5. FIELD IMPLEMENTATION, SAMPLE COLLECTION, AND PROCESSING

5.1 Introduction

Section 2 discussed some of the common sources of sampling error. To obtain representative field samples, sampling error must be limited or managed (Ramsey and Hewitt 2005). In the absence of error, a sample result by definition would be accurate. However, it is impossible to completely eliminate error and produce an accurate result unless the soil in the entire DU is included in the analytical determination, which is obviously impractical. Thus, limiting sampling error is a critical function of any sampling design and implementation. This section addresses those field practices that limit or manage sampling error and provides guidance for obtaining representative samples. It should be noted that for many types of contaminants (e.g., metals, VOCs, semivolatile organic compounds [SVOCs]) specific studies have not been conducted to evaluate the applicability of all of the approaches discussed in this section and in Section 6.

To help ensure data quality, all field sampling and field processing activities should be performed and supervised by personnel trained in ISM. Figure 5-1 is a flowchart for ISM field implementation.
Figure 5-1. Field sampling implementation flowchart.
5.2 Sampling Tools

The selection of the appropriate sampling tool for an ISM sample depends on the cohesiveness and composition of the soil substrate. To minimize the increment extraction and delimitation errors described in Section 2.5.5, the sampling tool should obtain cylindrical or core-shaped increments of a constant depth from the presented surface. The diameter of the sampling tool should be a minimum of three times the diameter \( d \) of the largest particle present in a coarse matrix \( (d \geq 3 \text{ mm}) \), and \( 3d + 10 \text{ mm} \) for a fine material (Pitard 1993). Caution should be taken to select tools that equally retain all of the particles over the entire depth of interest. In general, sampling tools should have a diameter of at least 16 mm. For less cohesive soils, attempts should be made to retain the entire, complete core increment.

See Figures 5-2a and 5-2b for examples of sampling tools for nonvolatile ISM sample collection and Figure 5-12 for examples of sampling tools for ISM collection of VOCs. These are provided as examples only. Various other hand augers, core sampling tools, step probes, etc., are available from environmental or agricultural suppliers and are applicable to ISM if the specifications meet project DQOs. Again, the sampling tool(s) selected should minimize increment extraction and delimitation errors.

The sampling tools required to collect core-shaped soil increments of required length in the field are necessarily site specific. Alternate sampling tools that meet the basic ISM principles and project-specific objectives may be available currently or in the future. A variety of tools to address different soil types or site conditions should be taken into the field for any given project.

Cylindrical increments of a controlled depth can be obtained from cohesive soils with a variety of commercially available manually and machine-operated coring tools. For depths of 10 cm (3.9 inches) or less, individual increments often can be rapidly collected and dispensed into a sample container using hand-operated tools. For noncohesive soils and sediments, short- and long-nose scoops (trowels) can be used; however, care should be taken to obtain a “core-shaped” increment over the entire depth of interest. For depths greater than 10 cm, or for hardened and unconsolidated rocky geological materials, coring devices can be advanced with a hammer, slide bar, or some other means of mechanical assistance. Depending on site familiarity, one or several sampling tools should be readily accessible during all sampling activities.

Sampling devices can be used within a DU without decontamination but should be decontaminated or disposed of between DUs. If sampling tools will be used for two or more DUs, they should be cleaned of soil particles, decontaminated with the appropriate solutions or solvents, and dried between DUs. Typically, rinse (decontamination) blanks can be used to evaluate the potential effects of cross contamination, if needed.
Figure 5-2a. Examples of coring devices for nonvolatile soil increment collection. Top to bottom: Multi-Incremental Sampling Tool (MIST™), EVC Incremental Sampler, JMC Backsaver Handle, and Soil Tube.
5.3 Field Collection

5.3.1 Surface ISM Samples

ISM samples are composed of increments collected from specific points throughout the DU. The positioning of the collection points can be set using one of three approaches, as described in Section 4.3.4.2: simple random sampling (SRS), random sampling within a grid, and systematic random sampling. SRS involves determining random locations across the entire DU. Note that “random” in this context does not mean wherever the sampling team feels like taking a sample and that a formal approach to determining the random increment locations must be used. With random sampling within a grid, the DU is overlain with a sampling grid and soil increments are collected from random locations determined in each grid cell (see Figure 4-8). Systematic random sampling is similar except that only the initial grid cell sampling location is randomly determined and the same relative location is sampled in each of the other grid cells (see Figure 4-7).

As predicted by statistical sampling theory and demonstrated by the ISM simulations discussed in Section 4.3.4.2 and Appendix A.1, SRS yields the most representative (least biased) estimate of the mean. However, it is also the least practical to implement since field staff have to navigate to predetermined locations nonuniformly positioned within the DU. SRS also may result in a sampling pattern that leaves large portions of a DU unsampled, which may not be acceptable to regulators, risk managers, members of the public, or other stakeholders. In practice, systematic random sampling is most often chosen for ease of implementation and to avoid the appearance of over- or underrepresentation of subareas within a DU, as may occur with SRS. Refer to Superfund Representative Sampling Guidance, Vol. 1 (USEPA 1995b) for additional information.

Incremental soil samples are prepared by collecting multiple increments of soil (typically 30 or more) from a specified DU and physically combining these increments into a single sample, referred to as the “incremental sample.” When the individual increment mass is adequate, this number of increments (n)
generally results in a soil sample with a contaminant concentration representative of the estimate of the mean contaminant level within a DU (i.e., a representative sample). That is, even when the distribution of individual data points (i.e., discrete sample results) is nonnormal, the distribution of sets of means from the population will approach a normal or Gaussian shape as the number of increments \((n)\) increases (Jenkins et al. 2005). See Section 4 of this document on the statistical basis of ISM for a more detailed discussion of increment number(s), adequate increment mass, and representativeness.

As sampling theory indicates, the number of increments collected depends on the amount of distributional heterogeneity present within the DU for the constituent of interest. A variety of factors may influence the amount of distributional heterogeneity within a DU. These include, but are not limited to, the following:

- contaminant type and physical characteristics
- soil type and physical characteristics
- contaminant release mechanism (e.g., spill, area-wide application, munitions range)
- others

As the DU gets significantly larger, the amount of distributional heterogeneity may increase. In these cases, depending on site specifics, CSM, and DQOs, it may be necessary to increase the number of increments per DU to 50 or more. Collection of a greater number of increments in each DU typically reduces the GSE (i.e., minimizes the variation among replicate samples). Alternatively, splitting larger DUs into two or more smaller DUs should be considered. It is not normally necessary to increase the number of increments unless there is reason to believe the DU has more distributional heterogeneity than can be controlled with 30–50 increments. See Section 4.3.4.1 of this document for the statistical information and evaluation of the number of increments for ISM sampling.

In general, a minimum of 30–50 increments is sufficient for most DUs. However, in published reports for solid/particulate-type chemicals of concern (COCs) (e.g., energetics/explosives, particulate metals, etc.) 50–100 increments per DU have been collected. USEPA SW-846 Method 8330B recommends collecting 30 or more evenly spaced increments to build a sample with a total mass of >1 kg. It is anticipated that as ISM matures, additional information on the optimal number of increments for other types of contaminants may become more readily available. The number of increments to be collected from each DU of a site investigation should be evaluated during systematic planning as part of the DQO process and documented in the sampling and analysis plan (SAP).

In general, individual soil increments typically weigh 20–60 g. Final ISM field samples typically weigh 500–2500 g. To minimize FE to an acceptable level, it may be necessary to calculate the target bulk ISM sample mass for collection prior to field implementation and ISM collection (Pitard 1993, Ingamells and Pitard 1986, see also Section 2 and Hyperlinks 14 and 18) It may be necessary to collect bulk ISM samples >2500 g to reduce FE to an acceptable level. Additionally, note that sieving of soil samples to the <2 mm particle size reduces the amount of soil mass available for preparation and

| Generally, a minimum of 30 increments should be collected for each DU, with each increment weighing 20–60 g. |
analysis, so this fact needs to be taken into consideration during systematic planning if minimizing FE is a DQO. Additionally, sieving is not applicable for the collection of VOC samples (see Section 5.4.2). Based on the required final mass of the ISM sample, as dictated by FE considerations and the number of increments determined by distributional heterogeneity, the minimum mass of the individual increments can be calculated. The mass of any single increment depends on the depth of interest, soil density, moisture content, and the diameter or size of the sample collection tool. Typically, the mass of the final ISM sample is sufficient for the planned analyses, any additional QC requirements, or repeat analyses due to unanticipated field, laboratory, and/or QC failures. The number of increments to be collected per DU, the sampling depth, and the targeted mass of each sample should all be specified in the sampling plan as described in the following formula for estimating sampling equipment requirements based on a predetermined ISM mass and number of increments:

\[
M_s = \rho \cdot n \cdot D_s \cdot \pi \cdot (\theta/2)^2
\]

where

- \(M_s\) = targeted mass of sample (g)
- \(D_s\) = increment length (cm)
- \(n\) = number of increments
- \(\rho\) = soil or sediment density (g/cm³)
- \(\theta\) = diameter of sample core (cm)

These parameters, along with the density of the soil or sediment matrix, assist in the selection of the sampling tool to collect the appropriate individual increment mass for the total ISM sample (Walsh 2009).

Figure 5-3 and Table 5-1 (Walsh 2009) are provided as examples for estimating increment mass that can be collected for a given sampling depth and soil density, once the DU size, number of increments and total ISM sample mass have been established. Generally, a minimum of 30 increments should be collected for each DU, with each increment weighing 20–60 g. Individual increment mass should be similar provided the soil density and DU thickness are fairly uniform. Typically, however, individual increments are not weighed in the field during collection. Similar mass per increment is assumed with similar volume collected. Due to practical limitations, increments of similar volume rather than of similar mass are collected, provided that the thickness of the DU is fairly uniform. For DUs of nonuniform thickness, the available thickness at each increment location is collected to ensure spatial coverage and the increment is not required to have similar volume or mass.
Figure 5-3. Estimated sample mass based on number of increments for set increment and substrate density.

Dry Bulk Density Used: 0.75 (Loess) and 1.81 (Gravely Sand)

Formula:
\[ M_s = \rho \cdot n \cdot D_s \cdot \pi \cdot \left(\frac{\theta}{2}\right)^2 \]

- \( M_s \) = Targeted Mass of Sample (g)
- \( D_s \) = Increment Length (cm)
- \( n \) = Number of Increments
- \( \rho \) = Soil or Sediment Density (g/cc)
- \( \theta \) = Diameter of Sample Corer (cm)

Sample Masses for Commonly Used Increments:
- 2.5 cm Increment Length
  - 2.0 cm Core Diameter
  - 0.75 g/cc - 1.81 g/cc
  - 30 Increments = 177 - 424 g
  - 60 Increments = 353 - 848 g
  - 100 Increments = 589 - 1414 g

The online version of this document contains a working calculator for incremental soil mass: [http://www.itrcweb.org/ISM-1/5_3_Field_Collection.html](http://www.itrcweb.org/ISM-1/5_3_Field_Collection.html)
Table 5-1. Estimated sample mass for set increment length and substrate density

<table>
<thead>
<tr>
<th>Corer diameter (cm)</th>
<th>Soil density 1.6 g/cm³, increment length 2.5 cm</th>
<th>Soil density 1.8 g/cm³, increment length 2.5 cm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>500 g</td>
<td>750 g</td>
</tr>
<tr>
<td></td>
<td>1000 g</td>
<td>1500 g</td>
</tr>
<tr>
<td></td>
<td>2000 g</td>
<td></td>
</tr>
<tr>
<td>2.0</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>3.0</td>
<td>18</td>
<td>27</td>
</tr>
<tr>
<td>4.0</td>
<td>10</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.0</td>
<td>35</td>
</tr>
<tr>
<td>3.0</td>
<td>16</td>
<td>24</td>
</tr>
<tr>
<td>4.0</td>
<td>9</td>
<td>13</td>
</tr>
</tbody>
</table>

Substrate density may vary from 0.75 g/cm³ (for Loess) to 1.81 (for Gravely Sand) with substrate densities typically ranging 1.6–1.8 g/cm³ (Walton 1988, Domenico and Schwartz 1990).

If replicate ISM results indicate data variability is too high (i.e., interferes with decision making for the DU), additional data evaluation, sample analysis and/or resampling may be required to achieve project-specific DQOs. Note, however, that high variability between ISM replicates may also be a result of laboratory processing and subsampling procedures, which can be evaluated by examining the results of laboratory replicates (if analyzed). High data variability determined to be a result of DU heterogeneity and/or field sampling error may require revision(s) to the ISM design and implementation, including DU modification, additional increments, and/or increased increment mass (see Section 5.3.5).

Soil density across the DU should be reasonably uniform (e.g., the same general soil classification can be expected throughout the DU). When the surface of the DU contains both vegetated and nonvegetated areas, it is very likely that less soil (less increment mass) will be obtained from the vegetated regions within the DU. If a site has obvious areas with different soil lithologies and/or densities (e.g., areas of sand with areas of fat clay, areas of peat, etc.), those different soil type areas should be factored into DU determinations (i.e., location, shape, size of DUs). Assumed differences in contaminant concentrations in the different soil types should also be considered. In these cases, it may be necessary to redefine the DU to account for the possible heterogeneity of contaminant concentration.

For surface/exposed soil, common sampling depths are 2.5, 5, 10, or 15 cm; however, depths can be greater depending on the DQOs and CSM, including expected vertical distribution of the chemical of potential concern (COPCs) (due to infiltration, buried utilities/facilities, stockpiles, etc.), the exposure scenario, and/or regulatory requirements. Additional depths and/or DUs may be required for vertical delineation. Contaminant dilution should also be considered when determining increment depth. For surface-deposited energetics at active U.S. Department of Defense (DOD) training ranges, soil profile samples have shown 1–2 orders of magnitude decreasing concentrations within the top 10 cm (USEPA 2006c). For these types of sites, the desired sampling depth is approximately 2 cm, based on research conducted at Cold Regions Research and Engineering Laboratory (CRREL); greater increment depths result in dilution of the contaminant concentration. In general, the location, lateral extent, and depth of the DU should be selected to represent an area of known or expected similarity. For greater depths, use...
of a smaller-diameter sampling tool may be desirable but often is impractical due to presence of pebbles, rocks, and vegetation. In general, however, the smallest diameter sampling tool applicable to particle size requirements is recommended to minimize delimitation and extraction errors and to attain the necessary soil mass (see Section 5.2). Alternative technologies for site-specific conditions should be considered, as appropriate.

A square, rectangular, circular, or other naturally or structurally defined DU (e.g., 5 m perimeter around the exterior of a building) is first subdivided or gridded-off into uniform cells or subareas based on the desired number of increments to be obtained. That is, the number of cells is equivalent to the number of increments. Using the systematic random design, a random position is established for a given cell, and then the same position is repeated in all of the remaining cells in the DU. For the random sampling within grids design, a random position is designated and sampled in each cell. A random starting point or random position for each cell can be obtained with dice or a random number generator. The process is repeated for replicate samples; i.e., a new random position is established for the single collection point to be repeated in all of the cells, or for each cell, depending on the sampling design. A Global Positioning System (GPS) device should be used to delineate the DU. It may or may not be necessary to determine the exact location of each increment depending on the DQOs specified during the systematic planning process.

Depending on the size of the DU and terrain features, placement of markers (e.g., pin flags and posts) at the corners and or edges can assist with a visual delineation of the cells or subareas where increments are to be collected. That is, the markers can define lanes, grids, or collection points. When DUs are square or rectangular, the conversions for the spacing (steps) between increment collection points (cells) are fairly straightforward to calculate. For example, a square-shaped DU could be divided into five rows, with six increments collected from each row in a systematic random fashion, with an initial random starting point. For more rectangular-shaped DUs, fewer rows might be used with more increments per row collected (Figure 5-4). Row lengths and increments per row may be modified as needed for odd-shaped DUs. However, with other shapes, it is recommended that the perimeter be marked and flags be prepositioned across the DU in one or more perpendicular lines. Then a trial run with no sample collection is performed to quickly establish the distance between increment collection points to achieve the desired number of increments, while using the flags as guides that were positioned within or around the DU.

Although ISM sample collection may be performed by a single individual, a two-person team is often the most efficient method: ideally one person
collects the increments, and the other holds the sample container (e.g., clean polyethylene bag) and keeps track of the number of increments. However, site conditions may dictate that three or more individuals are required for the collection of a single ISM sample. The User’s Manual for the CRREL Multi-Increment Sampling Tool (Walsh 2009) lists common sampling supplies and vendors that would be appropriate for SVOCs and metals. Sampling tools are set for the appropriate depth. Flags may be used to mark DU boundaries and to aid in visualizing the travel paths and/or to mark the actual increment locations. The ISM sampler starts in one corner or end of the DU and collects an increment at the predetermined positions. For the systematic random sampling design, the location of the first increment is determined randomly, and subsequent increments are collected in the same relative location within each grid, resulting in a serpentine collection pattern ending at the opposite corner or end of the DU from where sampling was started (see Figure 5-5). Note that, for simplicity, Figure 5-5A depicts collection of duplicate ISM samples rather than the recommended triplicates. Additional guidance on ISM can be found in the following documents:

- Method 8330B, Appendix A (USEPA 2006c)
- Protocols for Collection of Surface Soil Samples at Military Training and Testing Ranges for the Characterization of Munitions Constituents (Hewitt et al. 2007)
- User’s Manual for the CRREL Multi-Increment Sampling Tool (Walsh 2009)
- Technical Guidance Manual (HDOH 2008b)
5.3.2 Subsurface ISM Samples

As discussed in Sections 2 and 3, DUs are by definition 3-D in nature and are intended to focus the investigation on a specified volume or mass of soil. Obtaining good spatial coverage and data quality for subsurface soils is more challenging but is still necessary. The objectives for surface vs. subsurface investigations may be similar in nature, for example, to estimate the representative concentration of targeted contaminants for targeted depth intervals (e.g., within the defined vertical limits) or to determine or confirm the lateral boundaries of the source area. For remedial purposes, the estimation of contaminant mass within the DU is also sometimes critical (e.g., mass of tetrachloroethene for design of soil vapor extraction system or mass of dioxins for design of in situ thermal desorption system). The practical application of ISM sampling must be considered during project planning, especially when considering implementing it for nature and
extent investigations of subsurface contamination. Often, alternative sampling techniques (e.g.,
discrete sampling, field screening, or field analytical methods) may be more applicable and/or
cost-efficient.

Soil samples collected as part of a subsurface investigation are intended to be representative of a specific depth interval. As
discussed in Section 3, this trait can be described as the resolution of the data collected. Discrete soil samples from borings or
excavations have traditionally been used to characterize subsurface soils. In most cases, however, discrete samples may provide less spatial coverage of the targeted
depth intervals and also increase laboratory analytical costs. As discussed below, alternative sample collection approaches to improve sample data quality and reduce laboratory costs include
options for ISM core sampling across targeted depth intervals.

5.3.2.1 Subsurface ISM samples using core sampling

If a coring device is used, samples should be collected from targeted depth intervals in a manner that ensures the best coverage of the interval. For example, the selected subsurface DU investigation strategy may require the collection of soil samples from specified 15 cm (6-inch) intervals over a 1 m depth (see Figure 5-6). In other cases, the mean concentration of a targeted contaminant over the entire 1 m DU (or larger) interval may be desired for risk assessment or remedial purposes (see Figures 3-7, 3-8, and 3-9).

![Figure 5-6. Schematic of a procedure to collect an ISM profile sample where two depths have been selected.](image)

Ideally, to be representative, the entire core depth interval should be considered as an increment, collected, combined with additional increments for an ISM sample and submitted to the laboratory. Collection of the complete core interval as an increment is the recommended subsurface ISM procedure. This method can result in large ISM samples (approximately 5–10 kg), making logistics, such as field storage and shipping, problematic. Additionally, the selected laboratory must have facilities available to store, dry (if required), and process these large amounts of soil mass. Consequently, depending on the core diameter and interval depth, inclusion of the entire core increment across a targeted depth interval in an ISM sample may be impractical. In such cases, individual cores may be subsampled to reduce the final mass of the ISM sample. Two options are described below.
Another option for collecting a representative subsample from a subsurface core increment for nonvolatile contaminants is to collect a “core wedge” sample. The simplest approach is to split the core in half vertically along the axis, reducing the increment mass by half. Alternatively, a single wedge of soil is taken from the entire length of the targeted depth interval. Removing a wedge of soil across the length of a larger core to encompass the entire depth interval rather than collecting the entire core depth interval as a whole, constitutes the mass of an individual increment of an ISM sample (see Figure 5-7). Individual wedges from 30 or more separate DU cores are then combined to form the complete subsurface ISM sample. This option results in a more biased and less precise estimate of the DU mean as compared with collecting the entire core. However, since the mass of each increment (and thus the ISM sample mass) is reduced, some of the practical constraints associated with handling full core increments are addressed.

**Figure 5-7. Example of removing a wedge from the entire length of a soil core.**

Replicate(s) can be collected from the same core, combined with other wedge increments, and submitted as separate ISM sample(s) to assess the precision of this subsampling strategy. However, core wedge replicates are not the same as ISM field replicates because ISM field replicates require completely separate incremental locations. Thus, core wedges should not be used as a measure of DU or overall sampling and analysis variability. Core wedge replicates evaluate only the variability in the subsampling process as opposed to collecting the entire core interval as the increment. The variability of wedge subsamples from alternative areas of the core is evaluated, e.g., replicate wedge collected 180° opposite the initial wedge subsample. ISM field replicates provide information on spatial variability and the variance in the estimate of the mean without specifically separating out the contribution of field and/or laboratory sample
processing/subsampling from other sources of variance. ISM field replicates are discussed in Section 5.3.5. Core wedge replicates may also be collected when COPCs require separate laboratory processing procedures (see Section 6.2.2.2).

This approach is not appropriate when VOCs are of concern since they can be quickly lost from an exposed surface (Hewitt, Jenkins, and Grant 1995). For VOCs, multiple “plugs” representative of the desired core depth are collected and immediately preserved in methanol (see Section 5.4.2).

The least preferred option for subsampling individual subsurface cores for nonvolatile contaminants is to collect a “core slice” from the targeted DU layer (see Figure 5-8). In this approach, a randomly selected perpendicular “slice” from within the larger targeted depth interval is collected as the ISM increment. For example, if the targeted depth interval was 2 feet in length (e.g., 8–10 feet bgs), a 4-inch perpendicular slice is randomly selected from within the targeted depth interval of each individual core and collected as the ISM increment. Individual, randomly selected core slices from 30 or more separate DU cores are then combined to form the complete subsurface ISM sample. This option introduces more bias than whole-core increment or core-wedge approaches. However, by reducing the increment mass, some of the logistical issues associated with handling the full core or the wedge increments are addressed. This is the least recommended approach for subsurface ISM core sampling since it is least likely to accurately represent the complete vertical length of the targeted DU layer.

**Figure 5-8. Examples of “core slice” sample.** Source: Illinois EPA LUST FAQ and BIOTREE websites.

### 5.3.2.2 Additional subsurface ISM considerations

As with surface ISM samples, it is recommended that a minimum of 30 increments be collected for each DU. In some cases, collecting the recommended minimum of 30 soil increments per subsurface DU may not be feasible or practical. Reducing the number of increments collected per sample may be the only viable option. In this situation, it is important to recognize that collection of a reduced number of sample increments generally increases the GSE and results in...
a less precise and more biased estimate of the mean contaminant concentration. Depending 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 a decision error. A sample containing fewer increments than required to estimate the DU mean concentration within the project-specific uncertainty level may not be considered a defensible ISM sample. Consequently, in these circumstances careful review of DQOs as well as any other sampling options that may be available is warranted. The subsurface sampling strategy chosen, the sampling constraints, and potential impacts on data quality should also be identified in the DQOs in the SAP and or Quality Assurance Project Plan (QAPP).

Increments from the same depth interval throughout the DU can be combined and used to create a single ISM sample for that depth interval. This is a useful approach for the characterization of vertically stacked DUs (see Figures 3-7, 3-8, and 3-9). Data for each ISM sample can be used to create a 3-D map of contaminant levels in the DU. This procedure can be especially useful where a large number of side-by-side DUs are designated for the investigation of large areas (e.g., redevelopment of a former golf course contaminated with pesticides).

5.3.3 Stockpile ISM Samples

Special considerations for selecting DUs during the systematic planning process for sampling soil stockpiles include the following:

- the source of the soil in the stockpile
- how the stockpile was created (over time, if applicable)
- how best to access the pile for sampling, (e.g., large or unstable)
- contaminants targeted for lab analyses

One of the best options is to coordinate sampling with the formation of any stockpiles on the site. When the stockpile is being formed, there is generally good access to sampling each portion of the pile over time, and ensuring access to the entire stockpile DU is provided for good sample representativeness. If an existing stockpile is relatively small, good options may include moving the pile and collecting the increments while it is being moved (e.g., from the front-end loader buckets, at appropriate intervals), or flattening or spreading out the stockpile sufficiently so that it is safely accessible to sample with a hand coring or other device. If the stockpile is very large or unstable, all available sampling tools or methods that safely provide access should be considered, with the goal of coming as close as possible to collecting a minimum of 30 systematic random or random within grids increments throughout the stockpile (both vertical and horizontal locations). Replicates are important to evaluate the precision of stockpile sampling and should be collected similarly to the original sample except in separate random locations. Large stockpiles could be divided or segregated into separate DUs (see Figure 3-10), especially if a specific portion or volume of the stockpile will be used in a manner that will become the primary exposure unit of concern in the future (e.g., certain portions or volumes of the stockpile will be hauled to residential lots as surface fill for backyards). A resource for additional information on ISM approaches for soil stockpile sampling is the Hawaii Department of Health.
(HDOH) Technical Guidance Manual (HDOH 2008b). Refer to Section 3.6.4.2 of this document for additional information on ISM sampling of stockpiles.

5.3.4 ISM Confirmation Sampling

Confirmation sampling may be performed during post-removal activities to verify that residual concentrations of target COCs are below the predetermined cleanup goals for the site. Confirmation sampling is often a requirement to achieve final clean closure certification. Confirmation samples are typically collected from the sidewalls and floors of an excavation to confirm that concentrations remaining after excavation are below specified concentration limits. Results from individual grab samples, an average or a 95% UCL from discrete samples, are often compared with the cleanup criteria for the site for this purpose.

An incremental sample result is specifically designed to estimate the mean concentration in a volume of soil designated as a DU. If excavation is performed for a site based on results from ISM sampling, it is usually because one or more DUs “failed” (i.e., had concentrations above the specified cleanup goals). Once the soil in a failed DU has been removed, the motivation for sampling the sidewalls and floor of the excavated DU is presumably to determine whether surrounding potential DUs also require remediation. If adjacent areas have already been designated as DUs, evaluated, and found to have soil concentrations within acceptable limits, confirmatory sampling in the conventional sense may not be necessary. If adjacent areas have not been adequately characterized, collecting ISM samples around the excavation can inform the need for or against further removal. In this situation, the expanded investigation requires new planning, including the designation of additional DUs and the determination of appropriate cleanup goals. One approach is to designate a volume of soil surrounding the excavated area as a new DU and sample from the walls and floor accordingly. This process is somewhat analogous to conventional confirmatory sampling. However, it is important to consider how the areas of the walls and floor relate to the volume of soil in the new DU and take increments in a manner that ensures a sample is representative of the DU. It is also important to recognize that the cleanup goal for a DU consisting of soil immediately surrounding an excavated area might be different from the original cleanup goals used for site evaluation because the objectives (e.g., addressing concern for potential for direct contact, leaching to groundwater, etc.) may be different, given the size and location of the new DU. As always, clear articulation of objectives and proper planning are essential.

In summary, the use of ISM samples to confirm excavation of a source area DU can be highly advantageous over a traditional, small number of discrete samples. The excavation floor and sidewalls should be treated as individual DUs (see Figure 3-11), with the investigation objective of determining whether the estimated mean concentration of COPCs for these areas exceeds targeted screening levels. Again, these issues should be evaluated and determined as part of the planning and DQO process. Collecting ISM samples within these areas rather than single discrete samples ensures good DU spatial coverage and a more representative estimate of mean COPC concentrations. There may be regulatory limitations to this approach, however. For example, if regulations require cleanup of releases to a not-to-exceed regulatory level (e.g., the maximum concentration determined by discrete
samples), then an ISM mean concentration may not be applicable and/or accepted by the regulating authority.

5.3.5 Collection of Field Replicate ISM Samples

In the field, replicate incremental samples (three or more) should be taken to ensure reliable estimates of the mean concentration within the DU. The number of replicates and frequency of taking replicate incremental samples should be specified in the SAP and comply with project DQOs.

To statistically evaluate sampling precision for each DU, additional completely separate replicate ISM samples are collected. The increments are collected in simple random, systematic random, or random within grid locations within the DU that are different from those used for the initial ISM sample. ISM field replicates are made of the same number of increments collected in the initial ISM sample and collected using the same sampling pattern from within the same DU. The replicate samples are prepared and analyzed in the same manner as the initial sample. Three replicate samples (i.e., the initial ISM sample plus two additional samples) should be considered the minimum. In some cases, more replicates may be necessary to reduce data variability and/or to calculate a 95% UCL of the mean that is closer to the actual mean of the DU. Section 4.3.4.1 discusses the statistical basis and evaluation of replicate ISM samples.

When sampling in a systematic random sampling pattern, the increments for an ISM replicate sample are generally collected along the same approximate directional lines established through the DU for the initial ISM sample. Increment locations for ISM replicate samples differ from each other by the selection of different random starting locations on the first line/row of the DU and continuing to sample at this different random interval throughout the DU for each replicate (see Figure 5-5). Thus, the increments for ISM replicates should not be collected from the same locations or colocated with those used for the initial ISM sample. When using the random sampling within grid pattern, replicates are constructed from increments taken from different, randomly selected locations within each gridded area. With simple random sampling, three sets of random locations across the DU are selected and increments collected for each set are used to create the replicates. Replicate ISM samples should be submitted to the laboratory as “blind” samples, meaning the laboratory does not know they are replicate samples of the initial ISM samples.

If only one DU is being investigated, a minimum of three replicate samples should be collected to provide a measure of variability. For sites with multiple similar DUs, “batch” type replicates may be a consideration; for example, three replicates in one DU could be used to provide an estimate of variability that is extrapolated to a number of similar DUs (similar to how labs use batch replicates for determining lab analysis precision). Each site and/or project is unique in terms of numbers of DUs and how similar these DUs are, so decisions on numbers of replicates are unique to each site and should be addressed clearly in the SAP. For the batch type of replicates to apply, each DU in the “batch” should have a similar CSM, including the same soil type, site use/history, contaminant deposition, etc. If considered, this batch approach must be discussed, clearly documented, and agreed to by all parties involved during the systematic
planning process (see Section 4.4.2). Section 7 discusses how replicate ISM sample data are used to assess sampling error and make decisions.

5.4 Field Handling of ISM Samples

5.4.1 ISM Samples for Non-VOCs

ISM sample processing techniques, such as milling and representative subsampling, are designed to ensure that the (typically small) mass of sample analyzed by the laboratory is representative of the DU or SU from which it was collected. These techniques reduce data variability as compared with conventional sample handling and processing approaches. However, these techniques introduce some amount of sampling error. It is recommended that all ISM sample processing be performed in a controlled laboratory setting to minimize these sampling errors. However, depending on site logistics, the type of soil, the total number and/or mass of ISM samples, etc., sample processing can be initiated in the field for some contaminants (e.g., SVOCs, pesticides, PCBs, and metals) with appropriate cautions as noted below.

Moist samples may need to be air-dried to facilitate sieving in an appropriate dust-free location where temperatures and ultraviolet (UV) light are not expected to cause degradation of COPCs. Samples with little vegetation and composed mostly of sands and silts that naturally have a very low moisture content and soils that have been air-dried can be sieved (typically using a #10 sieve, <2 mm particle size) in the field to remove pebbles and vegetative debris. Prior to air-drying or sieving or both, the field-moist sample weight should be recorded if specified in the SAP. The <2 mm soil particles are generally considered “soil,” while larger particles are considered gravel, rocks, or other materials (e.g., sticks and roots). Additionally, field sieving is an option that allows the user to calculate the mass of an bulk ISM sample needed to meet DQO requirements (including FE, see Hyperlinks 14 and 18), based on the soil particle size. Although sieving to the <2 mm particle size is typical, there may be contaminant investigations or analyses where alternative particle sizes may be of interest. In these cases, the rationale for sieving to other specific particle sizes and associated changes to lab processing/analysis should be clearly discussed in the SAP. Unless field subsampling will be performed (see paragraphs below), the entire sieved ISM sample fraction should be submitted to the laboratory for appropriate processing and subsampling.

When dealing with contaminants that have been deposited as solid particulates (e.g., energetics, metals at firing ranges, etc.), field subsampling is not recommended. Studies on energetics have shown that representative subsampling prior to grinding is problematic and likely not possible (Hewitt et al. 2009). In cases where sieving is conducted in the field to obtain a targeted particle size (particle size selection), the entire sieved ISM sample should be ground prior to subsampling (if particle size reduction is part of the SAP). Similar studies evaluating field subsampling for contaminants deposited as liquids (e.g., fuels, solvents, etc.) are not available at this time.

The SAP may specify particle size selection (sieving) and subsampling in the field for the analysis of SVOCs and specific metals. This procedure constitutes, or is similar to, the normal
laboratory subsampling step. It should be reiterated that it is recommended that all ISM sample processing be performed in a controlled laboratory setting.

If *field subsampling* is to be performed, the entire ISM sample should be air-dried (only if necessary) and sieved to the predetermined particle size (typically using a #10 sieve, <2 mm particle size). The sieved ISM sample should be spread out in a thin layer on a clean surface, e.g., a large, disposable, aluminum baking pan, allowing the entire sample to be accessed. A subsample is then obtained by removing 30 or more equal increments from systematic random locations (see Figure 5-5). The increments collected to form the subsample should equally represent the top and bottom of the processed material. This is achieved by using a rectangular, flat-bottom sampling tool with sides and a minimum 16 mm width (see Figure 5-9), as opposed to one that is curved or spoon-shaped (see Figure 5-10). Spoon-shaped sampling tools bias the mass of soil collected.

![Figure 5-9. Examples of rectangular and flat-bottom sampling tools.](image)

![Figure 5-10. Example of subsample being collected in the field.](image)
The mass of sample required for the analytical test or tests is used to determine the mass of each of the 30 or more increments. For example, if a mass of 30 g is required for the analytical extraction and analysis, 30 separate ~1 g increments are collected from systematic random locations. Depending on the project DQOs, replicates of the field processed soil should be collected and submitted for analysis to evaluate the precision of the ISM field processing procedure. The entire submitted subsample mass must be prepared for analysis due to possible particle size discrimination during sample transit (e.g., fines settling to the bottom of the sample container). If the entire contents of the submitted container are not to be analyzed, the laboratory must use proper techniques to ensure a representative particle size subsample is used for analysis. Laboratory replicates should be analyzed to evaluate the precision of the laboratory subsampling procedure. Refer to Section 6.2.2.7 describing analytical subsampling techniques and specifically the description of 2-D Japanese slabcake sampling.

Simply dividing an ISM sample (sieved or not) into separate volumes and placing each volume into separate sample containers for analysis is not an acceptable method of mass reduction. Likewise, manually mixing samples (i.e., “homogenizing”) in the field or lab may just serve to further segregate different particle sizes, because particles may settle in layers by weight or size during mixing. The process of spreading the entire sample out to a thin layer and collecting many increments in a systematic random fashion with a tool that can scoop to the bottom of the sample is the best way to collect a representative subsample of all the different sizes and types of soil particles present in the ISM sample.

Finally, if ISM sample processing and subsampling is performed in the field, it is recommended that at a minimum three replicate subsamples be collected and submitted to the laboratory for analysis. The subsampling (as described above) process is repeated on one ISM sample to form replicates. The replicate results are used to evaluate the precision of the field processing and subsampling. Note that the subsampling replicates should be collected in addition to the ISM field replicates described in Section 5.3.5.

Limitations to the field processing of ISM samples include the following:

- not recommended for contaminants deposited as solid particulates (e.g., energetics, metals at firing ranges, etc.)
- lack of commercially available, correct subsampling tools (e.g., 16 mm wide, flat-bottom scoop with sides)
- requires a controlled environment to air-dry, sieve, and subsample, if necessary, to minimize the potential loss or introduction of COCs during processing
- additional subsampling replicates need to be collected and analyzed to evaluate precision
- more knowledgeable/trained field personnel required

5.4.2 Volatile Organic Compound ISM Samples

ISM samples can also be collected for VOCs contaminant analyses from cores, excavation-pit bottoms and walls, stockpiles, underneath paved areas, etc. USEPA SW-846 Method 5035A
Section 8.2.2 (USEPA 2002b) describes the collection of discrete soil samples preserved in the field. The ISM VOC approach is similar to this method and to that described for sampling ISM nonvolatiles in the subsurface, except that numerous soil increments are placed directly into an adjusted volume of extraction solvent in the field (e.g., methanol, shown in Figure 5-11). Individual increment mass should be similar provided the soil density is fairly uniform. Typically, individual increments are not weighed in the field during collection. Similar mass per increment is assumed with similar volume collected.

For exposed soils, such as surface soils or exposed excavation sidewalls/bottom soils, the entire mass of soil collected at a single point represents an increment. These increments are collected using VOC coring devices (see Figure 5-12) and combined in a sample bottle containing a predetermined volume of methanol (see Figure 5-11). Thus, VOC ISM samples of exposed soils are collected and combined in similar fashion as non-VOC ISM samples with the exception that they are field-preserved in methanol.

ISM sampling may also be used for VOCs in the subsurface. As previously discussed in Sections 3 and 5.3.2.1, ideally the entire mass of soil collected in a subsurface core across the targeted DU depth represents the increment for that boring. The entire mass, therefore, would be preserved in methanol and incorporated into the ISM sample for the targeted soil layer. Realistically, this task is impractical, since the volume of methanol required to preserve entire core increments or the combination of increments from multiple cores would be impractically large. Additionally, preserving the entire core would prevent increments for non-VOC contaminants to be collected, if required.
Instead, the core may be subsampled by collecting numerous, small (e.g., 5 g) “plugs” at regularly spaced intervals along the targeted DU depth interval of the subsurface core. As with ISM VOC sampling of exposed soil, the plugs are immediately placed in a sampling bottle containing a predetermined volume of methanol. Figure 5-13 shows an example of this type of ISM VOC sample collection from subsurface cores. Nominal 5 g plugs of soil can be collected across the core using a VOC coring device (see Figure 5-12). The spacing interval of the VOC plugs along the core interval should be determined during the systematic planning process. It is possible to determine the optimal spacing on a site-specific basis, through the collection and analysis of differently spaced plugs along the core interval. However, based on limited field experience to date, plugs should be located no more than 2 inches apart as a starting point. This distance was determined to be adequate to capture the potential heterogeneity of VOC concentrations along the vertical length of the core. It may be necessary to decrease the spacing depending on the site-specific distribution of contaminant concentrations and DQOs. The coring device used to collect the increments should be filled completely so that each increment has the same volume of soil. The complete soil plug must be transferred to the sample container. Additionally, the ISM sampler should be aware of potential volatile loss once the core is opened. ISM VOC increments should be collected and preserved as quickly as possible to minimize potential loss. Potential loss of COPCs due to volatilization during collection of ISM increments is expected to be similar to discrete sample collection by USEPA SW-846 Method 5035A for the same sample density across a subsurface core.

In general, the potential to combine a larger mass of soil from multiple plugs from a subsurface core spaced along the entire length of a targeted DU depth results in a VOC sample that is more representative of the soil core. However, the handling and shipping of large volumes of methanol, as well as tracking and combining preserved increments into a single ISM sample, may present logistical issues, as discussed in the following paragraphs.

Soil increments should remain completely submerged in methanol at all times. If increments are combined in the field, it is important to use a volume of methanol large enough to accommodate all of the increments. Project planning must determine the number and size of increments (see Section 3). Laboratory personnel should be consulted during systematic planning so that sample size, methanol volume, and bottle size are determined in advance.

Shipment of solvent to and from the sampling activity can be problematic. When possible, methanol should be transported to the field via a surface transport to avoid or mitigate volume limitations common in air transport. Guidelines for the transportation of a solvent such as methanol can be found in 49 CFR §172, “Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, Training Requirements, and Security Plans.” Shipments via air transport may also be required to adhere to International Air Transport Association Dangerous Goods Regulations (IATA DGR, IATA 2011).
If the larger volume of methanol presents logistical problems for shipping which cannot be satisfactorily addressed, alternatives can be considered in consultation with the laboratory. With procedures and protocols in place ahead of time, these alternatives may include the following:

- The larger volume of methanol could be subsampled in the field, prior to shipment to the laboratory. With this option, the complete ISM methanol-preserved sample is disaggregated/extracted in the field by shaking periodically for at least 24 hours, allowing the solids to settle, decanting or pipetting 20–30 mL of methanol into a vial, and shipping this aliquot to the laboratory for analysis. The total mass of the ISM soil sample, as well as the total volume of methanol, must be recorded and provided to the laboratory.

- Increments for VOC analysis could be collected and preserved with methanol individually (e.g., 5 g soil in 5 mL methanol in volatile organic analysis vials per USEPA SW-846 Method 5035A) and submitted to the laboratory for combination of methanol aliquots before analysis. The laboratory would remove equal aliquots of methanol from all individual increment vials and combine them in a single vial to represent the complete ISM VOC sample, using the methanol handling techniques described in USEPA SW-846 Method 5035A (see Figure 5-14). This option also allows for analysis of individual increments or
alternate combinations of increment groups, if required. Additionally, this option allows flexibility for varying the number of increments without having a large variety of large volume ISM sample bottles. Disadvantages include increased supplies, labor costs, and sample tracking logistics.

![Image of methanol aliquots](image)

**Figure 5-14. Example of methanol aliquots from individual 5 g field-preserved increments being combined in the laboratory.**

- Individual increments could be collected in separate sampling devices that have vapor-tight seals and are designed for zero headspace (e.g., Core N’ One™, EnCore, or equivalent type sampler), and submitted to the laboratory at the appropriate temperature and within appropriate time frames (typically 24–48 hours) for combined placement in methanol before analysis.

- To fall under the small-quantity exemption of the shipping regulations, ISM volatile “subsets” could be collected, preserved with methanol in the field, and submitted to the laboratory for combining before analysis. For example, six increments of 5 g each would be collected in an appropriate container containing 30 mL of methanol. Five of these volatile subsets would be collected for a 30-increment ISM sample and submitted to the laboratory. The laboratory would then combine equal methanol aliquots from the five subsets for analysis.

ISM VOC sampling procedures should minimize soil disturbance and possible VOC loss due to volatilization. For this reason bottles that have a narrow neck or other means of restricting volatilization losses and containing the volume of appropriate solvent should be prepared prior to the sampling activity. Typically, the bottle and solvent are prepared and pre-weighed at the laboratory prior to shipment to the field. This method allows for laboratory calculation of the final ISM soil mass. The volume of solvent should at least equal the mass of soil that will be introduced. The headspace to preserved sample ratio (methanol + sample) should be less than or equal to that commonly achieved with discrete methanol VOC preserved samples (e.g., ~32 mL
headspace to 8 mL preserved sample). Details should be specified in the SAP, and any alterations due to unforeseen field conditions should be recorded in field logs. When target analytes require immersion in a solvent, trip and field blanks (no sample added) should be included, depending on DQOs. For example, when sampling for VOCs, if samples are immersed in methanol in the field, then trip blanks and field handling blanks, that is, bottles containing this solvent, should travel to and from the field and the field blank bottle(s) should be opened in the field under the same conditions and for the same amount of time as the sample bottles.

Increments should be collected using tools that minimize the loss of VOCs during sample collection and allow the collection of at least a 5 g mass of soil. Special coring tools should be used for the collection of sample increments to be analyzed for VOCs, and increments should be quickly transferred to bottles containing methanol or another appropriate solvent (Hewitt et al. 2008). Syringe-type devices that can be pushed directly into the soil are preferable (e.g., Core N’ One™, Terra Core Sampler, Easy Draw Syringe® and PowerStop Handle®, etc.). Examples of VOC coring tools are depicted in Figure 5-12. These types of devices, which are available in different sizes, can also be used for the collection of samples to be tested for nonvolatile chemicals. The device is pushed into the soil and retracted, and the increment collected is immediately extruded into a container with a premeasured volume of solvent (e.g., methanol). This procedure is repeated with each increment. Sampling devices can be used within a DU without decontamination but should be decontaminated or disposed of between DUs.

Additionally, a separate, unpreserved soil sample for percent moisture determination should be collected if necessary to report the ISM VOC results on a dry-weight basis. Typically, the unpreserved soil sample should be collected in the same manner as the ISM VOC samples, with a second increment collected at each ISM increment location and placed in an unpreserved container (4 ounces or larger) and submitted to the laboratory.

A minimum of a 1:1 ratio of solvent volume to sample soil mass (i.e., 1 mL of methanol to 1 g of soil) is recommended. This procedure is a conservative recommendation, since a 5 g plug of soil typically has a volume of around 3 mL. Soil increments should remain completely submerged at all times. Additional solvent may be required to ensure that the sample mass is completely submerged by the solvent. This requirement should be discussed with the laboratory. Select the sample container based on the total mass of soil to be collected and solvent required (e.g., 30 increments of 5 grams, approximately 3 mL volume of solid material per increment). For 30 increments a minimum of 150 mL solvent is recommended (see Figure 5-11). Use a container that is large enough to accommodate additional solvent (if needed) and to prevent loss of solvent through splashing as soil increments are dropped into the container. The headspace to preserved sample ratio (methanol + sample) should be less than or equal to that commonly achieved with discrete methanol-preserved VOC samples. Potential headspace loss in ISM VOC samples is expected to be comparable to conventional discrete methanol preserved VOC soil samples (refer to USEPA SW-846 Method 5035A). Note: An unpublished study from Hawaii using a large bottle with methanol-preserved VOCs was stored in the sun and repeatedly opened over the course of the day to simulate increment additions. VOC recovery was better than 80% for all analytes except dichlorodifluoromethane.
Typically, a 24-hour period is a long enough period to extract VOCs from most soils. Tight clays are an exception and may take several days (Hewitt et al. 1992). Therefore, caution should be taken if the plugs of soil do not readily disperse when submersed in methanol. Soils should be completely disaggregated or dispersed in the solvent to ensure efficient extraction.

Guidance on using ISM for the collection and handling of samples for the analysis of VOCs has been published by the State of Alaska (ADEC 2009). The Alaska guidance recommends that consultants provide a sampling and analysis work plan to the overseeing regulatory agency for review and comment prior to collecting any ISM samples. The analytical laboratory should also be consulted prior to sample collection to discuss sample containers, sample handling, solvent type and volume, shipping of samples in methanol, anticipated analytical detection limits, etc. A potential drawback of ISM for VOCs is that the methanol preservation (high-concentration method) approach does result in lower sensitivity. The methanol dilution step causes elevated analytical detection limits, method detection limits (MDLs), reporting limits (RLs), practical quantitation limits (PQLs), etc., as compared to the direct soil purge-and-trap, low-concentration method techniques. Analytical detection limits could be elevated above relevant screening levels for certain targeted contaminants (see Section 6.3.2). If the analytical detection limits (or other issues) present difficulties in using ISM for VOCs, this issue should be discussed with the laboratory and the overseeing regulatory agency prior to sample collection. If the projected analytical detection limits are too high to be of use or some other issue restrains the use of these methods at a specific site, then alternative approaches may need to be used. Options may include alternate analytical methods/techniques, such as selective ion monitoring (SIM), to achieve lower detection limits or select discrete sampling via USEPA SW-846 Method 5035A low-level VOC sampling. Research to improve detection limits from ISM VOC samples is ongoing and expected to improve in the near future. Consult with the laboratory for the latest detection limit capabilities.