Comments

Remediation and
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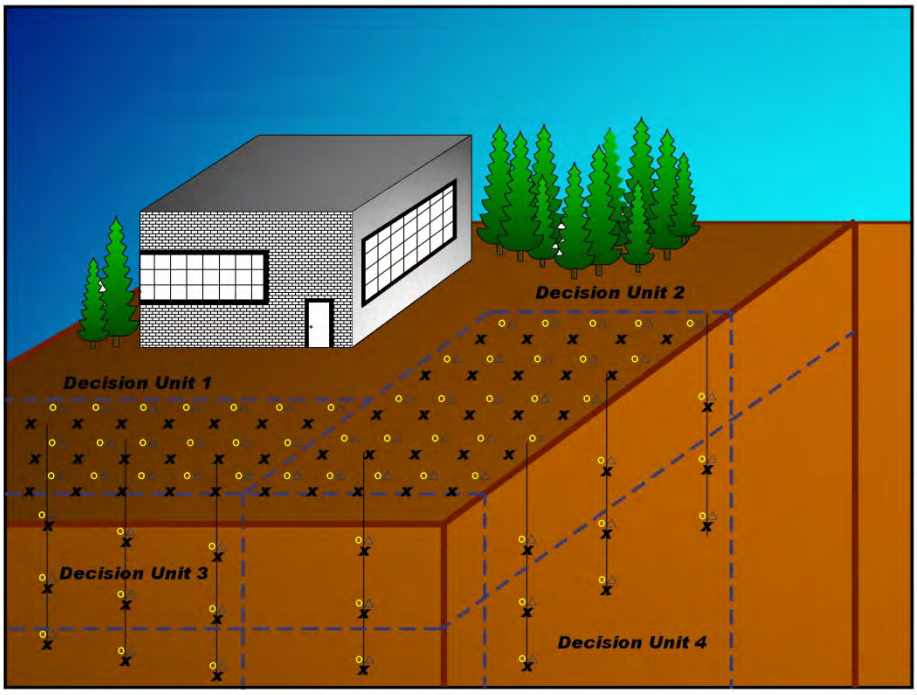
Michigan Department of Environmental Quality

INCREMENTAL SAMPLING METHODOLOGY AND APPLICATIONS

REMEDIATION AND REDEVELOPMENT DIVISION RESOURCE MATERIALS



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Protecting Michigan's Environment. Ensuring Michigan's Future.

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.

Approved: _____

Robert Wagner, Chief
Remediation and Redevelopment Division
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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 protocol designed to obtain a sample that is representative of the entire environmental media targeted for sampling, while providing reproducible results and improved decision making. *IS* is a suitable tool that may be used at sites of environmental contamination and may also be used to complement other sampling protocol.

This document is not intended to comprehensively describe all details and applications that are appropriate to using *IS* when evaluating sites of environmental contamination. 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. Section 7 of this document includes several references for available resources. *IS* may also be 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 single *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 that is designed to provide an unbiased and statistically valid estimate of the mean value of a contaminant (within pre-defined boundaries) at a site. IS reduces or limits the data variability commonly associated with more traditional discrete field sampling practices and laboratory methods. When properly executed, IS produces unbiased and reproducible data that, in most cases, are more representative of the contaminant concentrations at contaminated sites than could be achieved using traditional discrete sampling practices. As a result, the IS approach can provide better information from which decisions regarding contaminant concentrations at a site will be made.

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

- *IS Concepts*
- *Systematic Planning and Data Quality Objectives*
- *Decision Unit 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 *IS* CONCEPTS

Sampling of environmental media is typically performed to characterize a site of known, or suspected, contamination. Historically, the majority of environmental sampling has been conducted using discrete, and often biased, sampling practices. Common deficiencies that may be associated with the exclusive use of discrete sampling methods include poor spatial coverage, inadequate sample density, or data that cannot be used to statistically represent the entire area of interest (i.e., with a reasonable level of confidence). Discrete sampling often produces a sample data set that is not representative or reproducible, and therefore, difficult to make decisions with, and to defend those decisions with. Common laboratory subsampling methods can add additional error. The analysis of a “grab” aliquot from the field sample may not be representative of the sample submitted for analysis, let alone the area of a site that decision makers hope to represent. *IS* was developed to address these and other limitations of traditional sampling and laboratory analysis methods.

The high variability of data that is often observed between samples that are collected from a site using traditional discrete field sampling methods can primarily be attributed to the particulate nature of the media (compositional heterogeneity) and variability in the spatial distribution of contaminants throughout a site (distributional heterogeneity). Compositional heterogeneity describes the variability of contaminant concentrations between the particles that make up the sample population and introduces “fundamental error” when insufficient sample mass is collected and analyzed. *IS* minimizes this source of error by ensuring that adequate sample mass was 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 variability and occurs when 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 biased increments to adequately represent the spatial variability of the population. *IS* addresses this source of error through the collection of multiple and systematically random sample increments.

There are three fundamental elements necessary to properly conduct site characterization using ISM. These are: (1) systematic planning, (2) proper field sample collection, and (3) laboratory processing, subsampling, and analysis of the sample. A critical component of the systematic planning process is defining the area of interest (i.e., the decision unit [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.

IS is designed to obtain a single sample that has all constituents in the same composition as the DU. In the field, approximately 30 to 100 systematically random, equal volume increments are collected across the entire DU and combined into a single 1 to 5 Kilogram (Kg) *IS* sample. In the laboratory, the entire *IS* sample is processed in an effort to produce an aliquot for analysis that has the same composition as the sample. According to established guidelines, a multi-increment subsample (generally 10 to 30 grams [g]) is collected similarly, but on a much smaller scale, as the original *IS* sample. The entire subsample is then used for analysis. A minimum of three replicate *IS* samples from a DU can provide sufficient information to evaluate sample variability. When evaluating multiple DUs across a site, it may be appropriate to apply the replicate statistics from one DU to other DUs.

2.1 IS vs. Composite Sampling

Composite sampling is a technique that combines a number of discrete samples collected from a body of material into a single sample for analysis. This basic description may represent the only similarity to *I*S. Composite sampling poses greater limitations and inappropriate use may render the resulting data meaningless. The limitations of composite sampling can undermine an otherwise effective effort to determine the nature and extent of environmental contamination.

IS addresses the issues problematic to composite sampling, thereby becoming a different sampling method that provides significantly improved results. A reoccurring problem with composite sampling is that it does not adequately address the issue of common contaminant heterogeneity, at the site, and in the lab. The *IS* process controls for heterogeneity by considering the number of discrete subsamples (increments) (typically much greater than composite sampling), the sample and aliquot mass, the size and shape of the specific area of interest (DU), and a project-specific laboratory protocol of physical and chemical processing (e.g., aliquot mass). As such, *IS* represents a more rigorous form of sampling designed to produce results that are representative, reproducible, and defensible. *IS* provides a completely different level of data quality than traditional composite sampling provides.

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. The more successful of these "alternative sampling methods" were achieved by using the systematic planning aspects of *IS*. 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 when "something less" than *IS* is used. For example, collecting fewer than the recommended number of increments has been shown to result in significantly decreased data quality. The uncertainty and decreased data quality that results from this compromise (often associated with traditional composite sampling protocols) has resulted in less than adequate results. Alternative sampling methods that do not incorporate the minimum expectations of *IS* (see below) should not be referred to as *IS*. The benefits of implementing the proven method of *IS* in lieu of assuming or evaluating an alternative method as the comparative level of effort associated with implementing *IS* is usually negligible in comparison to the substantial improvement in data quality and decision making.

3.0 SYSTEMATIC PLANNING AND DATA QUALITY OBJECTIVES

3.1 Systematic Planning

Systematic planning is a fundamentally important and necessary step toward achieving success with *IS*. This involves a series of steps that help ensure the investigation results provide the information necessary for choosing the appropriate response actions. More specifically, systematic planning is used to determine the type and amount of data to be collected, the locations to be sampled, and the field and laboratory methods that will be used.

3.2 Data Quality Objectives (DQO)

The United States Environmental Protection Agency's (EPA)s DQO process serves as a good resource as it produces quantitative and qualitative statements that express the project-specific decision goals, which are used to guide the sampling and analysis plans. (EPA DQO 2000 reference at http://www.epa.gov/quality/qs-docs/g4hw-final.pdf and EPA DQO 2006 reference http://www.epa.gov/quality/qs-docs/g4-final.pdf) The DQO process is a systematic and flexible planning process. The DQO process was developed by the EPA to provide a common structure and terminology to practitioners designing environmental sampling plans.

A summary of the *IS* DQO seven-step process is presented below:

1. State the problem.
   The first step includes describing the general problem in a clear, uncomplicated manner within its regulatory context. It involves initial discussions and developments on the Conceptual Site Model (CSM). It also includes 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 any consideration of alternative actions that might be taken depending on the outcome of the study. These discoveries can be used to develop what is referred to as "decision statements." The output of this step is a decision statement or set of statements that link the principal study question to possible or potential actions that will resolve the problem.

3. Identify inputs to the decision.
   This step uses the decision statement(s) to identify what data are needed to make the decision. The "type" of information and the "source" of information are discussed. Decision criteria or "action levels" that may be used to choose among alternative courses of action are identified. An

identification of appropriate sampling and analysis methods are considered for meeting data requirements.

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

5. Develop a decision rule.
   This step converts the decision statement into a decision rule, with the decision rule based on the expected inputs to the decision. Select an appropriate population parameter (e.g., the mean) that will be estimated based on the data collected for each DU. Verify that the decision value (e.g., action level) will be clearly identifiable given the selected parameter and the data sources that will be used. Formulate "if−then" statements that will be used to guide decision making.

6. Specify tolerable limits on decision errors.
   The purpose of this step is to determine how much uncertainty can be tolerated while still making sound and defendable decisions. A determination of how much uncertainty can be tolerated is important to site decision makers, because the environmental data upon which site decisions are based have uncertainties associated with them. One challenge in developing sampling designs (see step 7) is to balance the potential for decision errors against the practical constraints of site investigations.

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

   - How much confidence is desired or 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 estimated mean DU parameter value to the decision criteria?
   - Are 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 least cost. This includes choosing the number of increments, final DU dimensions, and determining specific field and laboratory analytical methods.

4.0 DECISION UNIT DESIGNATION AND SAMPLING DESIGN

4.1 Introduction

IS is a method for estimating the mean concentration of contaminants in specified areas called Decision Units (DUs). The DU typically represents the smallest area for which a decision will be made. For additional information on DUs and Sampling Design please 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 most 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 the following:

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

4.2 Decision Unit (DU)

Determining the size, shape, location, and number of DUs is a critical component of the *IS* planning process. All representative parties should be in agreement prior to acceptance of the DU. The mean concentration of the DU should be the basis for deciding if the DU is properly sized; that is, it must be understood that the DU will pass or fail based on the acceptance of the results over the entire DU. The characteristics of the DU should not be assumed representative of any smaller portion (e.g., a subdivision) of the DU.

The primary types of DUs include those based on the known or suspected locations and dimensions of source areas, and those based on the size assumptions of risk assessment or exposure areas. The shape and size of DUs should consider the following:

- Areas that establish exposure areas.
- Contaminant transport and exposure pathways.
- Site contaminant distribution(s).
- Geologic and other physical characteristics (e.g., formation or soil type boundaries).

A DU source area is a distinct area containing elevated or potentially elevated concentrations of contaminant(s). These would include areas where:

- Stained soils, known contamination and/or where releases 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 assessments and risk-based decision making. Exposure areas include:

- Residential yards.
- Schools, playgrounds, and parks.
- Gardens and agricultural fields.
- Industrial lots.

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

It is important to recognize that when potential acute toxicity is of concern, *IS* is not recommended. The Part 201 soil criteria for cyanide are based on acute toxicity and, therefore, exposure estimates based on maximum concentrations may be more appropriate. When evaluating a DU for criteria based on an acute exposure scenario, random exposure across a property or exposure unit may not be assumed, and a point-by-point comparison of soil concentrations may be required to determine compliance.

4.3 Residential and Commercial Exposure Areas

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

Exposure areas for commercial or industrial properties are site-specific. Designation of exposure areas for these sites should be discussed during the DQO process. It may be advantageous to designate DUs to assist in evaluation of the property for future unrestricted land use (i.e., residential land use). Consideration of future unrestricted land use during the designation of DUs may help avoid unnecessary land use restrictions and/or the need for reinvestigation should future redevelopment plans call for a more sensitive land use.

4.4 Sampling Design

Once the DU is determined, the sampling design is developed. Data collection activities should be planned and developed through the DQO process and consistent with the CSM. DU sampling design, and in particular the number of increments appropriate for sampling, takes into account the objectives of the site investigation and type of information needed to make the decision. A minimum of 30 to 50 increments per DU is recommended to obtain a representative and reproducible DU mean concentration at a site that is characteristic of moderate heterogeneity. Depending on the degree of uncertainty that can be tolerated within the project-specific DQOs, a reduction in the number of increments may result in unacceptable uncertainty and/or decision error. An increased number of increments (e.g., 60 to 100) and/or a larger sample mass may be necessary where high heterogeneity is anticipated. Additional sampling design information is provided in the next section.

5.0 FIELD IMPLEMENTATION

5.1 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*.

Sample Planning

The first step in successfully implementing *IS* is to complete the DQO process as described in the previous section. 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 able to be conducted efficiently and without complication.

The total mass of an *IS* sample and the number of increments will be dependent (in part) on the heterogeneity of the DU and is typically between one and two kg (for the total sample) and a minimum of 30 to 50 increments. A sample mass of two kg or more and up to 100 increments may be more appropriate for highly heterogeneous soils.

5.2 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
Estimated Mass/Increment (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

*Mass identified applies to most fine to medium textured soils. Very moist to wet soil would require slightly more mass to account for the additional water content.

As an example, if the DQOs required a minimum 1 kg sample, 50 increments, and a 2 inch sampling depth, using the tables above, 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 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 0.75 inches or 16 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.

5.3 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.

5.3.1 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*

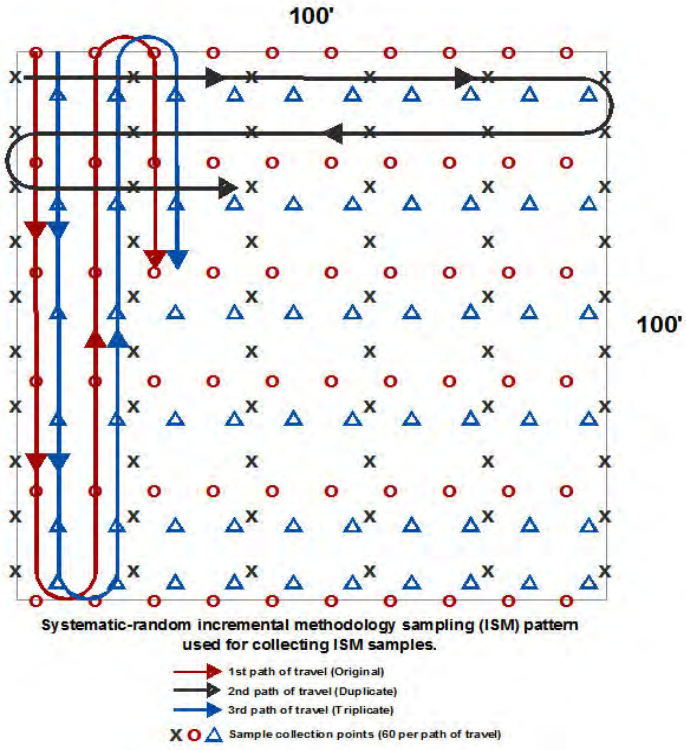

*Figure from ITRC 2012

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:

Grid spacing (feet) = square root of the area (feet^2)/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 is 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 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, 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 a sample 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 into the bag. If replicate samples are required, the same grid pattern is used but it is based on a new randomly selected starting point.

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 *IS* points. Sampling tools should be decontaminated between DUs or when collecting replicate samples.

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. Larger DUs may require markers at each *IS* point or the use of a global positioning system to locate each point. Grid patterns for regularly shaped DUs (square or rectangular) are relatively easy to lay out. 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.

5.3.2 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. While obtaining good spatial coverage and data quality for subsurface soils is more challenging than surficial soils, it can still be accomplished. The objectives are the same as they both can be used to estimate the representative concentration of contaminants for targeted depth intervals or to determine or confirm the lateral boundaries of a possible source area.

5.3.2.1 *Subsurface IS Sampling from Borings*

Subsurface IS sampling can be accomplished using a variety of boring tools that collect subsurface soil cores. These include hand tools (push probes, soils 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 will be conducted in a similar manner.

The entire core from any specified DU depth interval may be considered an increment and combined with similar additional increments for an *IS* sample. This can be accomplished with smaller cored depth intervals (6 to 12 inches thick), but can be problematic with thicker depth intervals or large diameter cores. The complete core interval can be subsampled to obtain an appropriate final sample mass when thicker intervals or larger diameters are encountered.

One technique for sampling non-volatile contaminants is to collect a “core wedge.” This is accomplished by vertically splitting the core in half, or in quarters, along the axis of the core. The collection of field replicates will normally require the same method of splitting of cores from separate areas (see Section 4.5 below).

Another *IS* soil subsurface sampling technique is to collect multiple plugs from core, or core sections, representative of the DU. Vertically defined DU’s may include pre-determined depth interval or lithology unit as determined by the DQO process. To complete the *IS* sample, multiple plugs are collected at regular systematic random intervals from the cores representing the DU.

5.3.2.2 *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 marked intervals down into the excavation at the proper grid spacing.

5.3.2.3 *Potential Subsurface Limitations*

As with surface *IS* samples, it is generally recommended that a minimum of 30 to 50 increments be collected for each DU. In some cases, as with direct push or drill rig borings, collecting the recommended minimum number of increments may not be feasible. Each increment requires its own separate boring and this increases cost. Although not considered IS, the development of an alternative sampling method (e.g., that uses less than the recommended minimum 30 - 50 increments) is an option if the project DQOs can be satisfied. In this situation, it is important to recognize that a reduced number of sample increments increase the uncertainty and decision error resulting in a less precise and more biased estimate of the mean contaminant concentration. Based on the degree of data variability that can be tolerated within the project-specific DQOs, a significant reduction in the number of increments may result in unacceptable uncertainty and/or decision error. In these circumstances, a careful review of DQOs, as well as, any other sampling options that may be available is needed. The DQOs will need to identify the sampling constraints and potential impacts on data quality, so that the most effective subsurface sampling strategy can be chosen.

5.3.3 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 will be determined through the DQO process, which will specify 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 to mass of soil (g). This amount of methanol will be 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. Typically, the individual increments 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 surficial 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.

A separate, unpreserved soil sample, representative of the increments collected for the *IS* sample, should be collected to determine percent moisture determination so the *IS* VOC results on a dry-weight basis. This sample should be collected in the same manner as the *IS* VOC sample. This is usually accomplished by collecting a second increment at each *IS* increment location and combining these increments in an unpreserved container (4 ounces or larger) for submittal to the laboratory.

5.3.4 Waste Pile *IS* Sampling

IS may be used to characterize waste piles and soil piles (e.g., disposal or treatment options). If heavy 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., 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. The DU may include one or more waste piles as determined by the DQO process. Collection of IS samples under these conditions may require a team of two or more persons to accommodate safety concerns.

If equipment is not available to move the piles, the process would likely include collecting surface and subsurface soil sample increments using appropriate coring tools. The suspected contaminant source, amount of mixing, prevention of loss for VOC and semi-volatile organic Compounds (SVOC) should be considered when determining the number and depth of increments collected from each DU.

5.4 Collection of Field Replicate *IS* Samples

Replicate *IS* samples (triplicates or more) should be taken during field sampling activities to quantify uncertainty and to ensure reliable estimates of the mean concentration within the DU. The number and frequency of replicates should be specified in the sampling plan and comply with project DQOs.

Separate replicate *IS* samples are collected to statistically evaluate sampling precision for each DU. These increments are collected in the same manner as the increments for the original *IS* sample for the DU. *IS* field replicates should consist of the same number of increments collected in the initial *IS* sample and be collected using the same sampling pattern from within the same DU. These replicate samples should be prepared and analyzed in the same manner as the initial 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 data approximates a normal distribution and that the average contaminant concentration reported accurately represents the DU(s). An RSD of less than 30 percent between replicates is generally precise enough to make decisions. This is typically achieved with the recommendations identified in this document.

The collection of triplicates from 10 percent of the DUs is normally recommended for *IS*. The number of replicates per DU and the frequency of replicate sampling must be clearly addressed in the DQO process. Site contaminant variability, the identification of separate populations, and the precision desired should be considered. For sites with multiple similar DUs, and where otherwise appropriate, replicates from one DU may be used to provide an estimate of variability that is extrapolated to other similar DUs.

5.5 Field Processing for Non-VOC Samples

IS sample processing techniques, such as sieving, grinding, and representative 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 some amount of sampling error, especially if conducted under field conditions. Because these errors can be reduced when these techniques are performed in a controlled laboratory setting as opposed to in the field, it is recommended that all *IS* sample processing be performed in a controlled laboratory setting. 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.

Moist samples may need to be air-dried 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 a 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 vegetative debris. Prior to sieving, the field-moist sample weight should be recorded. 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 will introduce bias into the mass of collected subsample. 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.

6.0 *IS* LABORATORY PROCESSING and ANALYSIS

6.1 Introduction

The procedures used in the laboratory to prepare and analyze samples are as important as how the samples are collected in the field. Laboratory subsampling can present the greatest potential for error of all the steps necessary to process and analyze environmental soil samples. 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.

6.2 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 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. 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.

6.3 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 increment collection locations 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.

6.4 Aliquot Mass

The ITRC and the EPA SW-846 Method 8330B (EPA a,b) guidance documents discuss the minimum aliquot mass required to reduce "Fundamental Error" or decision error of the laboratory analyses to 15 percent or less. The appropriate minimum mass is based on the maximum particle size in the soil sample. For samples with a maximum particle size of 2 mm, the minimum aliquot mass is 10 g (Note: This is a minimum analysis mass; there could be cases where 10 g is not enough to sufficiently reduce “Fundamental Error”). Laboratory analytical methods that use an aliquot mass of 10 g or more are preferred. Laboratory methods may need to be modified to meet project DQOs as determined by advance consultation with the selected laboratory. The ITRC has several suggestions for increasing the sample aliquot mass (e.g., combining multiple digestions).

6.5 Grinding

Grinding soil samples to achieve uniform small particle sizes is an option to reduce "Fundamental Error" and potentially reduce the aliquot mass required for certain (non-volatile) contaminants. Grinding samples also reduces the potential for segregation error. Suitable grinders are expensive and this service is something that laboratories may not offer. Thermal stability and volatility of the contaminant(s) should be considered before grinding. Also, grinding may not be appropriate for samples being analyzed for bioaccessibility/bioavailability.

6.6 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 8260 (EPA a,d). Typically, *IS* soil samples collected for volatile contaminant analysis include a minimum of thirty (30), 10 g increment soil plugs inserted into an equal volume of methanol (300 mL of methanol to 300 g of soil) in a 500 mL amber bottle. Replicate samples would be collected and analyzed separately.

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 *IS* samples, with a second increment collected at each *IS* increment location and placed in an unpreserved container of adequate size and submitted to the laboratory.

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

Contaminants of concern (COC) that include 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 COC in order to collect and process *IS* samples. Advance consultation with the selected laboratory is essential for the DQO planning process.

6.8 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 MDEQ investigations are handled by the Contract Laboratory procedure. The MDEQ contract pricing for *IS* related services are available from Test America. Samples may be sent to the MDEQ laboratory for shipment/transportation to a contracted laboratory.

7.0 RRD PROGRAM APPLICATIONS

7.1 Contaminated Sites and Stockpiles

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 assessing brownfields in the Brownfield Redevelopment

Program. *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 waste and for post-excavation verification.

7.2 Baseline Environmental Assessments (BEAs) and Due Care

IS may be the ideal sampling method for implementing and completing a BEA of a property. *IS* can be used for determining or demonstrating Due Care obligations. 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 these programs to the benefit of all interested parties.

A potential owner or operator may choose, or a lending institution may require due diligence in the form of a Phase I Environmental Site Assessment (ESA) (e.g., an All Appropriate Inquiry Phase I ESA) to determine whether environmental contamination is present on a property or portion of a property. A Phase II ESA of the property may be conducted as part of this evaluation, and typically includes soil sampling. The information obtained from the ESA may be used to determine whether the property is a Part 201 facility. If the property is a Part 201 facility, the prospective buyer or operator may submit a BEA to obtain liability protection from existing contamination. If an owner or operator of a Part 201 facility uses hazardous substances in their business, it is often in their best interest to determine the baseline contaminant concentrations from previous releases with accuracy suitable for distinguishing any new release from old releases. The increased accuracy and precision afforded by *IS* (in comparison to discrete sampling) may make *IS* the preferred sampling method to achieve these data quality requirements.

If the data set supporting the BEA is not sufficient to accurately determine if the new owner will be meeting their Due Care obligations, additional sampling may be helpful. An *IS* data set that provides a higher level of accuracy and reproducibility may be beneficial. IS sampling techniques can also be useful in the spatial determination of where exposure risks may exists and allowing for the implementation of any protective measures that may be necessary on portions of the property with greater certainty.

Appendix A.

REFERENCES

EPA DQO Reference 2000

EPA DQO Reference 2006

EPA "SW-846 - Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," http://www.epa.gov/epawaste/hazard/testmethods/sw846/index.htm, July 22, 2012, accessed on July 30, 2013.

EPA "SW-846 Method 8330B – "Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography," http://www.epa.gov/osw/hazard/testmethods/pdfs/8330b.pdf, October 2006, accessed on July 30, 2013.

EPA "SW-846 METHOD 5035A – "CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES", http://www.epa.gov/osw/hazard/testmethods/pdfs/5035a_r1.pdf

EPA "SW-846 METHOD 8260B – "VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)", http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/8260b.pdf

ITRC, "Incremental Sampling Methodology," February 2012, http://www.itrcweb.org/ism-1/

DEQ Data Quality Management reference: http://inside.michigan.gov/sites/deq/qmp/Quality%20Document/QMP%20August%206%202012%20%20PDF%20version.pdf