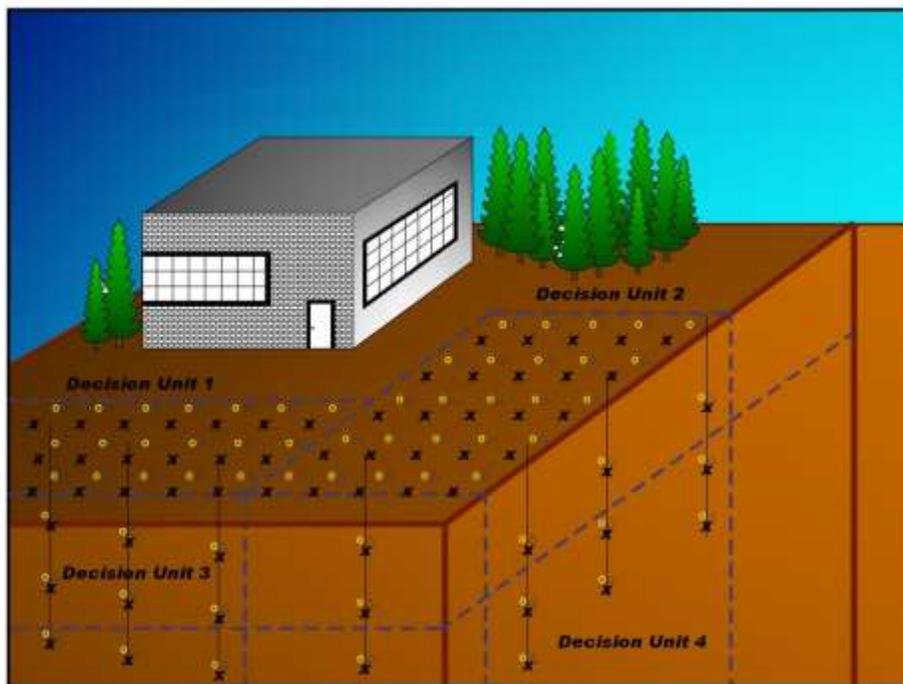




INCREMENTAL SAMPLING METHODOLOGY AND APPLICATIONS

REMEDIATION AND REDEVELOPMENT DIVISION
RESOURCE MATERIALS



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In order to promote a consistent and informed approach for Michigan Department of Environmental Quality (MDEQ) staff, this document was developed to provide information to MDEQ staff and contractors on methodology and applications for using incremental sampling techniques.

This document is available as a technical reference to assist any party interested in using incremental sampling techniques to evaluate contaminated media and make risk management decisions.

This document is explanatory and does not contain any regulatory requirements. It does not establish or affect the legal rights or obligations for incremental sampling. It does not have the force or effect of law and is not legally binding on the public or the regulated community. Any regulatory decisions made by the MDEQ regarding incremental sampling methodology and applications will be made by applying the governing statutes and Administrative Rules to relevant facts.

A handwritten signature in cursive script that reads "Kathleen Shirey". The signature is written in black ink and is positioned above a solid horizontal line.

Approved: Kathleen Shirey, Acting Director
Remediation and Redevelopment Division
January 2, 2018

Note:

For the purpose of this technical resource document, the term “facility” is being used as a general reference to a property with environmental contamination and is not intended to be applied as it is statutorily defined in the Natural Resources and Environmental Protection Act (NREPA), PA 451 of 1994, as amended.



Remediation and Redevelopment Division
Michigan Department of Environmental Quality



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SUMMARY

This document is provided as a resource for environmental professionals interested in applying Incremental Sampling (*IS*) methodology. *IS* is a structured sampling and analytical method designed to obtain a sample that is representative of the entire volume of environmental media targeted for sampling, while providing reproducible results for improved decision making. *IS* may be used at sites of environmental contamination alone or in combination with other sampling protocols. For purposes of these resource materials, *IS* applies to the evaluation of average contaminant concentrations in solid materials including soils, fill, and sediments within a horizontally and vertically defined area determined by utilizing the data quality objectives (DQO) process. The DEQ has successfully applied *IS* methodology to the investigation of organic and inorganic contaminants in these solid media.

This document is not intended to comprehensively describe all details and applications of *IS*. This document expands on information that may be difficult to obtain from other resources, and is brief in regard to subjects that are discussed at great length in available resources (see Appendix A for additional resources). *IS* is also referred to as “Multi-Incremental Sampling (MIS[®])”, “Incremental Composite Sampling (ICS)”, or “Incremental Sampling Methodology (ISM)” in the literature. The Interstate Technology and Regulatory Council (ITRC) document titled “Incremental Sampling Methodology” represents the most comprehensive *IS* reference available at this time and is recommended as a useful *IS* resource (ITRC 2012).



1.0 INTRODUCTION

Incremental Sampling (*IS*) is a combined field sampling and laboratory sample processing protocol designed to provide an unbiased, statistically valid estimate of the mean contaminant concentration (within a pre-defined volume of media). *IS* reduces the data variability commonly associated with traditional discrete field sampling practices and laboratory methods.

IS produces an unbiased and reproducible data set when properly implemented. The *IS* methodology produces data that is typically more representative of soil, fill material and/or sediment contaminant concentrations in a specified volume/area than that achieved by using traditional discrete sampling practices.

The purpose of this document is to describe, in general terms:

- *Incremental Sampling Concepts*
- *Systematic Planning and Data Quality Objectives (DQOs)*
- *Decision Unit (DU) Designation and Sampling Design*
- *Field Implementation*
- *Laboratory Processing and Analysis*

It is not the intent of this document to provide a detailed discussion of all *IS* concepts or strategies. A list of selected references, which provide a more thorough discussion of the concepts presented in this document, is presented in Appendix A.

2.0 SAMPLING CONCEPTS

2.1 *IS* and Theory of Sampling®

The understanding of the Theory of Sampling and what makes a representative sample is critical for sample collection. Knowledge of this theory is important for management, planners, sampling staff, laboratory analysts, and data users. The Theory of Sampling, its principles and practices, are applicable to any sampling situation and all media.

The Theory of Sampling describes and evaluates all errors in the sampling of materials as well as methods for minimizing error. Primary sources of errors in sampling include material heterogeneity and non-representative sampling. Material heterogeneity is comprised of: (1) the makeup of the material (compositional) and (2) the nonrandom spatial or temporal distribution (distributional heterogeneity) of elements within the material.

Compositional error is minimized by collecting more mass. Distributional error is minimized by increasing the number of samples from the population (i.e., predetermined area and volume). To control bias, correct sampling methods employ the following two principles:

1. Every element within the population has an equal chance of being in the sample
2. The integrity of the sample is preserved during the entire sampling process.

Therefore, a representative sample is one where both error and bias are controlled to an acceptable level.



2.2 *IS* Concepts

IS is a detailed sampling method that adheres to the Theory of Sampling to obtain a representative sample from a predetermined area and volume (i.e., DU). *IS* includes the collection of an appropriate mass, number of increments (i.e., portions of a DU sample), and a defined sampling protocol that includes quality control and procedures for maintaining sample integrity and minimizing error.

The high variability of data that is often observed between samples that are collected from a site using discrete field sampling methods can primarily be attributed to the particulate nature of the soils, fill material and sediment media (i.e., compositional heterogeneity) and variability in the spatial distribution of contaminants throughout a site (i.e., distributional heterogeneity). Compositional heterogeneity describes the variability of contaminant concentrations between the particles that make up the sample population and introduces what is called “fundamental error”. *IS* minimizes this source of error by ensuring that adequate sample mass be collected in the field and subsequently subsampled and analyzed by the laboratory in a manner that maintains representativeness.

Distributional heterogeneity is a function of spatial and temporal variability because contaminant particles are not randomly distributed across the sampled population. Distributional heterogeneity introduces “grouping and segregation error” when the sample consists of too few, or spatially and/or temporally biased, increments to adequately represent the variability of the population. *IS* addresses this source of error through the collection of multiple, systematically random sample increments. There are three fundamental elements necessary to properly conduct site characterization using *IS*. These are (1) systematic planning, (2) proper field sample collection, and (3) proper laboratory processing, subsampling, and analysis. A critical component of the systematic planning process is defining the volume of interest (i.e., the DU) that will be sampled using *IS*. The DU is site-specific and typically represents the smallest volume of environmental media about which a decision will be made. The *IS* method produces a single sample that will ideally have the same composition as the DU. To obtain an *IS* sample, 30 to 100 systematically random, equal volume and shape increments are collected from throughout the DU and combined to make a single (1 to 5 Kilogram [kg]) sample. In the laboratory, the *IS* sample is processed following *IS* protocol for subsampling to produce a representative aliquot for laboratory analysis. A minimum of three replicate *IS* samples are required to evaluate sampling precision. Replicate samples may be collected at any point in the sampling process (field sample, mass reduction, laboratory etc.).

3.0 *IS* VS. COMPOSITE SAMPLING

Composite sampling is a technique that physically combines a number of spatially discrete samples from a body of material into a single sample for analysis. *IS* samples are a type of composite sample. However, traditional composite samples as collected in the environmental industry, typically consist of too few increments, too small a sample mass (both field and analytical), and an insufficient laboratory subsampling to address the problems of variability inherent in contaminated soils, fill material and sediments.

IS addresses the issues problematic to traditional composite sampling, thereby becoming a different sampling method that provides significantly improved results. A reoccurring problem with traditional composite sampling is that it does not adequately address the issue of common contaminant heterogeneity, at the site, or in the lab. The *IS* process addresses heterogeneity by considering the number of increments, the mass of the sample, the size and shape of the specific volume of interest



(DU), and a project-specific laboratory protocol for processing. As such, *IS* represents a more rigorous form of sampling designed to produce results that are representative, reproducible, and defensible. Sampling methods that include “some of the elements of *IS*” have been proposed and used in the State of Michigan. These alternative sampling methods have provided mixed results. A major benefit of using *IS* is the confidence provided to site management (and other decision makers) by obtaining a representative, reproducible and defensible site characterization. The benefits of *IS* should not be expected unless the fundamental elements of *IS* are used, as described in this document. For example, collecting fewer than the recommended number of increments has been shown to result in significantly decreased data quality. Alternative sampling methods that do not incorporate the minimum expectations of *IS* (as described in this document) should not be referred to as *IS*.

IS is a proven method which provides substantial improvement in data quality representative of a site-specific area/volume when compared to traditional discrete or composite sampling. Additionally, because *IS* requires fewer analyses and less sample handling compared to effective discrete sampling schemes utilizing many samples, *IS* is often more cost effective.

4.0 SYSTEMATIC PLANNING AND DATA QUALITY OBJECTIVES

4.1 Systematic Planning

Systematic planning is a planning process that is based on the scientific method and includes concepts such as objectivity of approach and acceptability of results. Systematic planning is based on a common sense, graded approach to ensure that the level of detail in planning is commensurate with the importance and intended use of the work and the available resources. Systematic planning is at the core of the *IS* methodology.

4.2 Data Quality Objectives (DQO) Process

The United States Environmental Protection Agency (USEPA) seven-step data quality objectives (DQO) process is the recommended systematic planning tool for developing effective sampling and analysis plans. The DQO process should involve all stakeholders including the regulatory agency, owners, consultants and concerned parties. Use of the DQO process is often overlooked or abbreviated. Use of the DQO process is intended to reduce the need for additional sampling events, collecting more samples than necessary and/or disagreements about data interpretation.

A summary of the multi-step DQO process is presented below.

1. State the problem.

The first step includes describing the problem in a clear, uncomplicated manner within its regulatory context. It involves discussions and developments of the Conceptual Site Model (CSM), the identification of team members and decision-makers, as well as, defining the budget and schedule constraints.

2. Identify the decision.

This step identifies the principal study question(s) and defines alternative actions that might be taken depending on the results of the study. The output of this step is a decision statement or set of statements that link the principal study question to potential actions that will resolve the problem.



3. Identify inputs to the decision.

This step uses the decision statement(s) to identify the data needed to make the decision. The type and source of information are discussed. Establishing threshold criteria to be used for choosing alternative courses of action may also be identified. Identification of the appropriate sampling and analysis methods for meeting data requirements may also be identified.

4. Define the study boundaries.

This step includes defining and determining the population(s) of interest, the spatial boundaries within which data will be collected, the time frame for data collection, practical constraints on collecting data, and the smallest subpopulation, area, volume, and/or time for which separate decisions must be made. This step is where DUs are identified.

5. Develop a decision rule.

This step converts the decision statement (from step 2) into a decision rule. The Decision Rule includes the statistical parameter of interest (e.g., Upper Confidence Limit [UCL] of the mean), the action level(s), and integrates previous DQO outputs into “if-then” statements that will be used to guide decision making in regard to various alternative actions.

6. Specify tolerable limits on decision errors.

One challenge in developing sampling designs is to balance the potential for decision errors against the practical constraints of site investigations. All environmental data (as well as the decisions based on that data) include uncertainty. The purpose of this step is to determine how much uncertainty can be tolerated while still making sound and defensible decisions (i.e., the tolerable limit on decision errors).

Questions often explored in the process of completing this step include:

- How much confidence in the decision is required?
- What are the consequences of making an incorrect decision?
- What range of DU compositional and distributional heterogeneity can be expected?
- How close is the actual DU mean to the decision criteria?
- Are the populations adequately defined?

7. Optimize the design for obtaining data.

The final step of the DQO process uses the results of the first six steps to select and design a specific sampling program to achieve the desired goals at the lowest cost. This includes choosing final DU dimensions, the number of increments per DU, where replicate samples will be collected, and determining specific field and laboratory analytical methods.

5.0 DECISION UNIT DESIGNATION AND SAMPLING DESIGN

5.1 Introduction

IS is a method for estimating the mean concentration of contaminants in specified area/volumes called DUs. The DU represents each area/volume for which a decision will be made. Each DU is characterized by systematically collecting a predetermined number of increments which are combined to form the incremental sample. For additional information on DUs and Sampling Design refer to the ITRC “Incremental Sampling Methodology” document (ITRC 2012).

An effective sampling design (including DU development and number of increments) is dependent on a successful DQO process and a well-developed CSM. The CSM presents the current understanding of



the site, evaluates migration and exposure pathways, identifies potential data gaps, and assists in coordinating sampling strategies for achieving investigation objectives. Some investigation objectives may include:

- Characterization of source areas/releases.
- Delineation of the extent of contamination.
- Characterization of waste/fill material
- Determination of exposure concentrations.
- Confirmation sampling.
- Establishing background concentrations.

5.2 Decision Unit (DU)

Determining the size, shape, location, and number of DUs is one of the most critical components of the *IS* planning process. All involved parties should agree on the size, configuration and location of DUs. When considering the size of any DU, it must be understood that the entire DU will pass or fail based on the DU sample results (as established by the DQO process). The DU should represent the smallest area/volume for which a decision is to be made as established by the DQO process.

DUs may be based on the known or suspected locations and dimensions of source areas, or on the size of exposure areas used in risk scenarios. The shape and size of DUs should consider:

- Areas that establish exposure areas
- Contaminant transport and exposure pathways
- Spatial distribution of contaminants
- Geologic and other physical characteristics (e.g., formation and soil type boundaries)

A DU source area is a distinct area/volume containing elevated or potentially elevated concentrations of contaminant(s) in soils, solids and sediments as indicated by site history and the CSM. These may include areas where:

- Stained soils or contaminated soils are thought or known to exist
- Releases are thought or known to have occurred.
- Contaminants or contaminated material were suspected to be stored, handled, and/or disposed.
- Sampling data has identified elevated concentrations over a specific volume/area.

A DU exposure area is often defined as an area where receptors could come into contact with contaminants. DUs based on exposure areas are an invaluable tool in risk-based decision making. Exposure areas may include:

- Residential yards
- Schools, playgrounds, and parks
- Gardens and agricultural fields
- Non-residential lots
- Receptor home ranges

The primary use of *IS* data from an exposure area is to estimate the average exposure and, subsequently, chronic risk to human health or impact on the environment. Therefore, the exposure area DU should be based upon the area where exposure is or potentially could occur. The size and placement of exposure areas also depend on current use and/or proposed future use of the site. Site-



specific information and the CSM should be used in designating exposure. In situations where future land use is uncertain, the location of future residences and areas of known and/or suspected contamination may need to be addressed with appropriate sized DUs to account for potential future exposure and risk.

Particular sampling challenges exist for the following situations:

- Exposure areas with acute toxicity risks: The individual DUs within the exposure area may be so small that a large number of DUs will have to be sampled to make decisions about the exposure area that it may not be practical
- Risks from vapor sources: The individual DUs within the exposure area may be so small that sampling would not be practical. In addition, the vapor intrusion pathway considers vapor sources from multiple media; therefore, evaluation of the vapor intrusion pathway using concentrations of contaminants found solely in soils (or other solid media) is not appropriate.
- Contaminant conditions that change significantly over time (high variability): The variability of the contaminant should be considered and potentially addressed in the sampling protocol

To ensure sample correctness, consistent with the Theory of Sampling, every DU within the exposure area has to be assessable. Sampling is not recommended if the CSM establishes DUs that cannot be correctly sampled, regardless of the method, because the errors in the sampling process are unavoidable.

5.3 Residential and Non-Residential Exposure Area DUs

Exposure areas for residential use can vary in size (e.g., ¼ acre lot to 1 acre lot); however, if there are smaller areas within the exposure area that receive higher use in an exposure area, that area should be considered for evaluation as a separate DU. Swing sets and sandboxes in residential yards are examples of such exposure areas.

Exposure areas for non-residential properties are site-specific. Designation of these exposure areas should be determined during the DQO process. It may be useful to designate DUs and evaluate properties for future land use (e.g., residential land use). This may help avoid unnecessary land use restrictions and/or the need for reinvestigation should future development plans call for a more sensitive land use.

5.4 DU Sampling Design

Once the DUs are determined, the sampling design should be developed consistent with the DQO requirements. DU sampling design, and in particular the number of increments appropriate for sampling, takes into account the objectives of the site investigation including the type and quality of information needed to make the decision. A minimum of 30 to 50 increments per DU is recommended to obtain a representative and reproducible estimate of the mean concentration in a DU that is characteristic of moderate heterogeneity. Depending on the degree of uncertainty that can be tolerated within the project-specific DQOs, fewer increments may result in unacceptable uncertainty and decision errors. A larger number of increments (e.g., 60 to 100) and/or a larger sample mass may be necessary where higher levels of heterogeneity are expected. Sampling design concepts are provided in the next section.



6.0 FIELD IMPLEMENTATION

6.1 Introduction

This section addresses *IS* field practices. To help ensure data quality, it is recommended that all field sampling and field processing activities be performed or supervised by personnel trained in *IS*.

6.2 Sample Planning

The first step in successfully implementing *IS* is to complete the DQO process as described in Section 4.2. Once the specific objectives have been set, the proper sampling plan can be prepared. The sampling plan will take into consideration the mass of the sample needed, the number of increments, increment spacing, the depth of the increments, and the sample tool(s) to be used. Proper planning of the *IS* sampling procedures will ensure that a representative sample will be collected and that the physical sampling process will be conducted efficiently and without complication.

The total mass of an *IS* sample and the number of increments are dependent (in part) on the heterogeneity of the DU. Between 1 and 2 kg (for the total sample) and a minimum of 30 to 50 increments per DU is recommended for a DU that is characteristic of moderate heterogeneity. A sample mass of 2 kg or more and up to 100 increments may be needed for highly heterogeneous DUs.

6.3 Sampling Tools

The sample mass, number of increments, and increment depth can be determined through the DQO process, and the appropriate sampling tool can be determined based on the mass per increment and soil type expected to be encountered. The following tables (Table 1 and 2) can help in determining how to incorporate these factors to plan an appropriate *IS* sampling approach. Table 1 shows an estimate of the increment mass needed to provide initial sample masses of 1 to 2 kg based on the number of increments collected. Table 2 shows the mass obtained by using typical soil coring tools.

Table 1
Estimated Mass/Increment (grams [g])

		Sample Mass	
		1 kg	2 kg
# of Increments	30	~35 g	~70 g
	40	~25 g	~50 g
	50	~20 g	~40 g
	60	~17 g	~35 g
	80	~13 g	~25 g
	100	~10 g	~20 g

Table 2
Coring tool average mass per inch of core*

Core tool diameter	0.75 inch	1.0 inch	1.5 inch	2.0 inch
Mass/inch of core	~7 g	~12 g	~28 g	~50 g

*The mass identified in Table 2 applies to unsaturated fine to medium textured soils. Very moist to wet soil may require more mass to account for the additional water content.



As an example, if the DQOs require a minimum 1 kg sample and 50 increments at a 2 inch sampling depth, at least 20 g per increment would be required. Using a 1.0 inch diameter sample corer at a depth of 2 inches would provide approximately 24 g of soil per increment. This would produce the desired initial sample with an approximate 1.2 kg sample mass.

The cohesiveness and composition of the soil substrate should also be considered in choosing an appropriate sampling tool. The sampling tool should obtain cylindrical increments of a constant depth throughout the vertical increment, and should equally retain all particle sizes. The diameter of the sampling tool should be a minimum of three times the diameter of the largest particle present. In general, sampling tools should have a minimum diameter of at least 16 millimeters (mm). For less cohesive soils, such as dry sands, retainers may be needed so that the entire, complete core increment is retained.

For *IS* sampling projects where cylindrical coring samplers are not appropriate (i.e., non-cohesive soils, wet sediments, etc.), scoops or other devices can be used. However, care should be taken to obtain a “core-shaped” increment over the entire depth of interest. Depending on site familiarity, one or several sampling tools should be readily accessible during all sampling activities.

There are a variety of soil coring sampling tools that are available for nonvolatile *IS* sample collection. Many of these are commercially available and custom made tools can be designed for specific sampling needs. *IS* sample collection of volatile organic compounds (VOCs) can be accomplished using the same coring device used for the regular collection of methanol preserved VOC samples.

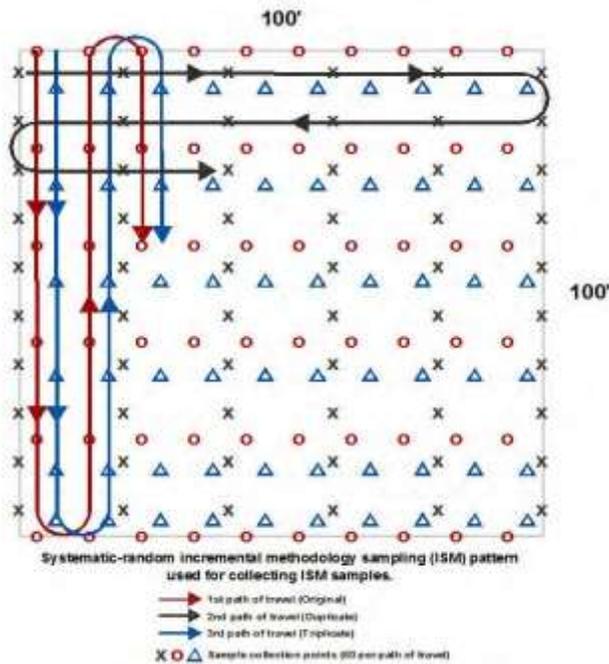
6.4 Field Collection

The field collection process can be made effective and efficient with appropriate planning. This is achieved after the DQO process and a determination of DUs, sample mass, depth, number of increments, and types of sampling tools are considered.

6.4.1 Surficial *IS* Samples

Surficial *IS* samples are generally the easiest type of *IS* sample to collect. Surficial *IS* samples should be collected in a random systematic approach throughout the DU. The positioning of the first *IS* point can be randomly selected (typically toward a corner of the DU) then the remaining *IS* points are dictated by a sampling grid based off this first point. With this random systematic sampling approach, a grid is laid out across the DU and all soil increments are collected from grid nodes (see Figure 1).

Figure 1
Random Systematic Sampling Approach



*Figure from ITRC 2012

The grid pattern for any individual DU can be roughly determined based on the area of the DU and the number of increments required. A simple equation can be used to determine the grid pattern as follows:

$$\text{Grid spacing (feet)} = \text{square root of the area (feet}^2\text{)}/\text{square root of the number of increments}$$

Table 3 shows several grid spacing distances based on specific increment requirements and DU sizes.

Table 3
Typical Grid Spacing (feet)

		Area of DU (Acres)							
		0.10	0.20	0.25	0.5	1.0	2.0	2.5	5.0
# of Increments	30	12	17	19	27	38	54	60	85
	40	10	15	16	23	33	47	52	74
	50	9	13	15	21	30	42	47	66
	60	8	12	13	19	27	38	43	60
	80	7	10	12	16	23	33	37	52
	100	6	9	10	15	21	30	33	47

It should be noted that while a specific number of increments may be specified, collecting a few more or a few less increments is acceptable as long as the appropriate mass is collected and the DU is systematically sampled over its entirety. For example, if 30 increments are specified and the grid pattern over the DU works out to be a 6 by 6 grid, a total of 36 increments will be collected. Additionally, if 50 increments are specified and a 7 by 7 grid is used, a total of 49 increments will be collected.



An *IS* sample is initially prepared by collecting multiple increments from a specified DU and physically combining these increments into a single sample. For *IS* samples collected for analyses, other than VOCs or phthalates, the sample is usually collected in a 1 or 2 gallon sealable plastic “Ziploc” -type bag. Carrying this bag in a 5-gallon bucket will aid in the ease of collection. VOC samples should be collected in a 0.5 or 1 Liter small mouthed amber glass bottle containing adequate methanol (see below). More detail regarding samples for VOC analysis is provided below.

Surficial samples are typically collected using a “step probe” where the probe is set to collect an increment from a specific depth. The probe is pushed into the soil to the specified depth and the sample increment is extracted from the ground. This increment is then removed from the probe and placed directly into the sample bag. The process is continued throughout the DU along the grid pattern until all increments are collected. If replicate samples are required, the same grid pattern is used but it is based on a new randomly selected starting point. Note that replicate samples can be collected on the same “pass” across the DU; each replicate is simply placed in a separate sample bag. Also note that the exact location within the grid where the increment is collected is not particularly important, however, collecting each increment in the same general vicinity within each grid may improve the overall coverage of the DU.

Since all increments collected for a specified DU are combined into one sample for analysis, there is no need to decontaminate the sampling tool between increments. Sampling tools should be decontaminated between DUs or when collecting replicate samples.

Grid patterns for regularly shaped DUs (square or rectangular) are relatively easy to lay out. Depending upon the size of the DU and terrain features or other obstructions, pin flags, or other markers can be placed along the edges of the sampling grid to assist with the visual delineation of the increment sampling point. The sampler can then use these markers to either pace out or otherwise measure the specified distance between *IS* points. The use of marked string between the edges of the sampling grid is sometimes used to demarcate the sampling grid. Larger DUs may require markers at each *IS* point or the use of a global positioning system to locate each point. For odd shaped DUs, a trial run with no sample collection can be conducted to quickly establish whether the proposed sampling grid will produce the proper number of increments and, in turn, the proper mass.

While *IS* sample collection can be performed by a single individual sampler, a two-person team is often the most efficient method. A two-person team permits one person to collect the increments while the second holds the sample container (plastic bag in bucket, amber bottle for VOCs, etc.) and keeps track of the number of increments collected. Replicate samples can be collected by the team at the same time as the original sample (using an additional sampling tool) or collected after the initial sample using a tool that has been decontaminated.

6.4.2 Subsurface IS Samples

All DUs are three dimensional and the *IS* DU characterization is designed to be representative of a specified volume or mass of soil/sediment. While obtaining good spatial coverage for subsurface soils is more challenging than surficial soils, it can still be accomplished. The objectives are the same as for surface soil in that a reliable and reproducible mean estimate of contaminants present in the DU is desired.



6.4.2.1 Subsurface IS Sampling from Borings

Subsurface DUs may include pre-determined depth intervals or lithology units as determined by the DQO process. Subsurface *IS* sampling can be accomplished using a variety of boring tools that collect subsurface soil cores. These include hand tools (push probes, soil corers, etc.), as well as, powered boring/coring machines (direct push and drill rigs) that are capable of collecting soil cores at depth. Regardless of the tool used, subsurface *IS* sampling is conducted in the same manner as surficial *IS* sampling.

Cores from multiple borings within each DU are collected. Each core from the specified subsurface DU is an increment and is combined with the other increments for the *IS* sample. The individual core may be subsampled to reduce the mass of the increment. Common techniques to subsample the cores include core wedge and plug. The core wedge technique entails vertically splitting the core in half, or in quarters, along the axis of the core. The plug technique involves collecting multiple plugs from specified intervals throughout the core. The collection of field replicates in the subsurface will follow the same *IS* methodology used to produce the initial sample (see Section 6.5). Refer to the ITRC *Incremental Sampling Methodology*, February 2012 document for detailed information.

6.4.2.2 Subsurface Excavation Sidewall and Bottom Sampling

While *IS* soil samples collected from excavations are subsurface samples, excavation side walls and bottoms are accessible, which allows for the use of methods similar to *IS* surface soil sampling. The difference in the accessibility of these excavated faces may dictate the proper tool needed for collecting an appropriate *IS* sample. When conditions are safe for the sampler to physically enter the excavation, *IS* sampling can be conducted in the exact same manner as surficial *IS* sampling utilizing the same tools. Sidewall sampling can be treated as if it were a horizontal sample with regard to laying out the proper grid for collecting the individual increments.

When it is not safe for the sampler to enter the excavation (i.e., too deep, steep sidewalls, etc.), *IS* samples can be collected from the edge of the excavation using a probe or scoop tool mounted on an extendable shaft. All appropriate precautions should be taken when sampling in this manner, as there are still potential dangers when working on the edges of excavations. Sidewall grids can be marked by placing flags along the top edge of the excavation or by lowering weighted strings with systematically random marked intervals down into the excavation at the proper grid spacing.

6.4.2.3 Potential Subsurface Limitations

The recommended number of increments to be collected from a subsurface DU is the same as that for a surface soil DU. In some cases, as with direct push or drill rig borings, collecting the recommended number (30 to 100) of increments may not be cost-effective or practical. Reducing the recommended number of increments may be an option if the project DQOs are satisfied. It is important to recognize that a reduced number of increments increases uncertainty and decision error resulting in a less precise and more biased estimate of the mean contaminant concentration.

6.4.3 Volatile Organic Chemicals (VOC) IS Samples

IS samples can be collected for VOCs contaminant analyses. *IS* VOC soil samples are collected using methanol as the field preservative. The mass and number of increments for each *IS* sample is determined through the DQO process, which specifies the total amount of methanol for the number and mass of increments. This is typically determined at a 1:1 ratio of volume of methanol (ml) to mass of



soil (g). This methanol is placed in an appropriately sized (usually a 500 or 1,000 milliliter [mL]) small-mouthed amber glass bottle. The increments are placed directly into the bottle as they are collected. Each increment mass should be as similar as possible however the individual increments typically do not need to be weighed in the field during collection.

The increments collected for a VOC *IS* sample in this manner can be collected in the same manner as when collecting samples for other analyses from surface and subsurface soils. The only real difference is in the mass of soil collected for each increment. Incremental mass for VOC increments is typically only 5 to 10 g.

As with all *IS* samples, collecting a larger mass of soil for an *IS* sample results in a sample that is more representative of the material sampled. However, the handling, shipping, and costs of large volumes of methanol usually present logistical issues that dictate that *IS* VOC samples be comprised of 30 to 50 increments and have a mass of 300 to 500 g.

Increments should be collected using tools that minimize the loss of VOCs during sample collection and allow the collection of the proper mass of soil. Sample increments should be quickly transferred from the tool to the bottle containing the methanol. Syringe-type devices similar to the ones used to collect discrete VOC samples are preferable.

For determining percent moisture (for reporting dry weight), a separate and unpreserved soil sample, representative of the increments must be collected. This sample should be collected in the same manner as the *IS* VOC sample. This is usually accomplished by collecting an additional increment at each *IS* increment location and combining these increments in an unpreserved container for submittal to the laboratory.

6.4.4 Waste Pile *IS* Samples

IS may be used to characterize waste piles and soil piles (e.g., disposal or treatment options). The DU may include one or more waste piles as determined by the DQO process. If equipment is being used to move the piles from an excavation source or the piles are moved to a staging area or roll-off box, the increments may be collected from the device transporting the material (e.g., shovel, pay loader, or track hoe bucket) using a systematic random approach. Typically, a small core device is used to collect the sample increment as the material is moved. If equipment is not available to move the piles, the process may include collecting increments from throughout the vertical and horizontal extent of the pile using appropriate coring tools. Collection of *IS* samples may require a team of two or more persons to accommodate safety concerns if heavy equipment is used.

6.5 Collection of Field Replicate *IS* Samples

Replicate *IS* samples should be taken whenever there is a reduction in mass (sample) to evaluate the precision of the sampling method. Replicates are used to evaluate precision of the sampling process in the field and in the laboratory. Three or more replicate *IS* samples are required to statistically evaluate the sampling precision of any particular DU. *IS* replicate samples are collected and analyzed in the same manner as the initial *IS* sample.

The relative standard deviation (RSD) between replicates is used to assess data precision and reproducibility (and, therefore, the confidence) in the data generated. The higher the RSD the less confidence there is that the mean contaminant concentration reported accurately represents the DU(s).



Although dependent on the site-specific level of uncertainty that can be tolerated, an RSD of less than 30 percent between replicates is generally considered precise enough to make decisions.

Depending on the degree of similarity between DUs of any particular investigation, the collection of triplicates from a minimum of 10 percent of the DUs is normally recommended for *IS*. The number of replicates per DU and the frequency of replicate sampling should be clearly addressed in the DQO process and needs to consider contaminant variability, the existence of separate populations, and the precision desired. If these qualities are considerably different between DUs, then replicate sampling should be performed for each different 'DU type'. For sites with multiple similar DUs and where otherwise appropriate, replicates from one DU may be used to provide an estimate of variability that can then be extrapolated to other similar DUs. For multiple similar DUs, the DU expected to have the highest variability should be selected for replicates.

6.6 IS Field Processing for Non-VOC Samples

IS sample processing techniques, such as sieving, grinding, and subsampling, are designed to ensure that the mass of sample analyzed by the laboratory is representative of the DU. These techniques are implemented, as appropriate, to reduce data variability as compared to conventional sample handling and processing techniques. However, these techniques can introduce sampling error, especially if conducted in the field. It is recommended that all *IS* sample processing be performed in a controlled laboratory setting rather than in the field. On a specific case-by-case basis and depending on site logistics, the type of soil, the total number and/or mass of *IS* samples, etc., sample processing may be initiated in the field for some non-VOC contaminants with the appropriate precautions as noted below. Any field processing of *IS* samples should be discussed with the laboratory in advance to enable the laboratory to adjust their standard operating procedures (SOP's) to produce representative aliquots from *IS* field subsampling for laboratory analysis.

Moist samples may require air drying to facilitate sieving. If done in the field, it should be done in an appropriate dust-free location where temperatures and ultraviolet light are not expected to cause degradation of certain contaminants. Mostly sandy soil samples with little vegetation and very low moisture content can be sieved (typically using a #10 sieve, less than 2 mm particle size) in the field to remove pebbles and organic debris. Sands and smaller size particles are generally considered "soil," while larger particles are considered gravel, rocks, or other materials (e.g., sticks and roots). Field sieving might be an option when the originally collected *IS* sample has a large amount of these larger particles and a proper amount of "soil" is needed to meet the total mass required for the sample (i.e., by DQO requirements). Alternative sieve sizes may be of interest on a case-by-case basis and these should be determined through the DQO process. Unless field subsampling is to be performed, the entire sieved *IS* sample fraction should be submitted to the laboratory for appropriate additional processing and subsampling.

As noted above, laboratory subsampling is recommended in lieu of field subsampling, especially when contaminants have been deposited as solid particulates (e.g., energetics, metals at firing ranges, etc.). Field subsampling may be appropriate when the laboratory performing contaminant analysis does not have the ability to conduct the proper subsampling in the laboratory (e.g., with a mobile laboratory).

If field subsampling is to be performed, the entire *IS* sample may need to be air-dried and sieved. Field subsampling should be conducted in the same manner as it would be in the laboratory. After the needed processing, the *IS* sample should be spread out in a thin layer on an appropriate clean surface. The subsample is then obtained by collecting a minimum of 30 to 50 increments from systematic random locations in the same manner as when collecting surficial *IS* increments. The increments



collected to form the subsample should equally represent the vertical depth of the processed sample. This is best achieved by using a rectangular, flat-bottom sampling tool (scoop) with sides and a minimum of 16 mm width. Curved or spoon-shaped sampling tools should be avoided as they will introduce greater bias into the subsampling process. The mass of the subsample required will be dictated by the requirements for the analytical test(s) needed. Replicates of the field processed soil should be collected and submitted for analysis to evaluate the precision of the *IS* field processing procedure.

If it is determined in the field that the total mass of an *IS* sample is too great, simply dividing or splitting the sample into separate volumes for analysis is not an acceptable method of mass reduction. While extra mass can add to the cost of laboratory sample processing, field sample processing in this manner would add unnecessary error to the sample results. Therefore, it is important to correctly estimate the number and volume of increments to achieve the desired total sample mass.

7.0 IS LABORATORY PROCESSING AND ANALYSIS

7.1 Introduction

The procedures used in the laboratory to prepare and analyze samples are as important as how the samples are collected in the field. The laboratory should be involved with project planning and the DQO process. The laboratory's standard operating procedures should be sufficiently developed to obtain representative subsamples from the field generated *IS* sample mass. Improper laboratory processing and subsampling increases error, which may cause failure to meet the DQO. The reader is referred to the Laboratory Sample Processing and Analysis Section of the ITRC Guidance (ITRC 2012) for more detail on laboratory methods (including Quality Assurance/ Quality Control) and processing options.

7.2 Laboratory Sieving (Non-VOCs)

The entire incremental sample is normally submitted to the laboratory for processing and analysis. In the laboratory, the sample is typically air-dried and may be sieved (typically at 2 mm). The less than 2 mm sized soil particles are generally considered "soil" and are of most interest for contaminant analysis while larger particles are considered gravel, rocks or other materials (e.g., sticks and roots). Sieving the soil sample to the less than 2 mm size also establishes the maximum particle size of the sample, which may be necessary to determine the minimum aliquot mass necessary for extraction/analysis in the laboratory (see below). Although sieving to the less than 2 mm particle size is typical, there may be contaminant investigations or analyses where alternate particle sizes may be of interest (e.g., lead). In these cases, the rationale for sieving to other specific particle sizes (and associated changes to laboratory processing/analysis) should be addressed in the project DQOs and the sampling plan.

7.3 Laboratory Subsampling (Non-VOCs)

Proper subsampling in the laboratory provides a representative sample (aliquot) for analyses. In the laboratory, the sample is either subsampled by hand or mechanically using a sectorial splitter (also called a rotary riffle splitter). When subsampling by hand, the entire dried and sieved sample is spread out in a thin layer (slab cake). Using a systematic random sampling scheme, 30 to 50 increments are selected from the slab cake. The mass of the aliquot (subsample) needed for all of the analytical tests is used to determine the mass of each increment.



7.4 Aliquot Mass

The final subsample mass (i.e., the aliquot mass) must be used completely in the analytical preparation step. The ITRC and the USEPA SW-846 Method 8330B (USEPA a,b) guidance documents discuss the minimum aliquot mass required to reduce "Fundamental Error" of the laboratory analyses to a minimum (e.g., 15 percent or less). The appropriate aliquot mass is (in part) based on the maximum particle size in the soil sample, with both greater mass and/or reduced particle size acting to reduce the fundamental error. Although the ability to increase the aliquot mass must be balanced with DQO requirements, laboratory analytical methods that use an aliquot mass of 10 g or more are generally preferred. This may, however, be dependent on the analytical method used, the laboratory equipment available, and other factors. Laboratory methods may need to be modified to meet project DQOs as determined by advance consultation with the selected laboratory. The ITRC guidance document has several suggestions for increasing the sample aliquot mass.

7.5 Grinding and Milling

Grinding and milling are options to reduce fundamental error. Grinding is used to achieve a reduction in particle size. Milling is used to achieve particles of uniform small size. Grinding/milling samples also reduces the potential for segregation error. These are services that laboratories may offer. Thermal stability and volatility of the contaminant(s) should be considered; therefore, grinding/milling is not recommended for VOCs and certain SVOCs. Grinding/milling may not be appropriate for samples being analyzed for bioaccessibility/bioavailability.

7.6 Laboratory Processing for Volatile Hazardous Substances

IS soil samples are collected for volatile contaminant analyses per a modified version of SW-846 Method 5035A (EPA a,c) and analytical Method 8260B (USEPA a,d). Typically, *IS* soil samples collected for volatile contaminant analysis include 30-100 increment soil plugs inserted into a "1 ml/1 g" corresponding volume of methanol (e.g., 40 10 g plugs into 400 mL of methanol). Replicate samples would be collected and analyzed separately. Alternatively, individual increments can be preserved in 40 mL vials with 10 mL of methanol per 10 g of soil. This may facilitate commercial shipping because the individual container volume stays below 30 mL. The methanol is then composited at the lab to produce the methanol extract for the entire DU sample.

A separate, unpreserved soil sample for percent moisture determination should be collected if necessary to report the results on a dry weight basis. Typically, the unpreserved soil sample should be collected in the same manner as the *VOC* samples, with an additional increment collected at each *IS* increment location and placed in an unpreserved container of adequate size and submitted to the laboratory. The subsample to be used for percent moisture determination should be collected using the hand sampling (2D slabcake) process (described above) on the sample prior to air drying.

7.7 Laboratory Processing for Semi-Volatiles, Polychlorinated Biphenyls (PCBs), Pesticides, Herbicides, Energetics, and Other Hazardous Substances

Some contaminants (including SVOCs, PCBs, pesticides, herbicides, phenols, energetics, and certain metals (Arsenic, Mercury, and Lead)) may require special laboratory and *IS* field processing and subsampling methods to avoid sample contaminant loss and promote sample representativeness. The DQO process should evaluate field collection procedures and laboratory subsampling methods for all contaminants of concern. Advance consultation with the selected laboratory is essential for the DQO planning process.



7.8 Scheduling Laboratory Analysis

During the preparation of the DQOs there should be discussion with the laboratory personnel about laboratory methods, capacity, and sample scheduling. Currently, samples for DEQ investigations are handled by the Contract Laboratory procedure. The DEQ contract pricing for *IS* related services is available. Samples may be sent to the DEQ laboratory for shipment/transportation to a contracted laboratory.

8.0 RRD PROGRAM APPLICATIONS

8.1 Contaminated Site Remediation, Brownfields, Stockpiles and Sediments

IS has been used to advance progress at contaminated sites in Michigan (e.g., under Part 201 and Part 213). *IS* has been used successfully in the Brownfield Redevelopment Program to evaluate direct contact risks associated with arsenic and lead for defined areas and to identify areas where other contaminants of concern are located. *IS* has been used increasingly in Michigan where large scale developments are being designed for residential (and other) uses, while evaluating the necessary property restrictions and exposure controls. *IS* has also been used in Michigan for characterizing stockpiled soils and waste to determine suitability for re-use versus disposal. *IS* has been used to evaluate contaminant mass in sediments for streams and rivers.

8.2 Baseline Environmental Assessments (BEAs) and Due Care

IS is a sampling method that may be used for conducting a BEA for a property and for determining or demonstrating Due Care for some exposure pathways. The increased accuracy, precision, and reproducibility typical of *IS* (in comparison to discrete sampling) may make *IS* the preferred sampling method for meeting these objectives. *IS* sampling techniques can also be useful in determining where exposure risks may exist on smaller portions of the property, allowing for the implementation of protective measures that may be necessary.

9.0 Conclusion

If the *IS* process is followed, it can provide more representative and reproducible results than other traditional discrete sampling methods. This translates into better decision making within RRD programs to the benefit of all interested parties. While the RRD has employed *IS* methodology in a variety of program applications, additional *IS* applications continue to evolve. Please contact RRD staff for assistance in applying *IS* for meeting program objectives.



Acronym List – IS

Acronym	Definition
BEA	Baseline Environmental Assessment
CSM	Conceptual Site Model
DEQ	Department of Environmental Quality
DQO	Data Quality Objectives
DU	Decision Unit
EA	Exposure Area
EPA	Environmental Protection Agency
ESA	Environmental Site Assessment
g	Gram(s)
/S	/ncremental Sampling
ICS	Incremental Composite Sampling
ISM	Incremental Sampling Methodology
ITRC	Interstate Technology and Regulatory Council
Kg	Kilogram
MDEQ	Michigan Department of Environmental Quality
MIS	Multi-Incremental Sampling [®]
mL	milliliter
ml	methanol
mm	millimeter
PCBs	Polychlorinated Biphenyls
NREPA	Natural Resources and Environmental Protection Act, 1994 PA 451, as amended
Part 17	Michigan Environmental Protection Act, of the NREPA
Part 31	Water Resources Protection, of the NREPA
Part 201	Environmental Remediation, of the NREPA
Part 213	Leaking Underground Storage Tanks, of the NREPA
Part 615	Supervisor of Wells, of the NREPA
Part 625	Mineral Wells, of the NREPA
RRD	Remediation and Redevelopment Division
RSD	Relative Standard Deviation
SOP	Standard Operating Procedure
SVOC	Semi-Volatile Organic Compounds
UCL	Upper Confidence Level
USEPA	United States Environmental Protection Agency
VI	Vapor Sources (in text) or Vapor Intrusion
VOC	Volatile Organic Compounds



Appendix A: REFERENCES and ADDITIONAL RESOURCES

REFERENCES

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<http://www.itrcweb.org/ism-1/>

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Laboratory Processing and Analysis

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ADDITIONAL RESOURCES

Incremental Sampling Methodology

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