data set for the target analyte
	N	Number of results in the data set for the target analyte
	x	Result for a target analyte for which the mean is to be calculated

DEC requires the RSD be 30% or less before the data can be considered sufficiently precise. If the RSD is greater than 30%, DEC considers the representativeness of the sample to be questionable. The RSD may be elevated differently when the detected analyte concentrations are near or below the limit of quantitation (LOQ) or relevant reporting limits (RLs). In these situations, the data must be evaluated on a case-by-case basis.

The 95% UCL is a value derived from the results of the three replicate samples for each target analyte. This value represents a statistically derived concentration for a target analyte for which there is a 95% probability that the true mean analyte concentration does not exceed within the given DU. The 95% UCL for each analyte should be compared to the applicable regulatory limits during reporting. The method by which the 95% UCL is derived is based on whether the concentrations of target analytes are assumed to be normally distributed or skewed within the soil mass. This assumption can be made by calculating the coefficient of variance (CV). The CV is calculated via the following formula (Equation 5):

Equation 5: Coefficient of Variance

Equation	Variable and Definition	
$CV = \frac{SD}{\mu}$	CV	Coefficient of variance
	SD	Standard deviation
	μ	Arithmetic mean of sample results for a target analyte

Typically, if the CV for a given analyte is found to be between 0 and 1.5, then that analyte is assumed to be normally distributed within the sample. Conversely, if the CV is between 1.5 and 3.0, the distribution of that analyte within the sample can be assumed to be skewed. A CV greater than 3.0 would imply a heavily skewed distribution.

For an analyte exhibiting a normal distribution, the 95% UCL should be calculated using the one-sided Student's t-factor. This is accomplished via the following formula (Equation 6):

Equation 6: 95% Upper Confidence Limit Using Student's t-factor

Equation	Variable and Definition	
$95\%\ UCL = \mu + \frac{(t \times SD)}{\sqrt{N}}$	μ	Arithmetic mean of the data set for the target analyte
	N	Number of results in the data set for the target analyte
	t	95% one-sided t-distribution factor (e.g. for N=3, t=2.92)
	SD	Standard deviation

For an analyte exhibiting a skewed distribution, the 95% UCL should be calculated using the Chebyshev's theorem. This is accomplished via the following formula (Equation 7):

Equation 7: 95% Upper Confidence Limit using Chebyshev's Theorem

Equation	Variable and Definition	
$95\%\ UCL = \mu + \sqrt{(1/\alpha) - 1} \times \frac{SD}{\sqrt{N}}$	μ	Arithmetic mean of the data set for the target analyte
	N	Number of results in the data set for the target analyte
	1 - α	Decision confidence level (*α = 5% or 0.05*)
	SD	Standard deviation

Assuming a Type 1 error tolerance of 5%, the expression $\sqrt{\left(\frac{1}{\alpha}\right) - 1}$ is simplified to the constant 4.36 for computational purposes.

By either method, replicate data sets that contain one or two non-detect results we will substitute the laboratory's most sensitive detection limit (normally the method detection limit [MDL] or detection limit [DL]) for the non-detect result during RSD and 95% UCL calculations.

CLOSING CONSIDERATIONS

Both methods of stockpile sampling have pros and cons. Method 1 is more scientific and does not require any accompaniment during sampling. However, an operator is needed to grade the stockpile prior to sampling and the sample collection process takes longer to complete. Grid layout and sampling greatly benefits from a second person onsite. For a single person, assume two days for replicate sample collection. One day per sample is sufficient with two people.

Method 2 has a higher margin for error and requires an operator onsite during sampling; however, this process is typically much quicker and requires only a single sampler. Assume two ISM samples per person per day, including replicates.

If you have any questions, please feel free to contact us.

Sincerely,

SHANNON & WILSON

adam Wyborny
Digitally signed by Adam Wyborny
Date: 2021.05.18 14:09:55 -08'00'

Adam Wyborny, PE
Environmental Engineer

Enc. Eurofins/TestAmerica SOP for ISM subsampling at the laboratory

Title: Incremental Sampling Methodology of Soils and Sediments

Approvals (Signature/Date):

Victoria Nihart — 02/25/2020
Victoria Nihart — Date
Department Manager

Joe Schairer — 02/25/2020
Joe Schairer — Date
Health & Safety Manager / Coordinator

Lisa Stafford — 03/25/2020
Lisa Stafford — Date
Quality Assurance Manager

Chris Williams — 02/25/2020
Chris Williams — Date
Laboratory Director

Facility Distribution No. Uncontrolled **Distributed To:** Sacramento Bids Folder

1. SCOPE AND APPLICATION

1.1. The purpose of this procedure is to obtain sub-samples from client provided samples which represent the concentration of material in the entire parent sample.

1.2. This SOP describes the procedures for laboratory staff to follow during the preparation of samples for the incremental sampling methodology (ISM) procedure. These are guidelines for the preparation and sub sampling of soil/solid/sediment samples to be analyzed for routine organic and inorganic analyses.

1.3. The incremental sampling methodology sub sampling procedures are not applicable to volatile soil samples collected on Encore® samples for Method 5035. These are discrete samples and the entire sample is used for analysis.

2. SUMMARY OF METHOD

2.1. Samples received from the field may require processing including drying, removal of extraneous material, and sieving to be performed for different analyses so that a representative concentration can be determined. An entire client sample is first processed and the sample is then sub sampled using an incremental sampling methodology approach.

2.2. Care should be taken to ensure that these subsamples are representative of the component samples and are properly prepared and stored in accordance with the appropriate method of analysis.

2.3. The basic formula to use when working with clients to select the optimal approach for other methods or other precision objectives is given in Appendix 1 to this SOP. The Appendix defines the trade off between subsample size, particle size, and the desired level of precision.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. ISM – Incremental Sampling Methodology

3.4. Dry – sample(s) require drying.

3.5. Disaggregate – The act of breaking the soil clumps into individual small particles, but keeping the small pebbles and hard crystalline particles intact for sample(s) that require grinding with mortar and pestle or ball mill.

3.6. Sieve – sample(s) require sieving (default #10 2mm sieve)

3.7. Ball Mill – sample(s) require ball mill procedure

3.8. Split – dividing a sample in half or smaller segments, but typically larger than analytical subsamples. There are several process options such as riffle splitter, rotary sectorial splitter and most commonly 1D slabcake. For Sacramento splitting has the same procedure as subsampling, therefore treat splitting as subsampling.

3.9. Subsample (also referred to as 2D slabcake) – sample(s) require subsampling to procure sample weights using the ISM.

3.10. As Received – sample(s) do not require drying

4. INTERFERENCES

4.1. If the samples are to be analyzed for metals testing, check the login/method/sample comments and with the PM to find out if the brass sieves (Teflon® screens are an alternative) and the aluminum foil (butcher paper is an alternative) used to spread samples out for drying are going to be problematic to the client. Ball mill processing risks contaminating metals testing with Fe, Al, and Sn. Brass sieves risks contaminating metals testing with Cu and Zn.

4.2. Some clients require the use of Teflon® coated spatulas instead of the regular sterile sample scoops (polystyrene) due to the possibility of contamination. These Teflon® spatulas should be washed and decontaminated as per method requirements of the tests in question.

4.3. Interferences can occur when using scoops or spatulas. All scoops or spatulas shall be used for only one sample and then disposed, or thoroughly cleaned between samples. Material that may be acceptable for one analysis may cause contamination for another analysis. All plastic should be avoided if organic parameters are requested.

4.4. On rare occasions a client may ask for ISM processing for volatile samples. Please confirm with project manager that the request is correct. If so, then follow these guidelines. Volatile analytes may be lost during subsampling from non-Encore containers. Subsampling for volatile analyses should be done from a previously unopened container to the end of tube (where possible), and subsampling should be done as quickly as possible to avoid analyte loss.

4.5. Volatile and light semi-volatile analytes may be lost during the sample drying and grinding procedure. Consult the appropriate analytical SOP and QAS for guidance on the required drying and grinding procedure for the samples.

4.6. When the procedure is used to process samples for PFC samples, the following precautions must me used to prevent contamination or inferences. No Teflon® or Teflon® coated materials may be used. Samples should be dried on non-coated paper

and sieved using brass sieves. Subsampling must be performed using scoops or spatulas made of Teflon®- free materials (metal, HDPE, polystyrene or wood). Store subsamples in plastic jars that do not have Teflon lined lids.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Extensive homogenization, subsampling, and/or compositing of soil/solid/waste or liquid samples presents an extreme risk of repetitive motion injuries for the individual performing the operation. No single employee will homogenize, subsample, or composite these types of samples for longer than one hour continuously without taking a five-minute break away from this type of work and stretching his/her hands, wrists and arms. If the manager/supervisor and the employee involved identify at the start of the process that the work will take longer than one hour, the employee should take mini-breaks of 2-3 minutes every 25-30 minutes. If there is extensive homogenization, subsampling, and/or compositing that must be performed, or if it is extremely time sensitive, managers/supervisors must assign additional personnel to the effort, or rotate different staff members through the job in order to prevent injury to any employee.

5.1.2. The use of the Ball Mill during this process presents an extreme risk of damage to the ears/hearing loss. Ear muffs must be worn by all persons in the room when the ball mill is in operation, and the hallway door must be closed when the ball mill is in operation. It is recommended that persons in the room wear disposable ear plugs under the ear muffs.

5.1.3. The opening and closing process for the paint cans used during Ball Mill operations present serious risk for repetitive motion injuries, impact injuries and lacerations. Following the instructions in paragraph 5.1.1 will reduce the risk of repetitive motion injuries. When adding soil/solid sample material to the paint can, ensure that no sample material falls into the groove in the top of the can that the lid will be fitted into. This will ensure that sample material does not fly out of the groove when the lid is seated, and also ensures that the lid seats easily. Use only the rubber mallet to seat the lid into the groove, not your hand or any type of metal hammer, and ensure that your hand is well

away from the impact point of the mallet. When opening paint cans after the ball mill operation is complete, do not use screwdrivers, spatulas, knives or any tools other than the commercial paint can openers that have been provided. Do not keep the hand that is not holding the paint can opener over or right next to the opener while in use in case it slips.

5.1.4. If sediment/soil samples have been frozen in glass jars, the freezing process may have cracked the jars when the sample expanded while freezing. After the samples have thawed, wear cut protective gloves while handling the jars until it can be confirmed that they have not cracked.

5.1.5. Any alternative procedures requested by a client must be reviewed by EH&S before they are put into practice.

5.1.6. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide sufficient protection when handling closed sample containers and most typical samples. Unusual or heavily contaminated samples must be evaluated to determine if there are any hazards for which a particular type of glove will not be appropriate.

5.1.7. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred, sub sampled, and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.2. Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
(1) Always add acid to water to prevent violent reactions.			
(2) Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Shallow metal trays, approximately 18" wide by 26" long.

6.2. Inert tray lining – depending on the analytes requested, either of these two materials may be used to line the trays in 6.1:

6.2.1. Aluminum foil (not used if tests for metals are requested).

Note: *As food-grade aluminum foil is often coated to alleviate sticking, it is not recommended when perfluorinated compounds are requested.*

6.2.2. Unbleached Kraft (butcher) paper

To line the trays, create a "weigh boat" of either foil or paper, approximately 18 inches by 26 inches with a small lip around the perimeter. Place this on the tray prior to spreading sample material on it.

6.3. Sieves, various sizes, including 2 mm (#10 sieve).

6.3.1. Sieves may be brass or patches of Teflon screening.

Note: *When perfluorinated compounds are requested, use the brass sieves only.*

6.4. Teflon® coated spatulas – do not use if perfluorinated compounds are requested.

6.5. Sterile sample spoons (that are not rounded on the sampling side), made of polystyrene. Fisher part # 14-375-254 or similar.

6.6. Tongue depressors

6.7. Analytical balance.

6.8. Mortar and pestle (manual and automated).

6.9. Sample containers, various sizes, glass and poly.

6.10. Fume hood.

6.11. Rubber mallets.

6.12. Commercial paint can opener.

Note: *Some samples such as biological tissues and pulp and paper products may require pre-preparation before subsampling or compositing. See the appropriate SOP for matrix specific procedures.*

6.13. Ball Mill (US Stoneware or equivalent) – for the disaggregation of solid samples.

6.13.1. 1 gallon and ½ gallon new paint cans are used (Manufactured by CL Smith). ½ gallon part number 12-2350ALL, ½ gallon lid 12-2400ALL.01. 1 gallon part number 12-1427STD.01 (includes lid). Paint cans are to be used one time then discarded.

6.13.2. The ball mill grinding material is manufactured by E.R. Advance Ceramics, part number BRUN125-90 (90% Burundum Grinding Media 1-1/4" x 1-1/4"). Grinding material can be reused after the cleaning procedure described in section 11.7.1.

7. REAGENTS AND STANDARDS

7.1. Acetonitrile, HPLC grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

All component, subsamples, and composites will be stored in compliance with the analytical method under which they will be analyzed.

9. QUALITY CONTROL

Samples used for Matrix Spike and Matrix Spike Duplicates (MS/MSDs) and sample duplicate and sample triplicates (DU/TRL) should be homogenized and sub sampled using the same procedure incremental sampling methodology as the parent sample(s).

10. CALIBRATION

10.1. Balances used for subsampling or compositing for analysis and preparation should be calibrated as per SOP WS-QA-0041.

10.2. Balances used for non-analytical subsampling, i.e., for trans-shipment, do not require calibration as the weight is a rough value only.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. This method is dependent on the client project provided Data Quality Objectives. Depending on the nature of the project, samples may need to be dried, extraneous material may need to be removed and replicates may need to be run per sample or per batch of samples. It is important that the analyst confirm with the Project Manager prior to performing this procedure. Any project specific changes or modifications to the procedure should be noted in the form of a client specific amendment to the SOP or in the Quality Assurance Summary (QAS). The procedures documented below incorporate the commonly performed procedure. There are two primary procedures to consider, sample prep and subsampling.

11.3. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

Note: *Drying, sieving or subsampling for volatile analyses may lead to loss of analytes or contamination from common laboratory solvents. Any incremental sampling methodology procedures for volatiles should be performed in a solvent-free area and the mixing should be*

minimized to reduce the loss of volatile analytes. The potential for loss of volatile analytes should be discussed with the client before initiation of the program.

11.4. Determine the required Incremental Sampling Methodology using the method code (see definitions above). The order in which the steps are stated are the order in which the steps need to be performed. For samples that have multiple requested methods, perform the method with the less rigorous steps first followed by the ISM with the more rigorous steps without repeating steps that were already performed. For example, ISM_R_SS (wet) is done before ISM_DD_SI_SS (dry). Anytime wet ISM is requested before drying, make sure that % moisture is also subsampled.

11.4.1. ISM_DD_SI_SS – Dry, Disaggregate, Sieve, 2D slabcake, Subsample

11.4.2. ISM_DI_SI_SS – As received, Disaggregate, Sieve, 2D slabcake, Subsample

11.4.3. ISM_DD_SP – Dry, Disaggregate, Split

11.4.4. ISM_DI_SP – As Received, Disaggregate, Split

11.4.5. ISM_CUSTOM and ISM_CUST_WS – Custom ISM procedure. Consult the PM. Default to ISM_DD_SI_SS if there are no method notes.

11.4.6. ISM_R_SS – As Received, 2D slabcake, subsample

Before the first ISM step, weigh the entire sample. If the sample weighs more than 1.1kg, notify the project manager of the weights.

Note: *For QSM samples, record the initial weight of the sample on the "ISM Drying and Additional Information" form, QA-526B (Appendix 6).*

11.5. For samples that require Custom ISM

11.5.1. For Custom ISM samples, consult the comment section of the backlog, project documents, and if needed discuss with the project manager for specific instructions.

11.5.2. For Custom ISM- Homogenization

11.5.2.1. Do Not Dry Samples

11.5.2.2. Homogenize/Disaggregate the entire container using ball mill or mortar and pestle.

Sample aliquots are taken from the homogenized sample volume for each analysis. 2D slabcake ISM is not required for this procedure.

11.6. For samples that require drying

11.6.1. Spread the entire sample evenly on a tray lined with aluminum foil or butcher paper that is inert material and is free from any analytes of interest or

interferences.

11.6.1.1. For samples that require 6010/6020/7471 aluminum foil cannot be used. For all analysis, butcher paper can be used.

11.6.1.2. For samples that require VOAs and % moisture, ensure the % moisture for VOA is done 'As Received' since VOA samples are preserved in MeOH.

11.6.1.3. Samples that are extremely wet or have considerable mass may required drying on multiple lined trays and re-combined later. Make sure to label trays to indicate there are multiple ones, e.g. 1 of 2 and 2 of 2.

11.6.1.4. For very wet samples, double or triple line the tray with butcher paper and/or add a layer of aluminum foil under the paper to ensure that water does not seep through to the tray.

WARNING: If sediment/soil samples have been frozen in glass jars, the freezing process may have cracked the jars when the sample expanded while freezing. Wear cut protective gloves while handling the jars until it can be confirmed that they have not cracked. If the jar is cracked/broken and not usable, do not put all the contents in a plastic bag. Instead move the material to a new jar, relabel it, and properly discard the broken glass.

11.6.2. Samples should be placed in the ISM drying towers which allows for proper ventilation, and allowed to air-dry at room temperature with the tower fans turned on. For very wet samples, it may be necessary to place the sample in a fume hood with the sash lowered to improve airflow over the sample. The sample should be dried until visibly dry and if uncertain a % moisture aliquot can be taken using the ISM to ensure dryness. Ensure to record the start and end date, time, and analyst initial in the QA-562 document (Appendix 4).

11.6.2.1. For QSM samples, the samples need to be dried till they are a consistent weight at room temperature (record the room temperature on form QA562-B). Consistent weight is determined by weighing an approximately 10 g aliquot of the sample twice with 2 hours between measurements. When taking the weight of the 10 g aliquot, do not include the weight of the aluminum weight boat. If the weight difference (Equation 1) is ≤5%, the sample is considered dry. If the difference is greater than 5%, the sample is dried for another two hours and this is repeated till the sample weight is consistent. The consistent weight measure is documented on form QA-562-B.

Note: *After drying is complete, remember to turn off the fans in the ISM drying tower.*

11.7. For samples that require disaggregating and sieving, splitting/subsampling.

11.7.1. Adequately decontaminate necessary equipment (sieves, pestles, mortars, grinding material) by brushing and washing with soap and water and rinsing with the appropriate solvent or reagent as outlined in the analytical procedure. Acetonitrile is an appropriate solvent for all analysis.

11.7.2. For samples requiring disaggregation/sieving

11.7.2.1. Break up any soil aggregate material with a clean object such as a mortar and pestle or ball mill (Section 11.8). Employ de-aggregation techniques if needed. Occasionally using a sterile scoop or tongue depressor may be necessary if the sample is sticky and a mortar and pestle is not effective. Ensure to disaggregate the entire sample. This may require repeating this step once with the materials that would not pass through a sieve.

11.7.2.2. Carefully sieve the sample using a 2mm sieve (#10), or the appropriate size as designated in the method method/login/sample comments. In some cases a smaller mesh size sieve may need to be used following 2mm sieve. Clients may ask to weigh the individual portions for particle size analysis.

Note: *For samples that require 6010/6020/7471 brass sieves may* ***not*** *be used. For samples that require PFC analysis (perfluorinated compounds) Teflon® sieves may* ***not*** *be used. If both metals and PFC analysis is required on the same sample, brass sieves may be used if copper and zinc are not analytes of interest. This variance needs to be documented in a non-conformance memo.*

11.7.2.3. Transfer the materials with particle size greater than the designated particle size (sieve mesh) back into the original sample container, or dispose of as outlined in Section 15 if the client does not want to save the material. If the unsieveable material is collected in a secondary container, ensure it gets properly labeled by sample control and returned to a storage location for disposal at a further date.

11.7.2.4. For QSM samples, record the total weight of the dried and sieved material (before ISM aliquots have been taken) and the unsieved waste material and document it on form QA-562B.

11.7.3. For samples requiring subsampling/splitting

11.7.3.1. Subsample in duplicate only if the aliquot is to be sent out to a sister lab or dried after wet subsampling will later be required. Ensure there is adequate volume left after subsampling for methods that will follow. If not, notify the project manager. Omission of the duplicate and/or matrix spike/matrix spike duplicate may be required.

11.7.3.2. Evenly distribute the sample into a weigh boat made out of butcher paper or aluminum foil (do not use aluminum foil if 6010/6020/7471 or perfluorinated compounds are requested). The sample layer should have a depth of approximately one-half inch or less to allow for sampling throughout the entire depth of the sample layer. If the depth is more the approximately one-half inch, split the sample evenly across a minimum amount of weigh boats.

11.7.3.3. Perform the subsampling by forming an imaginary 6x5 grid to visualize 30 squares in the sampling tray. Take one scoop using a sterile sample spoon (client may request use of Teflon® coated spatulas) from each square ensuring the aliquot is from the top to the bottom of the soil. (An unaltered tongue depressor may not be used because the rounded tip will not collect evenly from the top to the bottom of the sample). Each aliquot should contain approximately $1/30^{th}$ of the desired target mass. Collect the aliquot fractions in the appropriate containers. See Summary Sheet (Appendix 5) for targeted mass, appropriate containers for various analyses, and additional notes for each analysis. Ensure to include a MS/MSD for each preparation batch for each test, a sample duplicate and sample triplicate for 8330B, and a minimum of one MS/MSD pair per 10 samples for 6020 and 6010. For QSM samples, a duplicate and triplicate needs to be subsampled for every 20 samples.

11.7.3.4. In some cases with a smaller sample size, a 6x5 grid is not possible. Create a similar grid and subsample 30 times as evenly as possible.

11.7.3.5. If the final amount is less than the targeted amount, subsample from random locations until the targeted amount is obtained.

11.7.3.6. If the final amount is greater than the targeted amount, pour the entire sample back and re-sample.

11.7.3.7. Ensure to record the initial, balance ID, and mass in the QA 562 (Appendix 4) document.

11.7.3.8. Store the sub samples in the proper location for the test being conducted until they are ready for further preparation and document this storage location on the QA-562 form.

11.7.3.9. Place the unused dried and sieved material in a zippered bag that has been labeled properly. This bag can then be placed inside the original jar (with the unsieved waste) or bagged together with the original zippered bag the sample came in.

11.8. For samples that require ball mill (disaggregation)

11.8.1. Start with a dried sample, a new paint can (labeled with sample ID sticker and permanent marker), and clean grinding material.

11.8.1.1. Paint cans are ready to use from the manufacturer and are used only one time then discarded.

WARNING: When adding soil/solid sample material to the paint can, ensure that no sample material falls into the groove in the top of the can that the lid will be fitted into. Use only the rubber mallet to seat the lid into the groove, and ensure that your hand is well away from the impact point of the mallet. When opening paint cans after the ball mill operation is complete, only use the the commercial paint can openers that have been provided.

WARNING: The use of the Ball Mill during this process presents an extreme risk of damage to the ears/hearing loss. Ear muffs must be worn be all persons in the roof when the Ball Mill is in operation, and the hallway door must be closed while the mill is in operation. It is recommended that persons in the room wear disposable ear plugs under the ear muffs.

11.8.2. Load the sample in the paint can. Half gallon cans can not be more than 2/3rd full and 1 gallon cans can not be more than 3/4th full. If needed, use two or more cans for one sample.

11.8.3. Place 3-4 clean grinding material stones in the half gallon cans and 4-5 in the 1 gallon can. Seal the lid with the rubber mallet and ensure that it is fully closed.

11.8.4. Load the cans onto the ball mill and start the rotation. Document the start time on the ISM sheet (QA-562).

11.8.5. Rotate the sample for a minimum of 2 hours. If the samples are not rotating properly on the ball mill, they can be taped together with duct tape to ensure proper rotation. Make sure to tape the cans with the lids facing inward.

11.8.6. After removing the sample from the ball mill, record the time off in the ISM sheet (QA-562). Open the paint can in a hood and visually inspect the sample to ensure that it has been adequately disaggregated. If the sample is not adequately disaggregated after two hours, the sample can continue to rotate for up to 8 hours.

11.8.7. Transfer the material to an appropriate container or proceed with sieving and subsampling the sample (section 11.7.2 to 11.7.3).

11.9. Batch all samples

11.9.1. Using the method codes above, batch all samples into the appropriate method. No raw data needs to be entered. Ensure the batch start and end time is from the time the sample originally was started to the final ISM and ensure the samples are set to the batch time.

11.9.2. Scan all documents associated with each batch into the extractions folder under PM group. Rename file "zbatchnumber" and attach to the document tab in the batch in TALs.

11.9.3. Print out labels or the analytical batch paperwork. Once the ISM process is complete, email the extraction department the storage location of the ISM aliquots and the ISM batch number where they can find the TALs ID and weights.

11.10. All jars created with ISM must be labeled with a TALS label prior to being placed in the refrigerator. For sendouts, use the color code system found in CA-T-WI-011 (ISM Workshare Communication). Place sendout aliquots on shelf W14B in the walk-in fridge and notify sample control that they are ready.

12. CALCULATIONS/DATA REDUCTION

12.1. Consistent Weight Calculation

Equation 1: $$\frac{Initial\ Weight\ (g) - Dried\ Weight\ (g)}{Initital\ Weight\ (g)} * 100$$

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

14. POLLUTION CONTROL

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste

disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Contaminated disposable materials such as plastic vials, pipettes, empty sample containers, unused/excess sample matrix, and disposable spatulas that have come in contact with soil samples. Dump this solid waste into an orange contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the incinerate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Contaminated disposable materials that have not come in contact with soil samples. Dump this solid waste into a yellow contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the landfill steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. STL White Paper "Representative Sub-sampling Techniques for Inorganic Analytes", February 23, 2004.

16.2. "Guidance for Obtaining Representative Laboratory Analytical Sub samples from Particulate Laboratory Samples," USEPA, November 2003.

16.3. "Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities" ASTM D 6323-98 (Reapproved 2003)

16.4. EPA SW-846 Method 8330B. Nitroaromatics, Nitramines, and Nitrate esters By High performance Liquid Chromatography (HPLC). Revision 2, October 2006.

16.5. "Incremental Sampling Methodology [ISM] Program Minimum Standards", 05/9/2012 Revision 2

17. METHOD MODIFICATIONS

17.1. There are no deviations from the method unless otherwise specified by the QAS and checked with the group/team lead.

18. ATTACHMENTS

18.1. Attachment 1 – Consideration of Fundamental Error in Selecting ISM Options

18.2. Attachment 2 – Incremental Sampling Methodology

18.3. Attachment 3 – Sacramento Incremental Methodology Summary

18.4. Attachment 4 – ISM Drying and Additional Information for QSM Samples

19. REVISION HISTORY

19.1. WS-QA-0028, Revision 4.8, Effective 03/30/2020

19.1.1. Section 4.1 added, "Ball mill processing risks contaminating metals testing with Fe, Al, and Sn. Brass sieves risks contaminating metals testing with Cu and Zn."

19.1.2. Section 5.2 added acetonitrile to table.

19.1.3. Sections 6.1 and 6.2.2 revised, "30 inches" to "26 inches".

19.1.4. Section 6.5 added, "Fisher part # 14-375-254 or similar."

19.1.5. Added Section 7.1, "Acetonitrile, HPLC grade".

19.1.6. Section 11.6.1.3 added, "Make sure to label trays to indicate there are multiple ones, e.g. 1 of 2 and 2 of 2."

19.1.7. Added Section 11.6.1.4, "For very wet samples, double or triple line the tray with butcher paper and/or add a layer of Aluminum foil under the paper to ensure that water does not seep through to the tray."

19.1.8. Section 11.6.1.4 note added , "If the jar is cracked/broken and not usable, do not put all the contents in a plastic bag. Instead move the material to a new jar, relabel it, and properly discard the broken glass."

19.1.9. Section 11.6.2 revised, "on bakers racks or another location" to "in the ISM drying tower" and added, "with the tower fans turned on".

19.1.10. Section 11.6.2.1 added, ""When taking the weight of the 10 g aliquot, do not include the weight of the aluminum weight boat."

19.1.11. Section 11.6.2.1 added note, "After drying is complete, remember to turn off the fans in the ISM drying tower."

19.1.12. Section 11.7.2.2 note added, "If both metals and PFC analysis is required on the same sample, brass sieves may be used if copper and zinc are not analytes of interest. This variance needs to be documented in a non-conformance memo."

19.1.13. Section 11.7.2.3 revised, "into a second container" to "back into the original sample container".

19.1.14. Section 11.7.2.4 added, "(before ISM aliquots have been taken)."

19.1.15. Section 11.7.3.1 added, "and/or matrix spike/matrix spike duplicate".

19.1.16. Section 11.7.3.8 added, "and document this storage location on the QA-562 form."

19.1.17. Added Section 11.7.3.9, "Place the unused dried and sieved material in a zippered bag that has been labeled properly. This bag can then be placed inside the original jar (with the unsieved waste) or bagged together with the original zippered bag the sample came in."

19.1.18. Section 11.10 added, "Place sendout aliquots on shelf W14B in the walk-in fridge and notify sample control that they are ready."

19.1.19. Added Section 12.1, "Consistent Weight Calculation".

19.1.20. Added Section 15.1, "Contaminated disposable materials such as plastic vials, pipettes, empty sample containers, unused/excess sample matrix, and disposable spatulas that have come in contact with soil samples. Dump this solid waste into an orange contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the incinerate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment."

19.1.21. Section 15.2 revised to, "Contaminated disposable materials that have not come in contact with soil samples. Dump this solid waste into a yellow contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the landfill steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment."

19.1.22. Updated Attachment 2 with current version of form.

19.1.23. Updated Attachment 3 to reflect current laboratory procedures.

19.1.24. Updated Attachment 4 with current version of form.

19.1.25. Editorial changes.

19.2. WS-QA-0028, Revision 4.7, Effective 03/25/2019

19.2.1. Removed Section 3.7, "Puck Mill – sample(s) require puck mill procedure".

19.2.2. Section 5.1.2 removed references to "Pulverizing Mill".

19.2.3. Removed Section 5.1.3, "The heavy weight of the puck and bowl (ring) used in the process presents an extreme risk of injury due to the number of times they must be moved back and forth between the mill, the hood and the sink, and body position and exertion while cleaning them in the sink. Ensure that associates assigned this task use proper lifting procedures, and take sufficient breaks during the process to allow back, neck and shoulder/arm muscles to recover. If the work being done will take longer than one hour, a 2-3 minute stretch break shall be taken every 25-30 minutes. Managers/supervisors will strongly consider rotating this task between multiple associate to dissipate the

impact on any single associate. This is especially significant if work may extend over multiple days."

19.2.4. Removed Section 6.13, "Pulverizing Mill (ring and puck), ESSA model LM2-P or equivalent - for the grinding of soils per method 8330B" and its associated subsections.

19.2.5. Section 11.4 removed, "if ISM_DD_SI_SS and ISM_DD_SI_PM_SS are both requested on a sample, first dry, then disaggregate, sieve, split/subsample, then puck mill the remainder and split/subsample. For another example,"

19.2.6. Removed Section 11.4.2, "ISM_DD_SI_PM_SS – Dry, Disaggregate, Sieve, Puck Mill, 2D slabcake, Subsample".

19.2.7. Removed Section 11.4.4, "ISM_DI_SI_PM_SS – As received, Disaggregate, Sieve, Puck Mill, 2D slabcake, Subsample".

19.2.8. Removed Section 11.4.6, "ISM_DR_PM_SP – Dry, Puck Mill, Split".

19.2.9. Removed Section 11.4.8, "ISM_PM_SP = As Received, Puck Mill, Split".

19.2.10. Section 11.7.3.7 removed, "For 8330B after puckmill, record the same data in 8330B Compositing/Incremental Sampling Methodology **(QA-702)** (Appendix 2)."

19.2.11. Removed Section 11.9, "For samples that require puck mill" and its associated subsections.

19.2.12. Section 11.9.1 removed, "For samples with puck mill, ensure to include the grinding blank composite sample(s) and grinding LCS. Use 'GB' as the sample ID for TALs and in the sample comments include which composite it is. For the grinding LCS, use "LCS" as the sample ID."

19.2.13. Section 11.9.3 revised to, Print out labels or the analytical batch paperwork. Once the ISM process is complete, email the extraction departments the storage location of the ISM aliquots and the ISM batch number where they can find the TALs ID and weights."

19.2.14. Removed Attachment 2, "8330B Compositing/Incremental Sampling Methodology".

19.2.15. Removed Appendix 3, "Puck Mill".

19.2.16. Updated Attachment 5 to current version of form.

19.2.17. Removed revision history prior to 07/15/2016. It can be found in previous versions of this SOP.

19.2.18. Editorial changes.

19.3. WS-QA-0028, Revision 4.6, Effective 02/09/2018

19.3.1. Added Section 3.8, "Ball Mill – sample(s) require ball mill procedure".

19.3.2. Added Section 5.1.4, "The opening and closing process for the paint cans used during Ball Mill operations present serious risk for repetitive motion injuries, impact injuries and lacerations. Following the instructions in paragraph 5.1.1 will reduce the risk of repetitive motion injuries. When adding soil/solid sample material to the paint can, ensure that no sample material falls into the groove in the top of the can that the lid will be fitted into. This will ensure that sample material does not fly out of the groove when the lid is seated, and also ensures that the lid seats easily. Use only the rubber mallet to seat the lid into the groove, not your hand or any type of metal hammer, and ensure that your hand is well away from the impact point of the mallet. When opening paint cans after the ball mill operation is complete, do not use screwdrivers, spatulas, knives or any tools other than the commercial paint can openers that have been provided. Do not keep the hand that is not holding the paint can opener over or right next to the opener while in use in case it slips."

19.3.3. Added Section 6.12, "Ball Mill (US Stoneware or equivalent) – for the disaggregation of solid samples."

19.3.4. Added Section 6.12.1, "1 gallon and ½ gallon new paint cans are used (Manufactured by CL Smith). ½ gallon part number 12-2350ALL, ½ gallon lid 12-2400ALL.01. 1 gallon part number 12-1427STD.01 (includes lid). Paint cans are to be used one time then discarded."

19.3.5. Added Section 6.12.2, "The ball mill grinding material is manufactured by E.R. Advance Ceramics, part number BRUN125-90 (90% Burundum Grinding Media 1-1/4" x 1-1/4"). Grinding material can be reused after the cleaning procedure described in section 11.7.1."

19.3.6. Added Note after Section 11.4.10, "For QSM samples, record the initial weight of the sample on the "ISM Drying and Additional Information" form, QA-562B (Appendix 6)."

19.3.7. Added Section 11.6.2.1, "For QSM samples, the samples need to be dried till they are a consistent weight at room temperature (record the room temperature on form QA562-B). Consistent weight is determined by weighing an approximately 10g aliquot of the sample twice with 2 hours between measurements. If the weight difference is ≤5%, the sample is considered dry. If the difference is greater than 5%, the sample is dried for another two hours and this is repeated till the sample weight is consistent. The consistent weight measure is documented on form QA-562-B."

19.3.8. Added Section 11.7.2.4, "For QSM samples, record the total weight of the dryied and sieved material and the unsieved waste material and document it

on form QA-562B."

19.3.9. Added to Section 11.7.3.3, "For QSM samples, a duplicate and triplicate needs to be subsampled for every 20 samples."

19.3.10. Added Section 11.8, "For samples that require ball mill (disaggregation)", and subsections 11.8.1 through 11.8.7, which describe the current procedure for samples requiring ball mill disaggregation.

19.3.11. After Section 11.8.1 added warning, "**WARNING: When adding soil/solid sample material to the paint can, ensure that no sample material falls into the groove in the top of the can that the lid will be fitted into. Use only the rubber mallet to seat the lid into the groove, and ensure that your hand is well away from the impact point of the mallet. When opening paint cans after the ball mill operation is complete, only use the the commercial paint can openers that have been provided.**"

19.3.12. After Section 11.8.1 added warning, "**WARNING: The use of the Ball Mill during this process presents an extreme risk of damage to the ears/hearing loss. Ear muffs must be worn be all persons in the roof when the Ball Mill is in operation, and the hallway door must be closed while the mill is in operation. It is recommended that persons in the room wear disposable ear plugs under the ear muffs.**"

19.3.13. Added Section 11.9.6.3, "For QSM samples, a grinding LCS is required for each method. Please consult with the project manager or client if a Standard Reference Material (SRM) is not commercially available for the method requested."

19.3.14. Revision history prior to 2015 has been removed, it is available in previous revisions of this SOP.

19.3.15. Removed 537/537 modified, 1311 (TCLP), 1312 (TCLP), 8081 (3546), and 8082 (3546) from Appendix 5 table.

19.3.16. Added Appendix 6, "ISM Drying and Additional Information for QSM Samples".

19.3.17. Editorial changes.

19.4. WS-QA-0028, Revision 4.5, Effective 03/02/2017

19.4.1. Section 5.1.2, added the following: "The heavy weight of the puck and bowl (ring) used in the process presents an extreme risk of injury due to the number of times they must be moved back and forth between the mill, the hood and the sink, and body position and exertion while cleaning them in the sink. Ensure that associates assigned this task use proper lifting procedures, and take sufficient breaks during the process to allow back, neck and shoulder/arm

muscles to recover. If the work being done will take longer than one hour, a 2-3 minute stretch break shall be taken every 25-30 minutes. Managers/supervisors will strongly consider rotating this task between multiple associate to dissipate the impact on any single associate. This is especially significant if work may extend over multiple days."

19.4.2. Section 11.4, added the following: "For another example, ISM_R_SS (wet) is done before ISM_DD_SI_SS (dry). Anytime wet ISM is requested before drying, make sure that % moisture is also subsampled."

19.4.3. Added Section 11.4.10 to read "ISM_R_SS = As Received, 2D slabcake, subsample."

19.4.4. Added Section 11.5, titled "For samples that require Custom ISM," including sections 11.5.1 through 11.5.2.

19.4.5. Section 11.7.3.1, amended to read: "Subsample in duplicate only if the aliquot is to be sent out to a sister lab, dried after wet subsampling or Puck Mill will later be required."

19.4.6. Section 11.7.3.1, added the following: "Ensure there is adequate volume left after subsampling for methods that will follow. If not, notify the project manager. Omission of the duplicate may be required."

19.4.7. Section 11.8.1, added the following after the section: "**WARNING: The heavy weight of the puck and bowl (ring) used in the process presents an extreme risk of injury due to the number of times they must be moved back and forth between the mill, the hood and the sink, and body position and exertion while cleaning them in the sink. Ensure that associates assigned this task use proper lifting procedures, and take sufficient breaks during the process to allow back, neck and shoulder/arm muscles to recover. If the work being done will take longer than one hour, a 2-3 minute stretch break shall be taken every 25-30 minutes. Managers/supervisors will strongly consider rotating this task between multiple associates to dissipate the impact on any single associate. This is especially significant if work may extend over multiple days."**

19.4.8. Section 11.9.2, removed the following: "For level 3 and level 4 projects attach the document to the raw or prep data for an analysis requested. Attach the batch to TALS and the deliverable." Added the following: "Rename file 'zbatchnumber' and attach to the document tab in the batch in TALS."

19.4.9. Section 11.10, added the following: "For sendouts, use the color code system found in CA-T-WI-011 (ISM Workshare Communication)."

19.4.10. Appendix 2, uploaded version 1.

19.4.11. Appendix 4, uploaded version 4.

19.4.12. Appendix 5, for test 8081 and 8082 added "(3550)"; for 8081, 8082, 8270, and TPH, AK102/AK103, and 8270 changed "French Square" to "Clear tall (8 oz.)."

19.4.13. Appendix 5, added 8081 (3546) and 8082 (3546).

19.4.14. Editorial changes.

19.5. WS-QA-0028, Revision 4.4, Effective 07/15/2016

19.5.1. Appendix 5 – Added 1311 (TCLP) and 1312 (SPLP) methods, container types, and amounts to Table.

19.5.2. Editorial changes.

19.6. WS-QA-0028, Revision 4.3, Effective 07/06/2016

19.6.1. Amended Appendix 5 to include Method 537 (PFCs) with note to avoid use of Teflon.

19.6.2. Editorial changes.

Attachment 1
Consideration of Fundamental Error in Selecting ISM Options

The following formula given in ASTM D-6323 was used to produce the table that follows.

$$S^2 = 18 * f * e * d^3 / M_s$$

where,

S^2 = the relative variance of the contaminant concentration due to the fundamental error
f = shape factor, a dimensionless number, a value of 0.5 can be taken as typical (Pierre Gy, 1982)
e = the population's average density (g/cm^3). For this table a typical soil density of 2.5 g/cm^3 was used.
d = the diameter of the largest particle in centimeters, and
M_s = the mass of the sample in grams

Sample Mass and Maximum Particle Size to Achieve a Desired RSD

Subsample Mass (g)	Sieve Size (US Standard Mesh)	At 5% RSD Max Size (cm)	At 10% RSD Max Size (cm)	At 15% RSD Max Size (cm)
0.1	35	0.02	0.04	0.05
1	18	0.05	0.08	0.10
2	13	0.06	0.10	0.13
5	12	0.08	0.13	0.17
10	10	0.10	0.16	0.22
30	7	0.15	0.24	0.31
50	6	0.18	0.28	0.37
100	5	0.22	0.35	0.46

Attachment 2 (QA-562)
Incremental Sampling Methodology

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Sacramento Incremental Sampling Methodology

Page __ of __Batch(es)_____

Aliquot Storage Location_____

Wet ISM_____ Dry ISM_____

DoD Y / N PFC Y / N

Sample ID		Initial Weight (as received)	Date/Time/ Initials Sample Drying Started	Date/Time/ Initials Sample Drying Ended	Date/ Initial of ISM/ Weighing	Balance ID	Sieve Size	Project Specific Data (Method and Required Sample Mass in Header) Record Final Mass of Each Requested Test. Primary on top left, backup on bottom right.								
								8015	8081	8082	PAH SIM	6010	7471	%Moist.		
Login #	Smp #							30-31g	30-31g 15-16g	30-31g 15-16g	10-11g	10-10.2g	10-10.2g	11 ±6g		

Comments: Subsample(s) created using the Incremental Sampling Methodology (ISM) _____

Particle Size: C = Coarse = 10 mesh (2mm)
F= Fine = 30 mesh (0.6 mm)

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Attachment 3
Sacramento Incremental Methodology Summary

Test	Container	Weight	Notes
8081/8082 (3550)	Clear Tall 8oz	30 – 31 g	When both 8081/8082 are requested, ISM only one 30 – 31 g aliquot to be used for both tests. Perform a MS/MSD pair for both 8081 and 8082. Also called pesticides/PCB.
8081/8082 (3546)	Snapcap	15 – 16 g	When both 8081/8082 are requested, ISM only one 15 – 16 g aliquot to be used for both tests. Perform a MS/MSD pair for both 8081 and 8082. Also called pesticides/PCBs.
8270-SIM	Clear Tall 8oz (3550) Snapcap (3546)	10 – 11 g	Also called PAH-SIM. 10 g for either prep method 3550 or 3546.
TPH	Clear Tall 8oz	30 – 31 g	Also called TEPH/TPH/8015. 8015 DRO and 8015 MRO might be done at different times (wet vs. dry).
%Moisture	Snapcap	11 ± 6g	Ensure to do a sample duplicate but no MS/MSD. The weight amount can be altered if insufficient sample size as long as it is representative.
9045C	Snapcap	20 – 21 g	pH
6850	Falcon Tube	3 – 4 g	Use a VOA vial for the backup container
8330A	VOA vial	2 – 2.1 g	Should have a MS/MSD/Sample Duplicate/Sample Triplicate
8330B	VOA vial	10 – 10.1 g	Should have a MS/MSD/Sample Duplicate/Sample Triplicate
6010	Snapcap	10 – 10.2 g	Ensure a minimum of one MS/MSD pair per 12 samples
TCLP (1311)	Clean 8oz TCLP Falcon Tube pH	100 – 101 g TCLP 5 – 5.2 g pH	A 100g aliquot is good for 2 methods (e.g. 6010/7470 or 8081/8270). If more than two methods are requested, multiple 100 g aliquots may need to be taken, but only 1 pH aliquot is needed.
PFOA/PFOS	Falcon Tube	5 – 5.1 g	PFCs - Do not use any Teflon products.
6020	Snapcap	10 – 10.2 g	Ensure a minimum of one MS/MSD pair per 12 samples
7471	Falcon Tube	10 – 10.2 g	Ensure a minimum of one MS/MSD pair per 12 samples
8290	Snapcap	10 – 11 g	When both 8290/1668 are requested, ISM only one 20 – 22 g aliquot to be used for both tests. A MS/MSD is required only if the samples are QSM work. The MS/MSD can be shared between both tests as well.

Test	Container	Weight	Notes
1668	Snapcap	10 – 11 g	When both 8290/1668 are requested, ISM only one 20 – 22 g aliquot to be used for both tests. A MS/MSD is required only if the samples are QSM work. The MS/MSD can be shared between both tests as well.
AK102/AK103	Clear Tall 8oz	30 – 31 g	NA
8270	Clear Tall 8oz	30 – 31 g	NA
8280	Snapcap	10 – 11 g	A MS/MSD is required only if the samples are QSM work.
8151	125 AGJ	50 – 51 g	Extraction not performed locally. Ensure to do in duplicate with a MS/MSD pair. Label containers with labels from sample control. Try and get closer to 51g.

Attachment 4 (QA-562B)
ISM Drying and Additional Information for QSM Samples

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Sacramento
ISM Drying and Additional Information

Drying Room Temperature °C:	Obs	Thermometer ID:
	High	
	Lo	

Sample ID		Consistent Weight Measurements (taken from a ~10g subsample)					Total Sample Weights (gram unless otherwise noted)				
Login #	Sample #	Initial Weight/Time taken	Weight after 2hr/ Time taken	Weight after 4hr if needed/ Time taken	Balance ID	Initials and Date	Initial Weight (As Received)	Dried and Sieved Weight	Dried Unsieved (Waste) Weight	Balance ID	Initials and Date

Q: Document-Management/Forms/QA-562B r2 ISM Drying and Additional Info

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