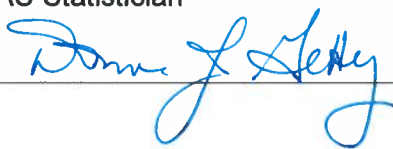

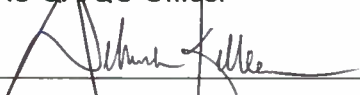



## STANDARD OPERATING PROCEDURE APPROVAL AND CHANGE FORM

Scientific, Engineering, Response and Analytical Services 2890 Woodbridge Avenue Building 209 Annex Edison New Jersey 08837-3679	
<b>STANDARD OPERATING PROCEDURE</b>	
Title: Incremental Sampling Methodology for Soil	
Approval Date: 12/15/2015	
Effective Date: 12/15/2015	SERAS SOP Number: 2019, Rev 0.0
<b>Authors</b>	
Name Donna Getty Title SERAS Statistician Signature  Date 12/15/2015	
Name Title Signature Date	
<b>Approvals</b>	
Name Richard Leuser Title SERAS Deputy Program Manager Signature  Date 12/14/15	
Name Deborah Killeen Title SERAS QA/QC Officer Signature  Date 12/11/15	
Name Kevin Taylor Title SERAS Program Manager Signature  Date 12/11/15	

The top row of this table shows the most recent changes to the controlled document. For previous revision history information, archived versions of this document are maintained by the SERAS QA/QC Officer on the SERAS local area network (LAN).

History	Effective Date
New SOP	12/15/15



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 1 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

### CONTENTS

- 1.0 SCOPE AND APPLICATION
- 2.0 METHOD SUMMARY
  - 2.1 Inorganics and Other Non-Volatile Contaminants of Potential Concern
  - 2.2 Volatile Contaminants of Potential Concern
- 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE
  - 3.1 Preserving and Handling Volatile Contaminants of Potential Concern
- 4.0 INTERFERENCES AND POTENTIAL PROBLEMS
- 5.0 EQUIPMENT/APPARATUS
- 6.0 REAGENTS
- 7.0 PROCEDURES
  - 7.1 Defining Decision Units
  - 7.2 Specifying the Target Population
  - 7.3 Soil Increments
  - 7.4 Sample Collection
    - 7.4.1 Basic Incremental Sampling Guidelines for All Contaminants of Potential Concern
    - 7.4.2 Incremental Sampling Guidelines for Volatile Contaminants of Potential Concern
  - 7.5 Laboratory Subcontracting Concerns: Bulk Sample Processing
  - 7.6 Field Replicates
  - 7.7 Stepwise Procedure for Collecting a 30 Increment Sample for Inorganic or Other Non-Volatile Contaminants of Potential Concern
  - 7.8 Stepwise Procedure for Collecting a 30 Increment Sample for Volatile Contaminants of Potential Concern
- 8.0 CALCULATIONS
  - 8.1 Standard Deviation of Replicate Incremental Sampling Methodology Samples
  - 8.2 Relative Standard Deviation of Incremental Sampling Methodology Replicate Samples
  - 8.3 95% Upper Confidence Limit
  - 8.4 Mass of Bulk Sample

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 2 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

### CONTENTS (cont'd)

- 9.0 QUALITY ASSURANCE/QUALITY CONTROL
  - 9.1 Standard Deviation of Incremental Sampling Methodology Replicates
  - 9.2 Relative Standard Deviation of Incremental Sampling Methodology Replicates
  - 9.3 95% Upper Confidence Limit
- 10.0 DATA VALIDATION
- 11.0 HEALTH AND SAFETY
- 12.0 REFERENCES
- 13.0 APPENDIX
  - A – Figures

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 3 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

### 1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) provides guidelines associated with the collection of representative soil samples using the incremental sampling methodology (ISM). The objective of ISM is to obtain a representative sample of contaminants, for a defined soil fraction, in exactly the same proportion as the sampling area. ISM consists of a field collection and an analytical preparation component. This SOP is based on the methodologies and information presented in The Interstate Technology and Regulatory Council (ITRC) guidance document, “*Incremental Sampling Methodology*” (2012).

ISM is based on sampling theory developed by Pierre Gy (Pitard, 2000a and 2000b) for the mining industry. Incremental sampling can be applied to both simple, such as characterization of individual residential plots, and complex scenarios, such as an industrial site consisting of a 100 acres requiring characterization of specific areas and also remediation of even smaller plots within those areas. Complex scenarios require a tiered application of ISM. A statistician should be consulted for the design and lay-out of a tiered incremental sampling design.

A Quality Assurance Project Plan (QAPP) in Uniform Federal Policy (UFP) format describing the project objectives must be prepared prior to deploying for a sampling event. The sampler needs to ensure that the methods used are adequate to satisfy the data quality objectives listed in the QAPP for a particular site.

The procedures in this SOP may be varied or changed as required, dependent on site conditions, equipment limitations or other procedural limitations. In all instances, the procedures employed must be documented on a Field Change Form and attached to the QAPP. These changes must be documented in the final deliverable.

### 2.0 METHOD SUMMARY

Incremental sampling is a methodology for collecting and processing samples which controls variability associated with the non-uniform/heterogeneous distribution of contaminants in soil. The objective is to obtain a representative estimate of the mean concentration of the contaminants of potential concern (COPCs) for a specified particulate fraction within an established area.

A systematic grid or transects, based on a random starting location, is established for each area where a decision will be made (Decision Unit [DU]; Section 7.1). Typically 30-sample increments are collected, using a coring device at each grid node (Figures 1 and 2), and combined into a sample collection container producing one bulk incremental soil sample. Methods for increment collection vary based on whether the COPC is a volatile or non-volatile compound.

#### 2.1 Inorganics and other Non-Volatile Contaminants of Potential Concern

For non-volatile COPCs, the bulk sample is disaggregated by hand or a mechanical grinding/milling device with the method of disaggregation based on the COPC and project goals. The bulk sample is then sieved and homogenized at the laboratory.

Sub-sampling of the sieved bulk sample in the laboratory is then conducted by spreading the entire sieved sample out to a thin layer on clean plastic or a decontaminated flat surface to create a slab cake. Approximately 30 sub-samples are then collected at systematic random locations across the sample (Figure 3), using a small rectangular-shaped scoop. These subsamples are placed into a clean sample container to form one composite sample for laboratory analysis.

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 4 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

### 2.2 Volatile Contaminants of Potential Concern

Incremental samples, which will be analyzed for volatile COPCs, are handled in such a way as to minimize volatilization of the COPC. Increments are collected and injected directly into a sampling jar preserved with an adequate amount of methanol to form a bulk sample. Additional sample material is collected in a separate jar for a corresponding percent moisture analysis. Alternately, individual increments are collected using separate sampling devices, 1 per increment, that have vapor tight seals and are designed for zero headspace, such as EnCore samplers. All increments are shipped to the laboratory where they are combined into methanol in the laboratory to form a bulk sample. In both cases, the bulk samples are not ground, milled, or sieved

*Note: This method only works for the analysis of medium/high level volatile COPCs. The volume of methanol that can be injected into a Gas Chromatograph/Mass Spectrometer (GC/MS) is a limiting factor; therefore, preserving the increments in methanol lowers the sensitivity of the analysis and increases the analytical reporting limit (RL).*

### 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

All bulk samples (volatile and non-volatile) must be cooled and maintained at less than or equal to ( $\leq$ ) 6 degrees Centigrade ( $^{\circ}$ C), and protected from sunlight to minimize any potential reaction. The amount of sample to be collected, proper sample container type and handling requirements is dependent on the COPC types and are discussed in the Scientific, Engineering, Response Analytical Services (SERAS) SOP #2003, *Sample Storage, Preservation and Handling*.

#### 3.1 Handling and Preserving Volatile Contaminants of Potential Concern

Increments collected for the analysis of medium/high level VOCs, must be placed directly into the appropriate volume of methanol in the field. Increments should remain completely submerged in the solvent at all times.

### 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

- Depending on the COPCs, grinding can introduce bias from loss of sample analyte or through an introduction of analytes from the grinding equipment.
- Preserving soil increments in methanol for the analysis of volatile contaminants decreases the sensitivity of the analytical method and increases the RL.
- A DU that is too large for the stated objective of the sampling event could misrepresent contamination by biasing low or diluting the contamination. Source areas, spill/release areas should be identified as a separate DU from the remaining site exposure areas (Section 7.1).
- A DU that is too small for the stated objective could increase cost without significantly impacting conclusions.

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 5 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

### 5.0 EQUIPMENT/APPARATUS

Equipment needed for collection of sediment samples may include:

- Maps/plot plan
- Safety equipment
- Global positioning system (GPS)
- Survey stakes, flags
- Camera
- Stainless steel, plastic or other appropriate composition bucket
- One-quart wide mouth jars w/Teflon lined lids for non-volatile samples
- EnCore or equivalent type sampling devices for VOCs
- 2 oz jars for analysis of percent (%) moisture
- Ziploc plastic bags
- Logbook or field data sheets
- Sample jar labels
- Chain of Custody records
- Chain of Custody seals
- Cooler(s)
- Ice
- Decontamination supplies/equipment
- Sample tube; coring device

### 6.0 REAGENTS

- Methanol in 32 ounce (oz) amber bottles to preserve VOC samples

Non-dedicated field sampling equipment should be decontaminated according to the procedures specified in SERAS SOP #2006, *Sampling Equipment Decontamination*. It is not necessary to decontaminate equipment between increments collected within the same DU, but field equipment must be decontaminated between samples and/or DUs.

### 7.0 PROCEDURES

Incremental sampling is a type of composite sampling which reduces the variability typically associated with discrete sampling and has greater reproducibility. ISM is utilized when the objective is to obtain a representative mean of COPCs for a specific soil fraction within a designated geographic area (DU) for which a decision/conclusion will be made. The typical application for this sampling methodology is for determination of exposure (human or ecological). Identification of an appropriate sized DUs is based on the defined project goals and data quality objectives.

#### 7.1 Defining Decision Units

DUs are defined by understanding the vertical and horizontal contaminant distribution across the site, known presence of source areas, transport and migration pathways, geologic formations, human and/or ecological risk model requirements, soil types, property boundaries, property use, such as

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 6 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

residential, farming, industrial, and sampling goals. The entire site can be classified as a single DU or the site can be broken down into smaller DUs, each with their own sampling goals.

DUs can be broken into smaller areas designated as Sampling Units (SUs), from which one or more ISM samples are collected. Decisions are not made based on the results of a single SU, but rather on the combined information of the SUs within a single DU. The mean concentration of the COPC, over the entire DU, is what supports the decision. SUs can be used to calculate confidence intervals and to better define the spatial distribution of contaminants within a DU.

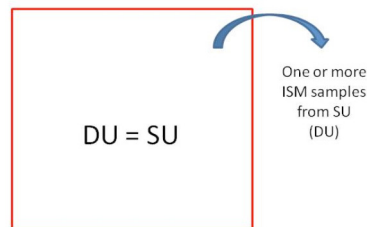


Figure 3-2a. DU = SU (SU concept is not needed).

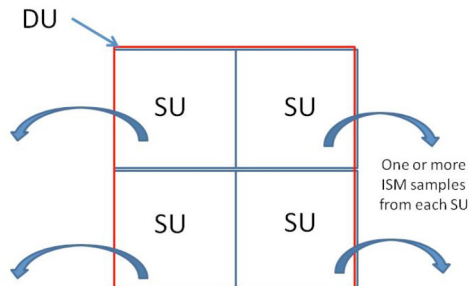


Figure 3-2b. DU is subdivided into 4 SUs

(IRTC, 2012; p.47)

IRTC (2012) divides DUs into two primary categories; those based on the:

- 1) Known or suspected locations of sources areas which would include areas of stained soil, site areas where contaminants are suspected to have been stored, handled or disposed, and areas where evidence indicates the presence of elevated concentrations of COPCs in relation to surrounding areas, and
- 2) Size assumptions of risk assessments; these areas are frequently referred to as, exposure units, such as boundaries of a residential property or exposure area of a receptor species.

### EXAMPLES:

DUs can be defined as individual properties (e.g., residential properties within a neighborhood, recreational fields, etc.), generally smaller than 0.25 of an acre. ISM is conducted within each DU. In this case, incremental sampling results in a representative measure of average COPC(s) per DU or residential property. Decisions based on the ISM results, are made per DU. A decision may include removal of surface soil from a specific residential property.

A larger industrial property (>0.25 acres) can be broken into multiple DUs defined by differences,

UNCONTROLLED COPY





# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 7 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

such as source area, chemical storage location or small mammal habitat with ISM conducted per DU. A specific removal/risk decision is made for the surface soil from the source area, from the chemical storage location and across the small mammal habitat.

A residential property can be divided into multiple DUs based on varying expected use and exposure. The property might be divided into a DU encompassing the drip line surrounding the perimeter of the house which accounts for potential run-off of lead-based paint or asbestos shingles; a second defined as the backyard where the children are most likely to play, excluding the drip line; and the third DU might be made up of the combined front and side yards, again, excluding the drip line).

### 7.2 Specifying the Target Population

Project goals must include a definition of the target population to be investigated. The definition of the target population includes the site-specific COPCs, soil fraction of interest, depth(s) of the soil under investigation, expected heterogeneity of the soil, and potential sources of variability. The definition should be as detailed as possible.

EXAMPLE: For a residential property thought to be impacted by vermiculite mining/production, the defined fraction and contaminant of interest might be: the average concentration of asbestos (Libby amphibole) in the surface top 2 centimeters (cm) of soil with grains less than 2 millimeters (mm) in diameter and within property boundaries.

Determine how many soil increments will be collected per DU resulting in one incremental sample. According to Gy's sampling theory a **minimum of 30 increments** should be collected. If the contaminant distribution within a single DU is expected to be extremely heterogeneous (based on historical information or professional judgment) decide whether increasing the number of increments to 60 or 100 will provide a more representative average. This can be done based on estimates of variability from historical data in consultation with a statistician. Another option is to break the DU into multiple SUs which are more homogeneous.

### 7.3 Soil Increments

Sample and increment volume is dependent on the selected laboratory analyses/methods. It is critical to ensure that enough volume is collected per increment to meet analytical needs. Typically **30 increments** are collected across a single DU using a soil coring sampler to collect a sample over the entire depth of interest (Figure 1). The size of the corer needed is based on the required sample volume. Use of inappropriate tools or collection of a sample that contains more soil particles from the top of the depth interval of concern than the bottom (or vice versa) leads to biased sample results.

The mass of any single increment is dependent on the soil density, moisture content of the soil and the size of the collection tool. Soil density should be similar across a DU. Individual soil increments that make up a composite sample are expected to typically weigh between 5 and 50 grams (g) each. When choosing the mass per increment, keep in mind that the field composite sample should typically weigh between 300 and 2,500 g after sieving to the target particle size. The 300 to 2,500 g suggestion is based on the mass sufficient to minimize Gy's Fundamental Error for sample collection (U.S.EPA 1999). The table below, taken from ITRC (2012, Table 5.1), presents the

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
 PAGE: 8 of 21  
 REV: 0.0  
 EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

number of increments needed to attain a specified sample mass based on the typical range of soil density and size of the soil coring sampler. Section 8.4 provides a formula to calculate sample mass.

**Estimated Sample Mass for Core Increment Depth and Substrate Density\***

Corer Diameter (cm)	Number of Cores to Obtain desired ISM Sample Mass				
	500 g	750 g	1000 g	1500 g	2000 g
<b>Soil density 1.6 g/cm<sup>3</sup>, core depth 2.5 cm</b>					
2.0	40	60	80	119	159
3.0	18	27	35	53	71
4.0	10	15	20	30	40
<b>Soil density 1.8 g/cm<sup>3</sup>, core depth 2.5 cm</b>					
2.0	35	53	71	106	141
3.0	16	24	31	47	63
4.0	9	13	18	28	35

\*Substrate density may vary from 0.75 grams per cubic centimeter (g/cm<sup>3</sup>) (for Loess) to 1.81 (for Gravely Sand) with substrate densities typically ranging 1.6–1.8 g/cm<sup>3</sup>.

**EXAMPLE.** The laboratory requests 2-64 oz. amber jars, approximately 1814 grams, per ISM sample so that there is sufficient sample material to analyze for a variety of parameters. Samples will be collected from the surface, 0 to 2.5 centimeters using a 2-cm coring device and the soil is expected to be sandy. Using the table above and based on a sandy soil density of 1.8 g/cm<sup>3</sup>, the number of cores needed to meet the required sample mass for the laboratory is:

$$106 < \# \text{ of cores per ISM bulk sample} < 141$$

The UFP-QAPP specifies 30 increment locations. This implies that:

$$106/30 < \# \text{ of cores per increment location} < 141/30$$

Or

$$3.5 < \# \text{ of cores per increment location} < 4.7$$

From this information, the project team decides to collect 4 cores from each increment location. The 120 cores will comprise one ISM sample.

The online version of the ITRC (2012) document contains a downloadable calculator which can be used in Excel to determine the number of increments needed to achieve the desired sample mass based on expected soil density, core diameter, and sample depth.

### 7.4 Sample Collection

A systematic grid or transects, based on a random starting location, is established per DU. Random starting locations and grids can be established using Visual Sample Plan software (<http://vsp.pnnl.gov/>), geographic information system software (GIS) or a random starting location generated using a random number generator, such as the @randbetween function in Excel. To use the Excel function, pace-off and count the number of steps it takes to walk the length and then again

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 9 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

the width of the DU. Using the random number generator, compute a random starting point by inputting the number of paces lengthwise and then widthwise.

For example if the length was 185 paces and the width was 122 paces:

Length Coordinate: @randbetween(0,185) = 142 paces

Width Coordinate: @randbetween(0,122) = 78 paces

The initial random sampling location would be where 142 paces lengthwise and 78 paces widthwise intersect.

The sampling grid is built from this random point. Sample increments are collected at regular intervals along the grid pattern/transect in such a way as to collect the pre-determined number of increments. For irregularly shaped DUs, row lengths and the number of increments per row are modified as needed to obtain regular spacing between increment collection points.

A trial run with no sample collection is performed to establish the distance between increment collection points. Flags are placed at each sample location. If replicate samples are to be collected (Section 7.8), a different color flag is used for the original ISM sample and each replicate. Locations for replicate samples should not be co-located or adjacent to the original locations. They are a pre-determined distance, one that ensures coverage of the DU, and direction away from each of the original increment locations (See Figure in Section 7.6).

Sampling is typically conducted by two people. One person collects and keeps track of the number of cores required at each collection point. The second person captures GPS coordinates of the collection locations.

Increment collection can be conducted following a systematic random approach or a stratified random approach.

- For systematic collection, individual soil increments are collected along evenly laid out transects within the DU. Increments are collected at regular intervals along each transect. Distances do not need to be individually measured rather just paced out, such as every 5 steps one increment is collected. When the samplers reach the end of a row, they proceed back along the next grid transect, once again collecting an increment of sample at approximately equal distances. This serpentine pattern is continued, until the determined number of increments have been collected, ending at the opposite corner or end of the DU. Sampling equipment is not decontaminated between increments.
- For stratified random sample collection, a grid is established across the DU, and a random location (using VSP, GIS, or a random number generator) is selected in each cell from which to collect a single increment.

For both sampling strategies, if an obstacle is encountered preventing increment collection at a specific location, a soil increment should be collected as close as possible to the original location.

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 10 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

### 7.4.1 Basic Incremental Sampling Guidelines for all Contaminants of Potential Concern

Basic guidelines for incremental sampling cited by the ITRC and based on Pierre Gy's sampling theory include:

- The pattern of incremental soil collection within a DU must be unbiased. This is achieved by starting the grid at a randomly generated location within the DU.
- There must be complete and uniform sampling across the entire sample depth interval of interest. If the defined sample depth interval is 0 to 6 inches then a proportionate amount of soil must be collected along the entire interval from the top depth down to the 6 inch depth by using a core sampling tool.
- Each increment should be approximately the same/uniform mass. This is achieved by using coring devices that ensure a uniform diameter core. A garden trowel or shovel will not meet this objective.

### 7.4.2 Incremental Sampling Guidelines for Volatile Contaminants of Potential Concern

Samples for volatile analyses such as volatile organic compounds (VOCs), benzene, toluene, ethylbenzene, and xylenes (BTEX), and gasoline range organics (GROs) are collected following specific protocols to minimize loss of contaminant concentration due to volatilization. This methodology includes collecting increments using a disposable plastic syringe or similar coring device (Figure 2) and extruding the sample directly into a 250-500 milliliter narrow-mouthed amber jar, containing the appropriate volume of methanol preservative, with Teflon lined lids (ADEC, 2009). The amount of sample to be collected, as well as the necessary volume of methanol, is considered when choosing the appropriate sized sample container.

A second unpreserved narrow-mouthed jar is required for determination of percent moisture. Sample volume should be increased by 3-times to help minimize total sampling error because these samples cannot be sieved. Additionally, every effort must be made to exclude large rocks and clumps from the sample. Sampling objectives may also require the removal of grass and other organic material; this should be identified in the project-specific UFP-QAPP.

Alternatively, individual increments are collected into separate sampling devices, at 1 per increment, that have vapor tight seals and are designed for zero headspace, such as EnCore samplers. All increments are shipped to the laboratory where they are combined into methanol in the laboratory to form a bulk sample. Note that samples collected in Encore-type devices must be removed from these devices within 48 hours of collection. Make sure that these samples are immediately shipped to the laboratory within a timeframe that allows the laboratory sufficient time to composite the increments into methanol.

In both cases, the bulk samples for volatile COPCs are not ground, milled, or sieved.

*Note: This method only works for the analysis of medium/high level volatile COPCs. The volume of methanol that can be injected into a Gas Chromatograph/Mass Spectrometer (GC/MS) is a limiting factor; therefore, preserving the increments in methanol lowers the sensitivity of the analysis and increases the analytical RL.*

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 11 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

### 7.5 Laboratory Subcontracting Concerns: Bulk Sample Processing

Because of the unique nature of incremental samples, in particular the larger volume of sample mass and required sample preparation, the selected laboratory must have documented familiarity with the ISM such as SOPs, a documented history of analysis of ISM samples, etc. The laboratory must also have the physical capability of handling the incremental samples including sufficient area for air drying the individual samples controlling for cross-contamination.

The SERAS Analytical Subcontracting Representative needs to be notified when ISM will be conducted, so that the laboratory can be appropriately subcontracted.

### 7.6 Field Replicates

ITRC recommends that field replicate incremental samples be collected from a minimum of 10 % of the DUs; however, **SERAS' policy is to collect replicate samples at all DUs**, unless otherwise specified in the project-specific UFP-QAPP, resulting in triplicate samples (the original sample plus two replicate samples) for each DU. The collection of triplicate incremental samples allows for the calculation of relative standard deviation (RSD), which measures the precision or reproducibility of the ISM. The greater the RSD the less confidence there is in the estimate of the mean COPCs for that DU.

Field replicates are collected similarly to the original incremental sample, consisting of the same number of individual increments. Increment locations for the field replicates are unique from the original sample and are not collected at co-located or adjacent locations. Once the original sample has been collected, the sampling team returns back to the initial increment collection point marked by a flag and moves to the right, left, forward, or backward to the previously marked replicate location (Section 7.4) and begins increment collection for the replicate sample.

Increment collection for a replicate sample follows a serpentine pattern similar to the original sample but along a different path (Figure 4). For example, if the original sample was collected by following a path from north to south starting in the west and going east, a replicate sample could be collected walking a path from west to east starting in the north and moving south as seen in the figure below modified to collect the designated number of increments. Another replicate could be collected from south to north, moving from east to west. Field replicates are given unique sample identification numbers that do not reveal their identity to the laboratory.

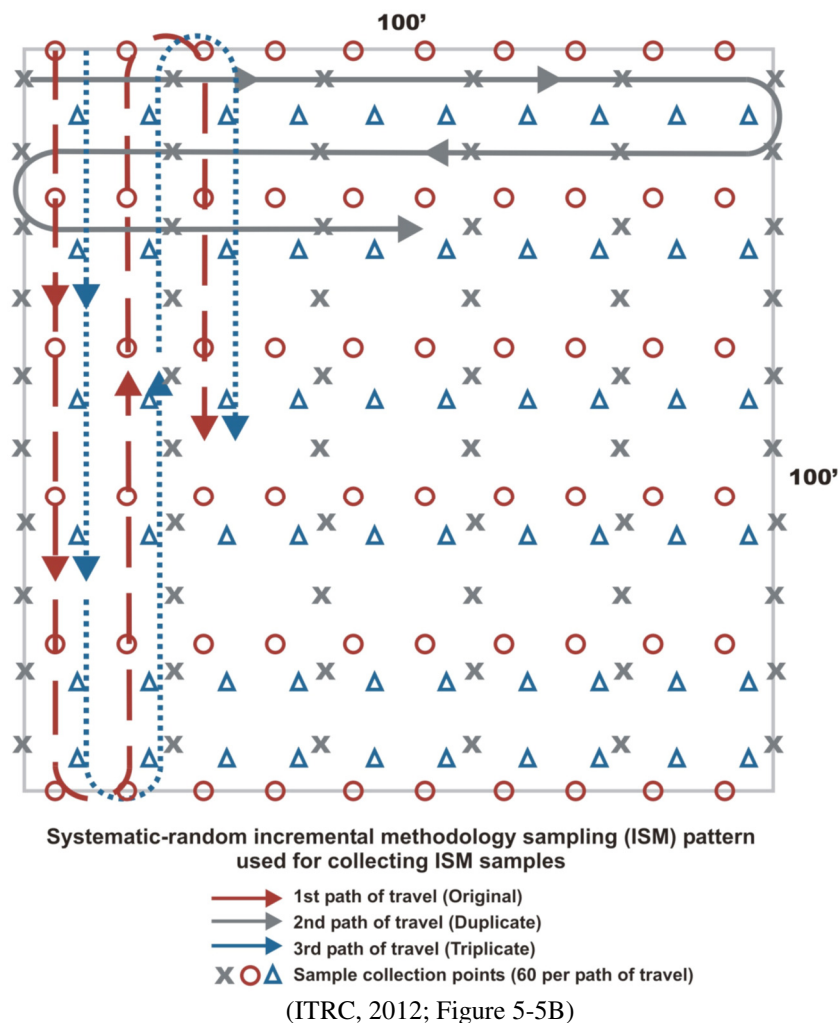
UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 12 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL



### 7.7 Stepwise Procedure for Collecting a 30-Increment Sample for Inorganic or Other Non-Volatile Contaminants of Potential Concern

1. Locate DU/SU and GPS its boundaries.
2. Pace the length and width of the DU counting the paces for each and use the information to generate a random starting location (See Section 7.4). Alternatively use VSP or GIS to generate a random grid.
3. Build a grid from the random location in such a manner that increments are spaced at regular intervals within the DU.
4. Determine the distance and directional moves that will be required to collect the replicate samples and document this information in the field logbook(s).
5. Use pin flags to define the sampling area and locate grid/transect pattern.
6. Determine path and direction that will be walked to collect initial and replicate samples.

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 13 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

7. Take a trial walk along the paths that will be followed for increment collection to ensure spacing is adequate to collect 30 increments.
  8. Place pin flags and GPS the increment locations along these paths. Use unique colored flags for the original ISM sample and each of the two replicate samples.
  9. Following the grid/transect line, locate the first increment collection point.
  10. Using a dedicated volumetric sampling device collect an increment.
  11. Place increment into sample container.
  12. Continue to the next increment collection point and repeat steps 10 and 11.
    - a. Repeat this process following a serpentine path until 30 increments are collected.
  13. Composite and adequately mix the 30 increments in a clean stainless steel bowl or foil pan.
  14. Place bulk sample into clean sample containers.
  15. Label each sample container with the proper identification label.
  16. Collect the replicate samples:
    - a. Move the pre-determined number of feet in the pre-designated direction (determined in Step 1) from the initial incremental collection point and begin collecting another IS.
    - b. Repeat steps 9 through 15.
    - c. Repeat steps 16a and 16b for the second replicate IS.
  17. Remove pin flags only after all increment collection point coordinates have been collected with a GPS.
  18. Decontaminate equipment.
- 7.8 Stepwise Procedure for Collecting a 30-Increment Sample for Volatile Contaminants of Potential Concern
1. Locate DU/SU and GPS its boundaries.
  2. Pace the length and width of the DU counting the paces for each and use the information to generate a random starting location (See Section 7.4). Alternatively use VSP or GIS to generate a random grid.
  3. Build a grid off of the random location in such a manner that increments are spaced at regular intervals within the DU.
  4. Determine the distance and directional moves that will be made to collect the replicate samples.
    - a. Document this information in field logbooks.
  5. Use pin flags to define the sampling area and locate grid/transect pattern.
  6. Determine path (direction) that will be walked to collect initial (and replicate samples).
  7. Take a trial walk along the paths that will be followed for increment collection to ensure spacing is adequate to collect 30 increments.
  8. Place pin flags and GPS increment locations along these paths. Use unique colored flags for the original ISM sample and each of the two replicate samples.
  9. Following the grid/transect line, locate the first increment collection point.
  10. Using a dedicated volumetric sampling device with a vapor tight seal and zero head space (e.g. EnCore samplers) collect an increment. Label the sample. Alternatively, using a disposable plastic syringe or similar coring device extrude the increment directly into a narrow-mouthed amber jar containing methanol (Section 7.4.2).
  11. At the same collection point, using a dedicated coring device collect additional sample for the analysis of % moisture.
  12. Place into a different sample container without methanol.
  13. Continue to second increment collection point and repeat steps 9 and 10.
  14. Repeat this process following a serpentine path until 30 increments are collected.
  15. Label each sample container with the proper identification label, if they have not already been labeled.
  16. Collect the replicate samples:

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 14 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

- a. Move the pre-determined number of feet in the pre-designated direction (determined in Step 1) from the initial incremental collection point and begin collecting another IS.
  - b. Repeat steps 9 through 15.
  - c. Repeat steps 16a and 16b for the second replicate IS.
17. Remove pin flags only after all increment collection point coordinates have been collected with a GPS.  
18. Decontaminate equipment.

### 8.0 CALCULATIONS

#### 8.1 Standard Deviation of Replicate Incremental Sampling Methodology Samples

$$\text{Standard deviation} = \sqrt{\frac{\sum y^2}{n-1}}$$

Where:

y: are the analytical results for the COPC of concern for each replicate  
n: the number of replicates (usually 3)

For sample results that are non-detect, consult a statistician or Risk Assessor.

#### 8.2 Relative Standard Deviation of Replicate Incremental Sampling Methodology Samples

The RSD is expressed as a percent and is calculated using the following formula:

$$\text{RSD} = \frac{100 \times \text{standard deviation}}{\text{average of replicates}}$$

#### 8.3 95% Upper Confidence Limit

$$95\% \text{ UCL} = \text{arithmetic mean} + \frac{95\% \text{ one-sided Student } t\text{-factor} \times \text{standard deviation}}{\sqrt{\text{number of replicate samples}}}$$

Where:

Student *t* factor: taken from a statistical table; if the number of (replicate) samples is 3, the 95% one-sided student *t* factor = 2.92

#### 8.4 Mass of Bulk Sample (ITRC, 2012)

$$M_s = \rho \times n \times D_s \times \pi \times (\theta/2)^2$$

Where:

$M_s$  = targeted mass of sample (g)  
 $D_s$  = increment length (g)  
n = number of increments  
 $\rho$  = soil or sediment density (g/cm<sup>3</sup>)  
 $\theta$  = diameter of sample core (cm)

UNCONTROLLED COPY





# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 15 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

### 9.0 QUALITY ASSURANCE/QUALITY CONTROL

Specific QA/QC activities that apply to the implementation of these procedures will be listed in the Quality Assurance Project Plan prepared for the applicable sampling event or in the sections below. The following general QA procedures will also apply:

1. All sample collection data must be documented on field in site logbooks.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer or instrument-specific SOPs, unless otherwise specified in the QAPP. Equipment check-out and calibration is necessary prior to purging and sampling and must be done according to the instruction manuals supplied by the manufacturer.

#### 9.1 Standard Deviation of Incremental Sampling Methodology Replicates

Standard deviation is used to measure variation from the mean among a group of samples. For IS a standard deviation is computed for the analytical results of the triplicate samples taken in one or more DUs. The lower the standard deviation, the more precise the site data will be as an estimate of average contaminant concentration in the DU under investigation.

#### 9.2 Relative Standard Deviation of Incremental Sampling Methodology Replicates

The RSD of the field replicates (triplicates) is used to evaluate whether the IS analytical results are reliable enough to make a decision that an average contaminant concentration is below/above the project action levels or other decision criteria. The RSD is expressed as a percent; the lower the RSD is the more reliable the estimate of average concentration for the DU is. Generally, an RSD of 35% or less indicates the amount of estimated total error is within a reasonable range for decision-making; however this is also dependent on the DQOs established for the project.

As stated in USEPA 2011, the incremental sampling approach provides averages that approximate a statistically normal distribution if the RSD of replicates is low. The higher the RSD the more heterogeneity and the less confidence there is that the averages approximate a normal distribution. There is also less confidence that the average contaminant concentrations are representative of the contaminant levels within the DU. If the RSD approaches or exceeds 50%, and if the average DU concentrations are near project action levels, there is increasing uncertainty that the data are adequately representative. Additional incremental sampling may be necessary, utilizing a larger number of sample increments and/or larger sample increment masses to obtain a more representative measure of the contaminant concentrations in the DU.

#### 9.3 95% Upper Confidence Limit

Another method to evaluate variation of the replicate (triplicate) samples from the mean is to calculate the 95% upper confidence limit (95% UCL) of the arithmetic mean. The 95% UCL of the arithmetic mean for the COPC(s) in the replicate DU are used for comparison to the project action levels or decision criteria. Application of the 95% UCL formula assumes contaminant data approximate a normal distribution which is indicated by an  $RSD \leq 35\%$ . Use of either the standard deviation or 95% UCL statistic is acceptable to determine sample data variation from the mean based on triplicate samples.

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 16 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

### 10.0 DATA VALIDATION

Data verification (completeness checks) must be conducted to ensure that all data inputs are present for ensuring the availability of sufficient information. This may include but is not limited to: location information, random starting location, number of increments and GPS measurements. These data are essential to providing an accurate and complete final deliverable. The SERAS Task Leader (TL) is responsible for completing the UFP-QAPP verification checklist for each project. Analytical results will be validated against the appropriate analytical method.

### 11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, Occupational Safety and Health Act (OSHA), and Corporate health and safety procedures.

### 12.0 REFERENCES

ADEC (Alaska Department of Environmental Conservation) *Draft Guidance on MULTI INCREMENT Soil Sampling*. March 2009. Division of Spill Prevention and Response. Contaminated Sites Program. [http://dec.alaska.gov/spar/csp/guidance/multi\\_increment.pdf](http://dec.alaska.gov/spar/csp/guidance/multi_increment.pdf)

ITRC (Interstate Technology & Regulatory Council). *Incremental Sampling Methodology*. ISM-1. February 2012. Washington, D.C.: Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team. [http://www.itrcweb.org/ism-1/pdfs/ISM-1\\_021512\\_Final.pdf](http://www.itrcweb.org/ism-1/pdfs/ISM-1_021512_Final.pdf)

Pitard, Francis F. Pierre Gy's Sampling Theory and Sampling Practice. Volume I. Heterogeneity and Sampling. 2000a. CRC Press, Inc. Boca Raton, Florida.

Pitard, Francis F. Pierre Gy's Sampling Theory and Sampling Practice. Volume II. Sampling Correctness and Sampling Practice. 2000b. CRC Press, Inc. Boca Raton, Florida.

Pacific Northwest National Laboratory (PNNL). Visual Sample Plan (VSP) software. <http://vsp.pnnl.gov/>

Technical Guidance Manual for the Implementation of the Hawai'i State Contingency Plan. Interim Final. November 12, 2008. State of Hawai'i. Department of Health. Office of Hazard Evaluation and Emergency Response. Honolulu, Hawaii. <http://www.hawaiidoh.org/tgm.aspx>

United States Environmental Protection Agency (U.S. EPA), 1999. Correct Sampling using the Theories of Pierre Gy. Office of Research and Development Technology Support Project. <http://www.epa.gov/esd/factsheets/coutpg.pdf>

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 17 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

APPENDIX A  
Figures  
SOP #2019  
December 2015

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 18 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

FIGURE 1. ISM Tools for Sampling for Non-Volatiles



**Figure 5-2a. Examples of coring devices for nonvolatile soil increment collection.** Top to bottom: Multi-Incremental Sampling Tool (MIST™), EVC Incremental Sampler, JMC Backsaver Handle, and Soil Tube. , ITRC 2012.

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 19 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

FIGURE 2. ISM Tools for Sampling for Volatiles



**Figure 5-12. Examples of coring devices for VOC soil increment collection.** Core N' One™ tool (left), Terra Core Sampler (center), and Easy Draw Syringe® and PowerStop Handle® (right). *Source:* Courtesy [www.ennovativetech.com](http://www.ennovativetech.com), ITRC 2012.

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 20 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

---

FIGURE 3. Laboratory ISM Sample Processing



Figure 6-3. Example of 2-D Japanese slab-cake incremental subsampling on dried and sieved soil. ITRC, 2012

UNCONTROLLED COPY



# STANDARD OPERATING PROCEDURES

SOP: 2019  
PAGE: 21 of 21  
REV: 0.0  
EFFECTIVE DATE: 12/15/15

## INCREMENTAL SAMPLING METHODOLOGY FOR SOIL

FIGURE 4. Collecting Replicate ISM Samples from a Decision Unit



UNCONTROLLED COPY