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REPLY TO
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CEHNC-CX-EG

21 July 2009

MEMORANDUM FOR SEE DISTRIBUTION

SUBJECT: Implementation of Incremental Sampling (IS) of Soil for the Military Munitions Response Program, Environmental and Munitions Center of Expertise Interim Guidance Document (IGD) 09-02

1. Interim Guidance Document 09-02 (enclosed) describes the general principles, concepts, and considerations necessary for the successful implementation of the Incremental Sampling methodology in sampling of surface soil for Munitions Constituents at Military Munitions Response Program (MMRP) projects. The document is intended to help determine proper application of the sampling methodology, and identify important design, execution, and data evaluation issues relevant to Project Delivery Teams and oversight personnel. The document has been coordinated within the U.S. Army Corps of Engineers (USACE), with selected USACE contractors, and the U.S. Environmental Protection Agency.

2. This guidance is applicable to the geographic military Districts and MMRP Design Centers, project managers, technical personnel, and contractor personnel executing and managing MMRP projects. It may also be applicable to chemical characterization of soil for Civil Works projects, and Hazardous, Toxic, and Radioactive Waste (HTRW) projects.

3. The information presented in this interim guidance can be used immediately. The interim guidance will remain in effect indefinitely, unless superseded by other guidance or policy. This guidance is approved for public release and unlimited distribution.

4. For additional information or to submit suggestions for improvements to this guidance, please contact Mr. Hugh Rieck at (402)-697-2660.

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Interim Guidance 09-02
20 July 2009

ENVIRONMENTAL QUALITY

Interim Guidance 09-02

Implementation of Incremental Sampling (IS) of Soil for the Military Munitions Response Program

Environmental and Munitions Center of Expertise
Interim Guidance

AVAILABILITY

Electronic copies of this and other U.S. Army Corps of Engineers, Environmental and Munitions Center of Expertise Military Munitions Response Interim Guidance Documents are available on the Internet at http://www.environmental.usace.army.mil/corpguide.htm. Publications are provided in portable document format (PDF).

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List of Acronyms

ADEC	Alaska Department of Environmental Conservation
AFB	Air Force Base
BERA	Baseline Ecological Risk Assessment
BIP	Blow-In-Place
BTAG	US Army Biological Technical Assistance Group
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CRREL	USACE Cold Regions Research and Engineering Laboratory
CSM	Conceptual Site Model
CWM	Chemical Warfare Materiel
DAF	Dilution Attenuation Factor
DMM	Discarded Military Munitions
DoD	Department of Defense
DQO	Data Quality Objective
EDQW	Environmental Data Quality Workgroup
ELAP	Environmental Laboratory Accreditation Program
EM	USACE Engineering Manual
EMCX	Environmental and Munitions Center of Expertise
EOD	Explosive Ordnance Disposal
EPA	United States Environmental Protection Agency
EPC	Exposure Point Concentration
FUDS	Formerly Used Defense Sites
GPS	Global Positioning System
HDOH	Hawai'i Department of Health
IS	Incremental Sampling
ITRC	Interstate Technology and Regulatory Council
LCS	Laboratory Control Sample
MC	Munitions Constituents
MD	Munitions Debris
MDC	Maximum Detected Concentration
MEC	Munitions and Explosives of Concern
MIS	Multi-Increment Sampling
MMRP	Military Munitions Response Program
MPPEH	Material Potentially Presenting Explosive Hazard
MRA	Munitions Response Area
MRS	Munitions Response Site
MS/MSD	Matrix Spike / Matrix Spike Duplicate
OB/OD	Open Burn / Open Detonation
PA	Preliminary Assessment
PAH	Polycyclic Aromatic Hydrocarbon
PCL	Protective Concentration Level
PE	Performance Evaluation
PWS	Performance Work Statement
QA	Quality Assurance
QC	Quality Control

QSM	Quality System Manual
RI	Remedial Investigation
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SI	Site Inspection
SLERA	Screening Level Ecological Risk Assessment
SOP	Standard Operating Procedure
SOW	Scope of Work
SSL	Soil Screening Level
SW	Solid Waste
TPP	Technical Project Planning
UCL	Upper Confidence Limit
UTL	Upper Tolerance Limit
USACE	US Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UXO	Unexploded Ordnance
VSP	Visual Sampling Plan

Executive Summary

This interim guidance document describes the general sampling principles, concepts, and other considerations necessary for the successful application of Incremental Sampling (also called Multi-Increment Sampling) to sampling of surface soil for Munitions Constituents at Military Munitions Response Program projects.

Due to the broad ranges of project objectives, contaminant properties, and site conditions to which Incremental Sampling can be applied, the document does not provide specific "cookie cutter" sampling designs, but rather provides general guidelines for designing sampling plans and evaluating Incremental Sampling data. The guidance is based on published sampling theory and field demonstrations of the Incremental Sampling methodology. Demonstration studies for sampling energetic compounds, conducted by the USACE Cold Regions Research and Engineering Laboratory at active ranges, form the basis of sample collection and processing recommendations made in US EPA Method 8330B. The concepts and approaches presented in this guidance address additional Munition Constituent analytes, and a broader range of project objectives applicable to Formerly Used Defense Sites as well as active sites, and to projects in Preliminary Assessment, Site Inspection, and Remedial Investigation phases.

The greatest source of error (uncertainty) in environmental soil sampling data is not from laboratory analytical error, but rather results from sample collection and processing procedures that do not adequately address the non-uniform distribution of contaminants in soil. This heterogeneity occurs at all scales, from the variation in composition of individual soil particles, to the uneven distribution of contaminants across a site.

Incremental Sampling is a sample collection and processing methodology having specific elements designed to control data variability due to heterogeneity in contaminant distribution. Required elements are in both the field collection and laboratory processing and sub-sampling procedures. The objective is to obtain a single sub-sample that contains all analytes in exactly the same proportion as the entire sampled area. This is achieved by collecting a sufficient number of discrete "increments" (typically 30 to 100) in an unbiased manner from throughout a specified area (the Sampling Unit), combining and variously processing the increments, and incrementally sub-sampling the processed material to obtain a representative aliquot for analysis. Properly executed, the method provides unbiased, representative and reproducible estimates of the mean concentration of analytes in the Sampling Unit.

The guidance emphasizes that the key to successful Incremental Sampling design lies in the proper designation of Sampling Units. Sampling Units should be based on the specific end use of the data that must be identified during comprehensive systematic planning process. Comprehensive and detailed Systematic planning is particularly important when implementing an Incremental Sampling strategy. Sampling Units should be defined so that the mean concentration value obtained is relevant to an explicitly articulated end use of the data.

Interim Guidance for Implementation of Incremental Sampling of Soil for the Military Munitions Response Program

Note: The practical application of Incremental Sampling for munitions constituents in soil is a relatively new and evolving subject. Several sections in this interim guidance are the subject of current research, particularly the processing of munitions constituent metals. This interim guidance will be updated as more information is obtained and more experience gained.

1 Introduction

Incremental Sampling, or Sampling by Increments, are terms used by Pierre Gy to describe the sampling theory he developed for obtaining representative samples from heterogeneous media composed of discrete particles (e.g. Gy, 1954; 1992; and 1998). Multi-Increment[1] Sampling is based on the fundamental principles developed by Gy, and that term also has been applied to the sampling methodology using the incremental approach.

The purpose of this document is to provide interim guidance for the development and implementation of incremental sampling (IS) strategies for munitions constituents (MC) in surface soil for the Military Munitions Response Program (MMRP). Note that any requirement for IS is at the Performance Work Statement (PWS) and Scope of Work (SOW) level, not at the guidance level. This guidance applies in situations where the PWS/SOW requires IS, or where the project team believes IS is the best sampling method to achieve data quality objectives and the contractor agrees to do IS during negotiations in performance based acquisitions.

Incremental sampling is a sampling method to provide unbiased and reproducible estimates of the mean concentration of analytes in a specifically defined volume of soil, i.e. a specific population. IS is a sampling methodology, not a sampling design. The Corps of Engineers Technical Project Planning Process (TPP) guidance (USACE 1998) describes a process for systematic planning, which determines the sampling objectives, sampling strategy, and sampling design that govern how the IS methodology will be adapted and applied.

In many cases, IS provides the data quality needed to satisfy MMRP project objectives more effectively than traditional grab sampling. The key to successful use of any sampling methodology, incremental, discrete, or other, is successful project planning. Data collection should be planned with the end use of the data in mind. This requires:

- a working Conceptual Site Model (CSM), including assumed or inferred mechanisms of analyte distribution or release, processes affecting transport and environmental fate, and mechanisms of potential exposure or other risk

[1] Multi-Increment is a registered trademark of EnviroStat, Inc.

- project objectives that are clearly formulated and explicitly stated

The goal of this document is to set out general design principles and considerations that will help those designing soil sampling plans for MMRP projects formulate clear objectives, and obtain representative data appropriate for comparison with decision criteria. Decision criteria included soil screening values, groundwater protection levels, or background concentrations to determine if a release occurred, the nature and extent of contamination; and use in human health and ecological risk assessment.

Available guidance for the general design and application of IS to environmental investigations is limited. Regulatory guidance for IS, or Multi-Increment Sampling, is limited to that published by two states, Alaska (2009) and Hawaii (2008). Both address contaminants other than explosive compounds. The most widely known description of IS for an environmental use is the US EPA publication of SW 846 Method 8330B, Appendix A (USEPA, 2006b). The specific sample collection and processing procedures described in Method 8330B were based primarily on studies conducted by the USACE Cold Regions Research Engineering Laboratory (CRREL). Those studies were designed to demonstrate and develop the methodology for application to the investigation of explosive compounds released at active military testing and training ranges. All recent research in the area of secondary explosives contamination at ranges supports the use of IS or multi-increment sampling, rather than discrete sampling (USACE, 2007, EM 1110-1-4009, Section 10-4, p. 10-6).

Not all procedures described in Appendix A of Method 8330B are necessarily appropriate for other environmental applications of IS. The Department of Defense (DoD) Environmental Data Quality Workgroup has published *Guide for Implementing EPA SW 846 Method 8330B* (EDQW, 2008), which strongly cautions against rote application of Method 8330B for all analytes and sampling objectives. When adapting IS for a specific site investigation, the project team needs to ensure that all aspects of the sampling and processing design are defined to meet project goals for each contaminant of concern and sampling objective.

2 Scope

This guidance focuses on development of sampling plans to obtain reliable mean analyte concentrations of conventional MC in surface soil using the IS method. This document does not address sampling for chemical warfare materiel (CWM). Depending on sampling design, the estimated mean concentrations can be used to assess whether potential MC

- are present within the sampled area at an average concentration greater than the analytical method detection limit or reporting limit
- may pose an unacceptable risk to human health, or ecological receptors
- may contribute to significant contaminant concentrations to groundwater
- concentrations exceed mean background or ambient concentrations unrelated to munitions activities

The principles and concepts discussed are applicable to a wide range of sampling investigations and circumstances. They can be adapted to sediment, sub-surface soil, or surface water sampling. Applications of IS other than sampling surface soil are not well developed and are beyond the scope of this guidance, although some considerations for subsurface sampling are mentioned in Section 4.1.3.

The primary focus of this document is on the field aspects of IS. Some laboratory issues that influence or overlap with field considerations and project planning are addressed. It does not address all logistical issues that may need to be resolved prior to or during sampling.

3 Concepts of Incremental Sampling

Incremental sampling is a sample collection and processing method having specific elements designed to obtain an aliquot for analysis that contains all analytes in exactly the same proportion as the entire sampled area. The required elements form a two-tiered methodology that addresses both:

1) field sample collection, to ensure that a representative sample is collected, and

2) laboratory processing and incremental sub-sampling procedure to ensure that a representative sub-sample is analyzed.

The fundamental objective is to obtain a sample having mean analyte concentrations that are representative of a specifically defined population - i.e. the sampled volume of soil or Sampling Unit.

A Sampling Unit (sometimes termed Decision Unit) is the area and depth of soil (the sampled population) to be represented by the sampling process. Sampling Units must be delineated so that the mean analyte concentrations obtained are directly relevant to well defined project objectives. When using IS, project objectives and decision criteria must be satisfied in terms of mean analyte concentrations of the Sampling Unit(s).

There are two approaches to estimate a mean analyte concentration for a Sampling Unit:

1) collect and individually analyze a large number of discrete samples (i.e. individual, unique specimens), or

2) collect a sufficient number of "increments" in an unbiased manner from throughout the Sampling Unit and analyze a representative sub-sample from the combined increments.

Collecting and combining a large number of increments from a Sampling Unit to produce one incremental sample is the physical analog of collecting and separately analyzing an equal number of discrete samples from the Sampling Unit and arithmetically averaging the results. However, "Because there is no spatial averaging of soil concentrations with discrete sampling, a much larger number of soil samples [must be analyzed] to produce a reliable estimate of the mean contaminant concentration" (USEPA, 2002a, p. 2-8).

The greatest source of variability (error) in soil sample data results from heterogeneity. Heterogeneity is present at all scales, from the micro-scale due to compositional differences of individual particles (compositional heterogeneity), to the macro-scale due to the irregular distribution of analytes across a site (distributional heterogeneity). IS has specific requirements that effectively reduce sampling error due to both compositional heterogeneity and the distributional heterogeneity within a Sampling Unit.

Compositional heterogeneity, or "fundamental error" per Pierre Gy's theory (Pitard, 1993; Gy, 1998), requires that some minimum representative mass is needed for a sample. The required minimum representative mass for soil depends on particle size and analyte concentration. Ten grams is the minimum sample mass recommended for laboratory analysis by Method 8330B after laboratory processing (including particle size reduction by grinding). Note that 10 grams may not be sufficient mass to achieve good reproducibility if "nuggets" of contaminants are present, e.g. in a sample that has not been ground.

Distributional heterogeneity, or grouping and segregation error, is addressed by collecting a sufficient number of increments to form a field sample having adequate volume or mass. A 1 to 2 kg (dry weight) sample usually ensures that sufficient sample mass has been collected. To be representative of the Sampling Unit, increments must be collected in an unbiased manner from the entire Sampling Unit.

Field sampling procedures that distinguish IS from conventional composite sampling include:

- collecting increments from a single Sampling Unit (population) specifically delineated to meet a project objective
- collecting a sufficiently large number of increments (typically 30 to 100) to address the distributional heterogeneity of analytes;
- ensuring that the increments are of equal mass
- ensuring that the increments are collected from throughout the entire Sampling Unit in an unbiased manner;
- collecting an adequate total sample mass (typically 1 to 2 kg dry weight) to overcome effects of compositional heterogeneity due to the inherent particulate nature of soil and sediment;

Laboratory processing and sub-sampling procedures that enhance representativeness include (for non-volatile analytes):

- air drying of the *entire* field sample (for ease of handling);
- reducing particle size by sieving and possibly grinding, depending on target analytes and DQOs (consistent with Pierre Gy's theory and many EPA SW 846 Methods);
- multi-increment laboratory sub-sampling from the entire processed sample to obtain an aliquot for analysis having sufficient mass to reduce the fundamental error to < 15%.

4 Systematic Planning

Systematic planning determines the type and amount of data to be collected, the locations to be sampled, and the field and laboratory methods that will be used. Sampling design must be tailored specifically to support the intended end use of the data. Without systematic planning, the likelihood data will not meet project objectives, i.e. be unusable for the intended purpose, is unacceptably high.

Systematic planning must formulate project-specific data quality objectives (DQOs) sufficiently explicit that criteria for meeting them are unequivocal and measurable. EM 200-1-2 *Technical Project Planning (TPP) Process* (USACE, 1998) is the USACE guidance for conducting systematic planning. The TPP process was developed to provide comprehensive planning guidance to ensure effective and efficient progress to site closeout within all project constraints. EM 200-1-2 describes the TPP process for identifying project objectives, identifying data required to meet those objectives and designing data collection programs. The preparation of DQO statements is a culmination of many of the TPP activities.

The TPP process involves four phases of planning activities. Phase I activities bring together a team to identify the current project and to document both short-and long-term project objectives through completion of all work at a site (site closeout). Phase II efforts involve an evaluation to determine if additional data are needed to satisfy the project objectives. The data need requirements for the additional data are then identified during the balance of Phase II efforts. Phase III activities involve identifying the appropriate sampling and analysis methods for the data needed. During Phase IV, the TPP team finalizes a data collection program that best meets the customer's short- and long-term needs within all project and site constraints.

Guidance similar to that in EM 200-1-2 for preparing DQOs is provided in the *DQO Process*, a seven-step strategic planning process discussed in *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA QA/G-4, USEPA, 2006a), and in *Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives* (ASTM D5792 -02, 2006). EM 200-1-2 provides a "crosswalk" to the EPA's 7-step process as an appendix.

Because IS provides an estimate of only the mean concentration of an analyte within an individual Sampling Unit, the size and configuration of Sampling Units is critically important in determining the relevance of the data to its intended end use. Project objectives should be framed in terms of a specific quantitative decision that can be made based on mean analyte concentrations of the individual Sampling Units. An appropriate IS design requires that the primary end use of the data (e.g. what the results will be compared to) be explicitly identified during systematic planning, and the Sampling Units designed so that their mean concentrations are relevant for that end use. Sampling Units designed for one end use may not be equally appropriate for other uses. Deficiencies in systematic planning become starkly evident if Sampling Units are inappropriate to meet the intended end use of the data.

Figure 4-1 – Flow Chart Overview of Systematic Planning for Site Inspection

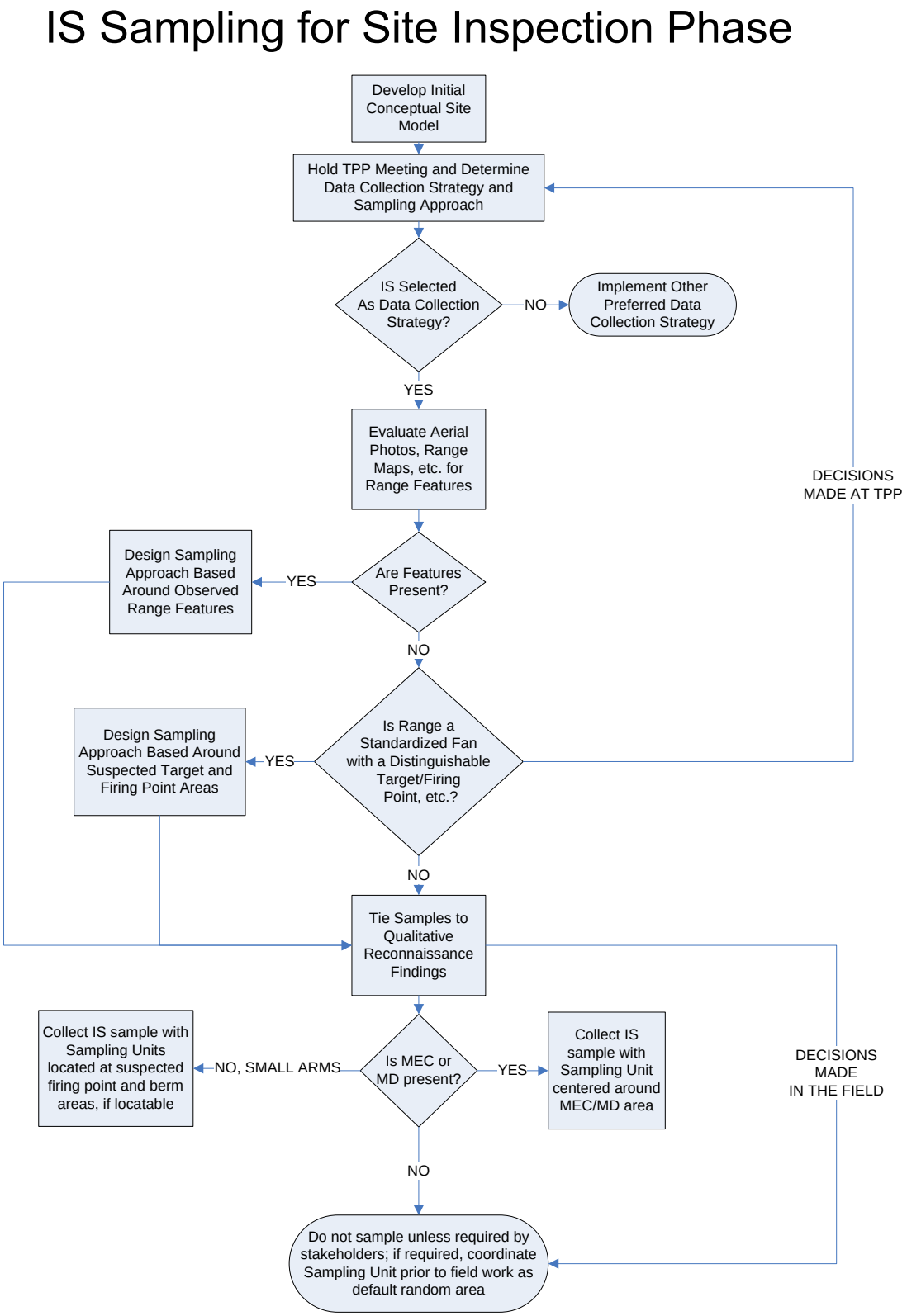
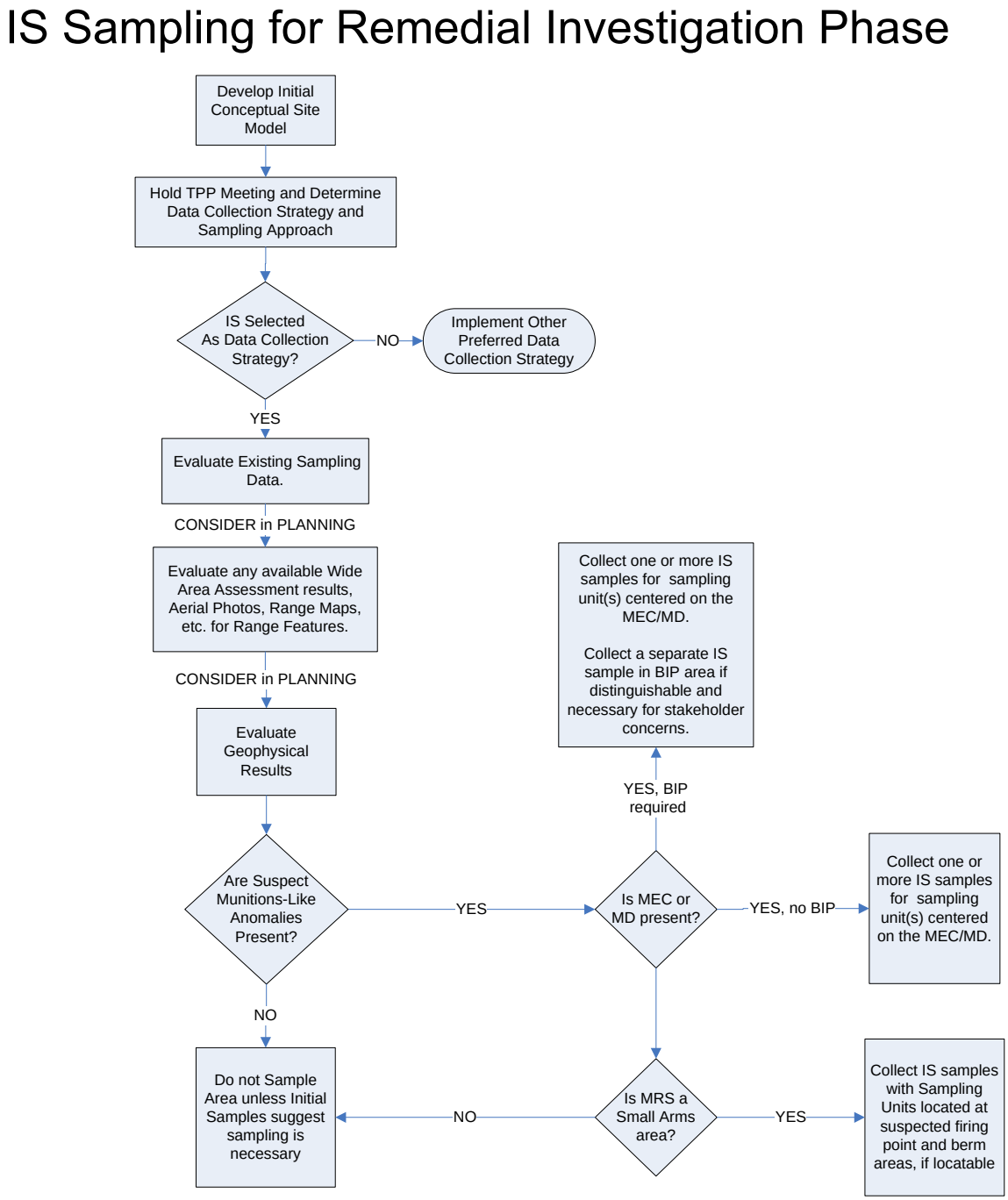


Figure 4-2 - Flow Chart Overview of Systematic Planning for Remedial Investigation



4.1 Quality Control

Field sampling reproducibility of IS results should be subject to Quality Control (QC) requirements similar to those traditionally required to demonstrate only laboratory analytical reproducibility. Field replicates provide a measure of the variability or "total error" of the data set (field sampling error + laboratory sample processing and subsampling error + laboratory analytical error). It is important to emphasize that field replicates for IS are not field splits; rather they must be independently collected incremental samples from the same Sampling Unit. Reproducibility of IS results by replicate sampling is key to demonstrating that data are scientifically defensible and representative (USEPA, 2006b, Method 8330B), and the only means by which confidence can be quantified.

> ***If evidence for representativeness is not presented, then the data cannot be characterized as effective for project decision making.*** (Crumbling, 2001; 2002)

Targeted levels of precision or confidence for the mean concentrations and other statistical parameters should be identified in the systematic planning process. The degree of precision required to support the decision, expressed as percent Relative Standard Deviation (RSD) of replicate samples from a Sampling Unit, should be specified as part of the DQOs.

$$\%RSD = 100\ (s) / \bar{x}$$

where
s = standard deviation
$\bar{x}$ = sample mean (mean of replicate IS results)

The Sampling and Analysis Plan (SAP) should provide for enough replicate (QC) sampling to verify that the targeted precision was met in all facets of sampling and analysis.

As a general rule, collection of triplicate IS from 10% of the Sampling Units is recommended. For investigations where high precision is not required (e.g. simply demonstrating presence of a contaminant for an SI), a single IS result for most Sampling Units may be sufficient, with QC triplicates taken from only one or two Sampling units at the site to assess general adequacy of the sampling design. Where greater knowledge of precision of the results is necessary, for example where values are anticipated to be close to an action level in an RI, triplicate samples should be a default minimum, with up to 10 or more replicates from a Sampling Unit, if needed, to more closely define the uncertainty. Sampling Units delineated to determine site-specific background or ambient concentrations of analytes unrelated to the site activities being investigated should be sampled in triplicate.

The central limit theorem in statistics states that the distribution of sets of means (e.g. IS replicates), even when based on individual data having a decidedly non-normal distribution, will approach a normal distribution as the number of means (*n*) increases.

Because each IS result is an independent estimate of the mean analyte concentration determined from many points (increments) in the Sampling Unit, the variance of replicate IS samples from the same Sampling Unit tends to be low, and the distribution of values (the distribution of estimated means) tends to be normal. This simplifies statistical evaluations, and precision can be quantified using relatively few samples.

The fact that replicate IS results from a Sampling Unit tend to have low variance and normal distributions, results in significantly lower calculated 95% Upper Confidence Limits (95% UCL) of the mean than data sets of similar size collected by conventional sampling techniques.

If the total %RSD (total error) between three to five field replicates from the same Sampling Unit is less than 30%, then the sampling design and execution are likely to be adequate, and the distribution of replicate results can be assumed to be approximately normal. Although a %RSD of 30% or less from only three to five replicates alone cannot conclusively demonstrate that a distribution is normal, a larger %RSD would be indicative of deviations from normality and suggest that the field sampling design and/or laboratory processing procedures were not adequate to control effects of distributional or compositional heterogeneity. It is anticipated that only in special cases would the DQOs require collecting a sufficiently large number of IS replicates (e.g. 10 or more) for robust statistical evaluations. The State of Hawaii (HDOH, 2008, Section 4.2.5.1) is accepting 35% RSD based on triplicates for the same purpose, in their guidance. The Alaska Department of Environmental Conservation guidance states a requirement that %RSD for triplicate IS samples be 30% or less (ADEC, 2009). Hewitt and others (2008) suggest that if the %RSD is less than or equal to 30%, one could assume a normal distribution, and thus calculate a meaningful UCL based on only three IS results. Note that at very low concentrations (i.e. at or near Method Reporting Limits), %RSD (relative error) will tend to increase even though absolute differences in results may be small.

If there are a number of similar Sampling Units (e.g., areas affected by similar activities in the CSM), collection of triplicate IS from a subset of the Sampling Units (e.g., 10%) may be used to make inferences about the precision of results for the other Sampling Units from which only one IS sample was collected. The Hawai'i technical guidance for IS (HDOH, 2008, Section 3.7.2) suggests that QC triplicate samples typically be analyzed for "10 percent of the decision units" (i.e. Sampling Units).

It is important to understand that the above discussion suggests general guidelines for acceptable %RSDs. They are merely suggestions to evaluate whether or not IS results can be assumed to be reasonably representative estimates of mean analyte concentrations in a Sampling Unit, and whether or not indications of non-normal distribution are evident in a IS data set. If IS gives an estimated mean concentration that is many times greater than or less than an action level or screening value, a much larger %RSD and high degree of uncertainty may not affect data usability. But by the same reasoning, if an estimated mean concentration is close to an action level, a greater degree of precision may be desirable.

If improved precision in total error (field sample replicate %RSD) is required to meet project DQOs, attempt to determine the source of the excessive error. Unacceptably large total error is more likely to result from an insufficient number of increments per unit area (increment density) or other field sampling causes, than from laboratory analytical variability. Laboratory sample processing and sub-sampling error can also be a significant source of variability, and should be evaluated as discussed in Section 9.1.3.

4.2 Project Constraints that may Influence Selection of IS

By adjusting the size of the Sampling Units, IS can be adapted to address a very wide range of project objectives. Sampling Units as small as a few square meters can be used to provide spatial resolution necessary for some project objectives (e.g. accurately delineating the extent of a release requiring remediation). Nonetheless, IS may not be the most cost-effective or preferred sampling method for all project objectives or data end uses. Some examples may include:

- where an inexpensive (per individual measurement) field technique that provides a very large number of discrete measurements can meet project objectives and DQOs for precision and accuracy (e.g. field portable XRF, or scintillometer)

- where the nature of the analyte or physical limitations of the method limit its applicability (e.g. analysis of VOCs requiring lower reporting limits than can be achieved by methanol extraction)

- where safety clearance concerns at sites having a high density of UXO or other hazards may prevent proper IS.

Adequate characterization of acute or sub-chronic risk from a very small concentrated mass of contaminant may be an unrealistic objective using soil data, and may not be met with certainty by IS, or even if a large number of discrete samples are analyzed. In some situations where laboratory analyses are required to meet DQOs, inexpensive field analytical methods using a large number of grab samples or readings can help develop appropriate Sampling Units for IS for laboratory analysis (the Triad approach).

5 Implementation Issues

A lack of general guidance and understanding of the requirements for proper IS has hindered the regulatory acceptance and widespread use of the methodology. A broad range of considerations must be taken into account to develop and implement a sound IS sampling plan for the MMRP. Many of these considerations will be site specific and depend on the sampling objectives and analytes of interest. Just as with conventional discrete sampling, there are no “cookie-cutter” templates that will assure successful and scientifically defensible IS implementation for all circumstances. An understanding of the principles and concepts of IS is needed.

Much of the recently published information on IS for MMRP projects has involved studies on active military ranges. Appendix A of SW 846 Method 8330B (USEPA, 2006b) evolved from the collection and analysis of explosive and propellant compounds in surface soil samples from active military ranges. Objectives included investigation of mass loading of contaminants for range sustainability issues, and the demonstration/validation of the IS methodology, for that application. Thus, Appendix A of 8330B addresses analytes, objectives and requirements that may differ from those for other soil investigations where IS might be used (EDQW, 2008). Not all of the recommendations in Appendix A of Method 8330B will necessarily be appropriate for other MMRP analytes and project objectives, but it presents valuable information on the underlying principles of IS.

5.1 Acceptance of IS Data

Although some regulatory agencies recently have begun requesting the use of IS the acceptance of IS data by state and federal regulators must be considered. There may be regulatory prohibitions regarding the use of IS or composite sampling. There also may be prejudice, misconceptions, and biases against any form of composite sampling. Data from poorly conceived or poorly executed IS may not be acceptable because project objectives and DQOs were not clearly defined and the data cannot properly inform the decision to be made. Some project team members or stakeholders may be concerned that the mean concentrations obtained by IS do not provide spatial information on the distribution of contaminants within a Sampling Unit. They may also suggest that the identification of "hot spots" *per se* would be a technically valid objective, but without adequately defining the term. ITRC (2008) lists many definitions of “hot spot” that differ widely between different State regulatory agencies.

A project team needs to be prepared to address concerns regarding IS “diluting out hotspot contamination”, as well as not obtaining information about the spatial distribution of contaminants within a single Sampling Unit. All stakeholders should have a basic understanding of IS, and how the sampling objectives are explicitly formulated to obtain meaningful and reproducible results.

Soil investigations typically evaluate concentrations relative to natural or ambient background concentration, support risk assessments, evaluate long-term mass loading and migration to groundwater, or provide estimates of site-specific natural or ambient background concentrations. IS results from Sampling Units of relevant size are appropriate for comparison with such fixed numeric thresholds (e.g. risk-based screening values protective of direct exposure, or protective of groundwater from leaching of soil contamination). Because soil concentrations deemed protective are model-based mean concentrations over an exposure area or source specified in the model, the mean concentration in the exposure area or source area is most appropriate for comparison. Natural background concentrations also are mean concentrations. Variability ("hot spots") within a Sampling Unit appropriately designed for the end use of the data does not matter.

Risk-based soil screening levels (SSLs [2]) to which soil results are often compared are intended to evaluate the average concentrations of analytes, within a specified volume (area) of soil (i.e. the Exposure Unit) to which a receptor would be exposed, for a specified period of time. Generally, a baseline risk assessment evaluates the mean concentration over an exposure unit, considering all exposures within that area as equally possible. The risks associated with chronic exposures are then assessed by evaluating those average or mean concentrations with exposure factors and the appropriate exposure/toxicity values. Use of a 95% UCL of the mean is simply to ensure that the average concentration is not underestimated.

If exposure of sensitive subpopulations within relatively small areas is a valid concern (such as a children's play area contaminated with lead), smaller exposure units are of concern, and smaller Sampling Units should be delineated to obtain mean concentrations representative of the smaller exposure areas. However, a Sampling Unit generally should not be smaller than the smallest area identified as being of concern. From a risk assessment perspective, the only "hot spots" (an area smaller than an exposure unit) of valid concern would be concentrations of an analyte sufficiently high to pose unacceptable risk from acute or sub-chronic exposure.

Stakeholders need to determine and agree upon the appropriate delineation of the Sampling Units (equivalent to the appropriate exposure unit in many cases) to meet sampling objectives that are expressed in terms of mean contaminant concentrations within those Sampling Units.

There may be situations where IS will simply not be allowed and discrete sampling will be selected. In these cases, clearly defined DQOs will help insure that the project objectives are met with an understanding of the uncertainty and the corresponding confidence in the results. Note that even when analyzing a discrete soil sample, a single result represents a mean concentration over a (very small) volume of soil.

[2] As used in this document, Soil Screening Level (SSL) may include any potential soil screening levels that have been calculated for protection of human and ecological receptors, or for the protection of groundwater.

5.2 Comparison of IS Data to Data from Discrete Samples

A mean concentration value obtained by IS may be compared with a mean concentration from a set of discrete sampling results from the same Sampling Unit. In theory, given the same Sampling Unit (sampled population), IS and discrete samples will produce similar estimates of the population mean. If valid sampling designs were implemented, the means of the two data sets should not be statistically different from each other. However, in practice, such comparison should be done with caution.

Simple comparisons of means from replicate IS data with a mean from discrete samples, such as the Student's Two Sample t-Test, require that 1) the variabilities (the variance) of the two data sets are approximately equal, and 2) the distributions of both data sets are approximately normally distributed. If the two variances are not equal, but distributions are normal, the use of Satterthwaite's t-test is recommended. If requirements of normality and equality of variances are not met, nonparametric procedures may be applied (USACE, 2008a). Problems in comparison of means are most likely to result when the number of discrete samples is insufficient to accurately represent the population.

As discussed in Section 4.1, IS replicate data tend to have normal distribution and smaller variance than data sets of discrete values (Central Limit Theorem). A soil data set of discrete samples is likely to have a positively skewed distribution. Discrete sample results typically have higher variance than IS results. An insufficient number of discrete samples will tend to underestimate the true (population) mean because the likelihood of sampling small areas of contamination within a Sampling Unit will be small (i.e. distributional heterogeneity within the Sampling Unit will not have been adequately addressed).

Because the distributions of data produced by the two methods are typically different, percentiles from the IS and discrete data are not directly comparable. For example, it would be inappropriate to compare the IS mean from a Sampling Unit to a 95% Upper Tolerance Limit (UTL) determined from a background data set of discrete samples. The 95% UTL is the upper confidence limit for the 95^{th} percentile.

5.3 Safety Considerations

In areas where unexploded ordnance (UXO), discarded military munitions (DMM), or materials potentially presenting an explosive hazard (MPPEH) are present or may exist, field activities shall be under the direct supervision of military EOD personnel or qualified UXO technicians. Anomaly avoidance procedures in accordance with USACE Engineering Pamphlet 75-1-2, chapter 5 (USACE, 2004) will be used to ensure all sampling areas are free of UXO or anomalies that could be UXO before samples are taken. The on-site UXO technician will conduct a surface access survey and a subsurface survey for anomalies before any type of activities commence, including foot and vehicular traffic. This survey will be performed in accordance with paragraph 5-6 of the

above reference. Procedures for these measures shall be described in the Contractor's Site-Specific Work Plan and modified in their site-specific plans as required.

Although uncommon, soils containing explosives at concentrations that present risk of detonation or deflagration may be encountered in the vicinity of ruptured munitions, low-order (partial) detonations on ranges, at manufacturing facilities or in disposal areas (OB/OD, burial pits, etc.) (Department of Defense, 2004, Chapter 12). Often, visible pieces of the explosive compounds can be recognized in such situations. Visible or otherwise identifiable pieces of explosive compounds should not be incorporated into the soil samples. The Expray Kit (Plexus Scientific, Silver Springs, MD) or EPA Methods 8510, 8515, 4050 and 4051 may be useful for screening suspected explosive material prior to sample collection.

6 Sampling Strategy

Sampling strategies include non-probabilistic sampling (sometimes termed judgmental, biased, or targeted sampling), and probabilistic sampling. Non-probabilistic sampling relies on the subjective, professional judgment of the sampler to select appropriate sampling locations. The strategy often is chosen with the intent of collecting a minimal number of discrete samples to minimize cost. This approach can be effective, depending on the sampling objectives, accuracy of available information and the CSM, and the abilities of the sampling design team. However, reproducibility of the individual discrete sample results is usually unknown, and often likely to be poor. Because relatively few samples are collected, and they are collected in an intentionally biased manner, the results do not support meaningful estimation of statistical parameters (e.g., mean concentration) for a given area. Results from such sampling approaches are intended to be biased high. Any inferences drawn from the results about the overall population and unsampled locations are questionable.

Probabilistic sampling designs are statistically based (i.e. unbiased) to represent a specific population. Results can be used to make reliable inferences about the sampled population (Sampling Unit). They support statistical evaluations and can provide quantitative estimates of sampling error. Typical grid-based sampling is one example of probabilistic sampling. IS within a single Sampling Unit requires a probabilistic sampling design. (Refer to the USEPA, 2002b, QA/G-5S document for additional guidance on probabilistic sampling designs.)

Sites may be stratified into three types of areas (USEPA, 1996b):

1) Areas known or indicated to be highly contaminated,
2) Areas suspected to be contaminated, and
3) Areas unlikely to be contaminated

Relatively large areas of most MMRP sites are uncontaminated. When the project objective is to determine the nature and extent of contamination, or areas of greater uncertainty regarding contamination, sampling efforts may need to be focused on the

boundaries of contaminated areas known or likely to be contaminated. These are areas of higher variability or uncertainty.

The objective of identifying areas suspected or potentially having contaminants at levels of concern may be met with a higher degree of confidence by using a "hybrid" approach, combining probabilistic IS within appropriately sized Sampling Units located on the basis of non-probabilistic professional judgment. This is the approach typically used for an SI (Figure 4-1). Because IS sampling can cost-effectively provide more thorough coverage than discrete sampling of areas identified as most likely to contain any contamination at levels of concern, the method is less likely than discrete sampling to miss any significant contamination within a Sampling Unit.

When judgmentally targeting the locations of Sampling Units, consideration should be given not only to likely initial release mechanisms and contaminant distribution, but also to how post-release processes or disturbance may have changed the spatial distribution of analytes.

For RI objectives, the "nature and extent" of contamination must be determined. Unless we know that specific areas have been sufficiently characterized, or have other evidence that indicates that they are not contaminated, probabilistic sampling strategies in multiple Sampling Units may be required. Sampling objectives (e.g., based on current or future site use) will need to be considered to determine the required number, size, and geometry of Sampling Units to provide adequate coverage.

7 Sampling Units and Decision Units

A Sampling Unit is the specific volume of soil (i.e. the population) that is represented by a single incremental sample. It is the smallest volume of soil for which a concentration value will be obtained, and the basic unit about which a decision or conclusion based on an analytical result could be made. No information about the spatial distribution (extent) of contaminants within a Sampling Unit is obtained. A Sampling Unit is the portion of a lot (population) that is actually sampled (USEPA, 2003b). The Sampling Unit has sometimes been termed the Decision Unit.

A Decision Unit is a specific area (or volume of soil) about which a decision is to be made (Ramsey and Hewitt, 2005; HDOH, 2008; ADEC, 2009). A Decision Unit may be composed of a single Sampling Unit, or may include multiple Sampling Units. In the ideal and most direct case, the Decision Unit and Sampling Unit are the same volume of soil. Depending on project objectives and practical constraints, however, there are cases where multiple Sampling Units may be the most effective means of obtaining an estimate of mean analyte concentration in a Decision Unit. (See Pitard, 1993, Section 21.6.4.1 for in-depth discussion of sampling unit hierarchy.)

A single Sampling Unit is not typically an entire site (Munitions Response Site (MRS)) or an entire study area. Decisions about an entire site often are based on results from one or more Sampling Units within the site. Appropriate boundaries of a Sampling Unit should be dictated by the intended end uses of the data. The importance of delineating Sampling Units that are appropriate and relevant to the end use of the data cannot be overemphasized.

A Sampling Unit is inherently *three dimensional*. Although Sampling Units for surface soil often are discussed only in terms of area, the vertical dimension (sample depth or thickness) should be specified. Regulatory definitions or other project-specific definitions of the vertical extent of surface soil may differ by project or area, but should be explicitly stated in defining Sampling Units.

Ensuring that Sampling Units and Decision Units are delineated clearly, and that they are *relevant to the project objectives and decision criteria*, is critical for obtaining meaningful results and meeting sampling objectives. Sampling Units and Decision Units can be effectively determined only after the project objectives and data end uses of the environmental study or remedial activity are clearly defined and explicitly stated.

7.1 General Considerations for Establishing Sampling Units

Sampling Units should be site-specific and project-specific. The rationale for their delineation should be documented in the project planning process, and explicitly stated in work plans and final reports. Two common rationales for Sampling Units are: 1) a likely contaminant Release Area, or 2) a receptor Exposure Area. Sampling Units designed to

meet one objective, may or may not be appropriate for a different site, different analytes, or for different project objectives or data end uses. For example, Sampling Units in some of the published CRREL range studies were designed primarily to demonstrate the capabilities, requirements, and statistical characteristics of IS methods for characterizing the distribution and total mass loading of energetic compounds on active firing ranges. Those Sampling Units are not necessarily appropriate for direct comparisons of results to regulatory or risk-based screening criteria.

An individual Sampling Unit should encompass an area having similar characteristics throughout. The area should include only a single component of the Conceptual Site Model. In general, Sampling Units to investigate a release area should not be laid out in such a way that they unnecessarily combine areas of high and low levels of contamination (USEPA, 1996b, Section 4.1.4). For example, if the sampling objective requires characterizing a known or suspected contaminant release, a single Sampling Unit typically should not include areas where no contamination is anticipated. However, if the objective is to determine exposure risk (i.e. if the result is to be compared to risk-based soil screening values) a single Sampling Unit should encompass only areas of equally probable anticipated use by potential receptors (e.g. preferred vs. non-preferred habitat). If naturally occurring background constituents are analytes of interest, Sampling Units should have only a single native soil type. Sampling Units encompassing areas of dissimilar characteristics may contribute to decision error.

Practical limitations and unforeseen field conditions may require modifying the delineation of Sampling Units as defined during planning. For example, areas may have limited or difficult access, or be entirely inaccessible for a variety of reasons such as pavement, buildings, exposed bedrock surface without soil, or denied right-of-entry. Areas that cannot be sampled, but that would otherwise have been part of a Sampling Unit, should be excluded from the final Sampling Unit area. For these excluded areas, decisions may need to be deferred, or agreements reached to apply data from the sampled areas. Adjustments to the location or shape of the Sampling Unit may be the preferred solution. In general, significantly changing the total area of the Sampling Unit should be avoided if possible. Changes and their rationale should be fully described in reports.

The IS from a single Sampling Unit provides an estimate only of the mean analyte concentration of that Sampling Unit; no information about the spatial distribution of analytes within the Sampling Unit is obtained. If information about the spatial distribution of analytes within a Sampling Unit is of concern, and IS protocol is to be used, the Sampling Unit must be divided into multiple smaller Sampling Units, each having a sufficient number of increments, prior to sampling.

Sampling Units can be determined using Visual Sampling Plan. Visual Sampling Plan is a software tool developed by Pacific Northwest National Laboratory to assist in determining a defensible sampling strategy for discrete samples based on statistical sampling theory. It is available at http://vsp.pnl.gov. The developers have recently added a capability to address IS (multi-increment sampling). Their application includes an alternative approach of using multiple “Multi-Increment Samples” samples, but using

only 4-6 increments for each Sampling Unit. At this time, sufficient studies may not be available to support the use of this technique on MMRP sites. If additional studies documenting these alternative assumptions are conducted successfully, this restriction will be revisited in the future. Therefore, the use of VSP is allowed on MMRP sites, but only as far as the assumptions follow the traditional IS assumptions, i.e., each sample (Sampling Unit) must have at least 30 increments. VSP may be useful for sites with complicated features, i.e., sites with inaccessible areas that must be excluded from sampling, leaving Sampling Units where increment locations are chosen by the software.

7.1.1 Sampling Unit Size

The size of Sampling Units is critical to meeting project objectives. It must be small enough to detect contaminants at a level of concern, and it must be large enough to be practical and cost-effective. Sampling Unit size can be scaled to achieve the spatial resolution required to meet the identified project DQOs (USEPA, 2006a, Section 4.2). The size of a Sampling Unit must be matched to the intended end use of the data. Typical Sampling Units could be as small as 1 m x 1 m or as large as 100 m x 100 m.

To ensure that a contaminant will not be "missed" or "diluted out", it is essential to specify early in the planning process not only the action level concentration, but also the size of the Sampling Unit. Where a contaminant is non-uniformly distributed (i.e. where there is distributional heterogeneity), the mean concentration will depend on the size of the Sampling Unit. Changing the size of the Sampling Unit will change the ratio of contaminant mass relative to the mass of non-contaminant material represented by the sample.

A Sampling Unit that is inappropriately large for a stated objective may under-represent contamination that would be of concern in an appropriately sized Sampling Unit. For example, small areas having marginally detectable analyte concentrations could average out to below detection levels in a larger Sampling Unit. Conversely, delimiting multiple Sampling Units smaller than necessary to meet project sampling objectives (i.e. analyzing more samples than necessary) could increase cost without substantively increasing the quality of the decision or significantly impacting conclusions from the study. The appropriate size Sampling Unit should be just small enough that distributional heterogeneity within the unit is not of concern for project objectives. The smallest area that will be of concern should be identified.

Multiple Sampling Units within a Decision Unit ideally should be of the same size. If results from multiple Sampling Units will be directly compared to the same screening values, or to each other, they should be comparable in area, and in thickness or depth. The significance of IS results should be evaluated in the context of the area/volume of soil represented by the samples. The mean concentration from a Sampling Unit of a given size may not be directly comparable to mean concentrations from Sampling Units of different size with respect to contaminant mass.

In any case, site-specific work plans should state the rationale for the size of the Sampling Units and how the mean concentrations of the Sampling Units relate to the criteria that will be used to evaluate the results. IS Sampling Units of up to 100 m x 100 m (10,000 square meters or 2.47 acres) have been documented in the CRREL studies on active firing ranges (e.g. CRREL, 2004b), and may be appropriate for identifying potential contamination at levels of concern at some sites. The effectiveness of Sampling Units larger than this for characterizing MC releases is not documented. The sampling design team must weigh the cost efficiency of large Sampling Units with the risk of underestimating (i.e. diluting) or missing contamination that may be present at a level of concern.

7.1.2 Sampling Unit Shape

Sampling Units can be of any shape that will best meet sampling objectives – rectangular, circular, concentric rings, radial wedges from a release point, irregular shapes, etc. In some instances, a Sampling Unit circumscribing a feature (e.g. a building) may be appropriate. Properly designed and executed, the IS methodology will provide a reliable estimate of mean concentration for each Sampling Unit regardless of its size or shape. The only requirement for Sampling Unit geometry is that the mean concentrations in the area(s) represented by the sample be relevant to satisfying the sampling objectives. Where practical, a rectangle is recommended for ease of planning and execution.

A Sampling Unit should define a single, contiguous area. Combining multiple non-contiguous areas into a single Sampling Unit should be avoided. The result will not be representative of any single area, and the relevance of the mean analyte concentrations to project objectives is likely to be ambiguous, at best. Such a sampling approach lacks the structure and unbiased approach required for IS.

7.1.3 The Vertical Dimension of Sampling Units

Although sometimes not explicitly stated, all Sampling Units delineate a volume of soil and thus have a vertical dimension. For surface soil investigations, the vertical dimension may be no more than a few inches, but the thickness or depth interval of a Sampling Unit, and the rationale for it, should be documented during project planning.

The depth of surface soil can be defined differently by different regulatory entities, or for different sampling objectives. For human health risk assessment, surface soil can be defined as the top 6", top 12", or sometimes the top 24". It can be even deeper for some ecological receptors or exposure scenarios.

At firing points and most impact areas, residues from energetic compounds initially are deposited on the surface, including on surface vegetation. At active ranges, significant concentrations of explosive compounds generally are not found below the top few inches of soil. Sampling deeper into the soil profile may result in smaller measured concentrations of the surface contamination. Appendix A of EPA Method 8330B recommends including surface vegetation and plant matter in the sample increments from

active ranges. However, vegetation should be included only if necessary to satisfy DQOs, for example if the study is trying to determine the total amount of a contaminant deposited by airfall onto a recently used range. If vegetation is included, it remains with the sample until it is sieved. During this step the vegetation should be broken in smaller pieces to release trapped particles. The majority of vegetation does not pass through the sieve, and therefore is not part of the sub-sample extracted for analysis.

At Formerly Used Defense Sites (FUDS) or other sites where surface vegetation clearly post-dates any contaminant release, intentionally including vegetation and surface plant matter is not recommended. If vegetation is incorporated, it can be removed during laboratory processing. At FUDS, contamination may have migrated deeper into the soil, or been buried or transported by post-release processes. These factors should be considered, and described when delineating Sampling Units at FUDS.

At firing ranges, MC from projectiles may be embedded in target backstops or berms. At demolition ranges (OB/OD areas), pits or trenches may have been excavated to partially contain the detonations. At some ranges (e.g. hand grenade range) craters may have formed. At such areas, pits and craters may be filled periodically as part of site maintenance activities or by subsequent range use. Under certain site conditions contaminants susceptible to migration by infiltration, may have been carried into the subsurface. These situations will influence the appropriate sampling depths, and require additional consideration in designing Sampling Units. These types of conditions should be explicitly incorporated into the CSM and considered when defining the project objectives.

The IS approach that has been used to characterize subsurface soils is to sample the 3-dimensional volume of soil from discrete layers (horizontal integration) by collecting increments from uniform depths or depth intervals at a large number of unbiased soil boring locations within a surface Sampling Unit area. The number of borings is the number of increments for each IS sample; the number of sampled depths is the number of incremental samples.

Collecting IS samples from depth intervals greater than six inches to a foot, depending on soil characteristics, can significantly increase the difficulty of IS sampling, but may be necessary to meet some sampling objectives. Note that hand held analog magnetometers typically provide UXO safety clearances/anomaly avoidance to maximum depths of 30 to 45 cm (12 to 18 inches). Sampling at depths greater than 6 inches should be avoided unless necessary to meet project objectives, and the increment locations have been cleared for sampling by an EOD or UXO technician.

7.2 Sampling Units to Determine Contaminant Release or Nature / Extent

Project objectives may simply be to determine whether a contaminant release has occurred, or a more detailed investigation of the nature and extent of a contaminant

release, depending on the CERCLA phase of an investigation (e.g. SI or RI). The Sampling Units and Decision Units needed to perform a preliminary site screening assessment (PA or SI) will differ significantly from the data needed to fully characterize the nature and extent of a release (RI). The design of Decision Units and Sampling Units will depend on whether a statistical confidence level is required to support project decisions, or if a decision based on professional judgment will suffice (i.e., probabilistic sampling or authoritative/biased sampling).

Past use of a study area (e.g. a Munitions Response Site (MRS)) is essential in development of the site CSM. Information about likely mechanisms of contaminant release is crucial in delineating appropriate Sampling Units. Development of the CSM and IS Sampling Units should consider the site layout. For a former or operational range, separate Sampling Units might be defined for the firing point, the target area, the range floor, and the range fan area. Researchers at CRREL have identified energetic contaminant release patterns and sampling protocols for a number of common range types. Specific examples are presented by CRREL (2005; 2007). If energetic compounds are of concern, this information can be used to help determine the original patterns of their release. However, other types of analytes (e.g. metals) may have different release mechanisms or fate and transport characteristics that would need to be considered for determining their likely spatial distribution.

Sampling Units may be designed to coincide with known or suspected release areas or visibly contaminated areas identified by the field team. These could include features not identifiable during planning, such as a bomb crater, dump site, stained soil, ruptured munitions, etc. Because areas of obvious release can be expected to have contaminant concentrations significantly different from other areas of a site, it may be most useful to sample them as separate Sampling Units. However, it is important to note that whether a mean contaminant concentration result poses unacceptable exposure risk depends on the size of the Sampling Unit.

A Sampling Unit around a ruptured munition should encompass the area of any visible residue chunks and any surface discolorations. When chunks are present, they should be removed by EOD personnel or UXO technicians, so they are not inadvertently incorporated into the sample. To prevent cross contamination, samples collected where chunk residues were present should be segregated from other samples during transportation, storage, and laboratory processing (USEPA, 2006b, page A-13)

When ordnance disposal (blow-in-place) coincides with site characterization activities, pre- and post-detonation samples should be collected (USACE, 2007, EM 1110-1-4009). This is more likely at the RI stage during intrusive operations, and during removal/remedial actions. The objective of this sampling activity is to establish if residual MC is pre-existing or due to the blow-in-place operation, or both (Pennington and others, 2008). Safety considerations may require excluding the immediate vicinity of the ordnance from the Sampling Unit.

Although past use of an area, or visible indications, can suggest how extensive a contaminant release is, or might have been, they should not be the sole factor for determining the size and geometry of a Sampling Unit. Other project objectives may govern the size and geometry, rather than the spatial extent of a release. Note that post-release processes or activities (erosion, chemical degradation, re-grading) during the time elapsed since any release, may play a significant role in redistributing or covering any released contaminants. Once energetic residue particles have completely dissolved it is unlikely that the compounds will remain detectable in surface soils (CRREL, 2007). However, the dissolution of energetic materials can take decades.

7.2.1 SI Example - Former Air Force Practice Bombing Range

An example objective of determining presence or absence of a release for a Site Inspection investigation of a bombing range illustrates the points made above (see Figure 7-1). If the entire bombing range (e.g. 640 acres) is the area of possible concern, a Sampling unit encompassing the entire range could conceivably be selected. However, for reasons explained in Section 7.1.1, above, the mean concentration over such a large area is unlikely to be meaningful or relevant for direct comparison to typical screening values. Because a Sampling Unit encompassing an entire bombing range would contain very large areas unaffected by any release, mean analyte concentrations would be expected to approximate background concentrations. The likelihood of detecting an analyte above a risk-based concentration limit (or in the case of explosive compounds, above laboratory detection limit) would be very low, even if some smaller areas within the range contained contamination at a level of concern relative to the screening criteria. However, one possible advantage of defining a single Sampling Unit encompassing the entire range (if the entire range is accessible) would be the extensive reconnaissance required for collection of the increments and the increased likelihood of discovering features (e.g. residue from low-order detonations) that could then be the focus of additional sampling. The selection of default residential risk-based SSLs for screening level evaluation of results, as is commonly done, implies that the exposure unit for which the values are intended (e.g. 0.5 acres or less) may be the unit of area with which we should be concerned.

For most projects, cost constraints would preclude sampling the entire bombing range at a scale of half-acre or smaller Sampling Units. However, to reliably determine if contamination is present at the level of concern implied by a residential risk-based screening criterion, we would need to use Sampling Units at this scale. For a general assessment of whether contaminants might be present at some level of concern, Sampling Units larger than 100 m x 100 m (2.48 acres) are discouraged. This is the largest Sampling Unit documented in CRREL research. Larger Sampling Units increase the chance of missing, or diluting beyond recognition, contaminants that might be present at a level of concern in only a portion of the Sampling Unit.

For an objective of determining presence/absence at a level of concern using this example, ideally sized Sampling Units for comparison to typical residential risk-based values for direct exposure would be areas of 0.25 to 0.5 acres, centered on areas of most

likely contaminant release, such as target zones, impact areas, or features identified by the field team. However, in the example in Figure 7-1, larger Sampling Units of about 2.5 acres were determined to be acceptable to meet project objectives because they would provide a higher level of confidence than a few discrete samples that significant contamination within the unit was not missed. This sampling is representative only of the Sampling Unit areas and cannot be assumed to be representative of areas of the range that were not sampled. However, if the results from the areas most likely to have contamination show no evidence of a release, it may be reasonable to conclude, using professional judgment, that remaining areas of the range that were not sampled are very unlikely to have any contamination.

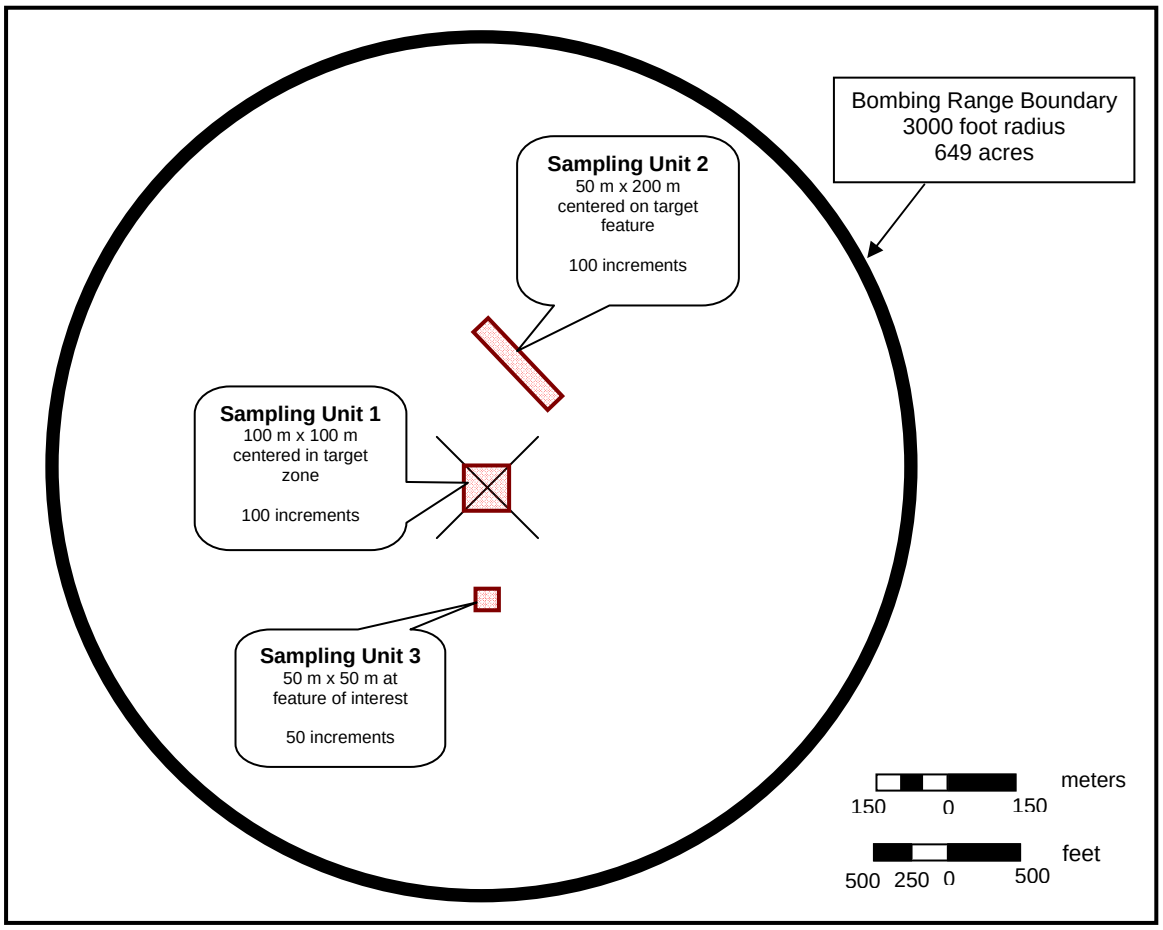


Figure 7-1 - Possible Sampling Unit Geometry for a Bombing Range SI
to evaluate for presence of a contaminant release. Sampling Unit 1 and Sampling Unit 2 each encompass about 10,000 square meters (2.48 acres), to be represented by 100 increments.

7.2.2 RI Example -

7.2.2.1 Use of Large Sampling Units

For an RI, the data need to adequately characterize the nature and extent of contaminants and provide a basis for response action. It may be appropriate to use a phased approach

starting with Sampling Units larger than those that would be needed for a definitive risk-based decision or remedial design, prior to committing funds for a full-scale, detailed investigation. This approach is described in the Hawaii Department of Health technical guidance as "investigating at the neighborhood scale" (HDOH, 2008, Section 3.4.2.1).

The approach is to use relatively large Sampling Units encompassing several acres to several tens of acres. Whether the mean concentration of an analyte within a large Sampling Unit reflects contamination at a level of concern depends not only on the total mass of contaminant, but also on its distribution within the Sampling Unit. Contamination at a level of concern in a large Sampling Unit will be recognized only if it is at a sufficiently high level over a sufficient portion of the Sampling Unit. If a very high concentration area is represented by enough increments of the overall sample, it may elevate the mean sufficiently to recognize that contamination is present somewhere in the Sampling Unit. Likewise, contamination at a lower level, but present over a large portion of the Sampling Unit might be detected.

Appropriate design of large Sampling Units depends to a very large degree on likely contaminant loading and distribution as inferred from the CSM. For example, a skeet range that was used for many years may have high levels of contaminants in the shotfall and target debris zones distributed over 3 to 5 acres. An elevated lead level from such a skeet range probably would be readily detected by IS in a Sampling Unit of 15 to 20 acres. However, if an equal mass of contaminant was released over only a quarter acre, the contamination might not be evident in the mean concentration of a 15 to 20 acre Sampling Unit. The contaminated portion of the large Sampling Unit might be represented by only a single increment of the IS sample, or such a relatively small release area might be missed entirely.

It is important to bear in mind that IS concentrations from large Sampling Units are unlikely to be appropriate for direct, 1:1 quantitative comparison with many regulatory decision limits or action levels. Also, large Sampling Units may contain areas having mean contaminant concentrations that exceed a level of concern at a scale relevant to the regulatory criteria, but which may not be recognized because of a large volume of uncontaminated soil within the Sampling Unit.

Although of limited use for most regulatory determinations, results from Sampling Units of several acres or more may help narrow the focus of more detailed follow-up phases of investigation, or depending on project objectives, may suffice to assess an area for contaminant presence, or other purposes. The thorough reconnaissance of the large areas required by the sampling team also helps ensure that smaller features that should be the focus of more detailed sampling (e.g. low order detonations, etc.) are identified.

7.2.2.2 Probabilistic Assessment of Large Areas

For Decision Units so large that they would require an impractical number of Sampling Units for complete coverage at an appropriate and useful scale, (e.g. sampling every 0.5 acre area for comparison to residential risk-based screening values), an alternative is to sample a selected number of appropriate size Sampling Units from the large Decision

Unit. The large Decision Unit should be sampled in an unbiased manner using IS Sampling Units, rather than discrete "grab" samples as the "increments". All Sampling Units should be of a uniform size that is relevant to the end use of the data, but the Decision Unit can be very large.

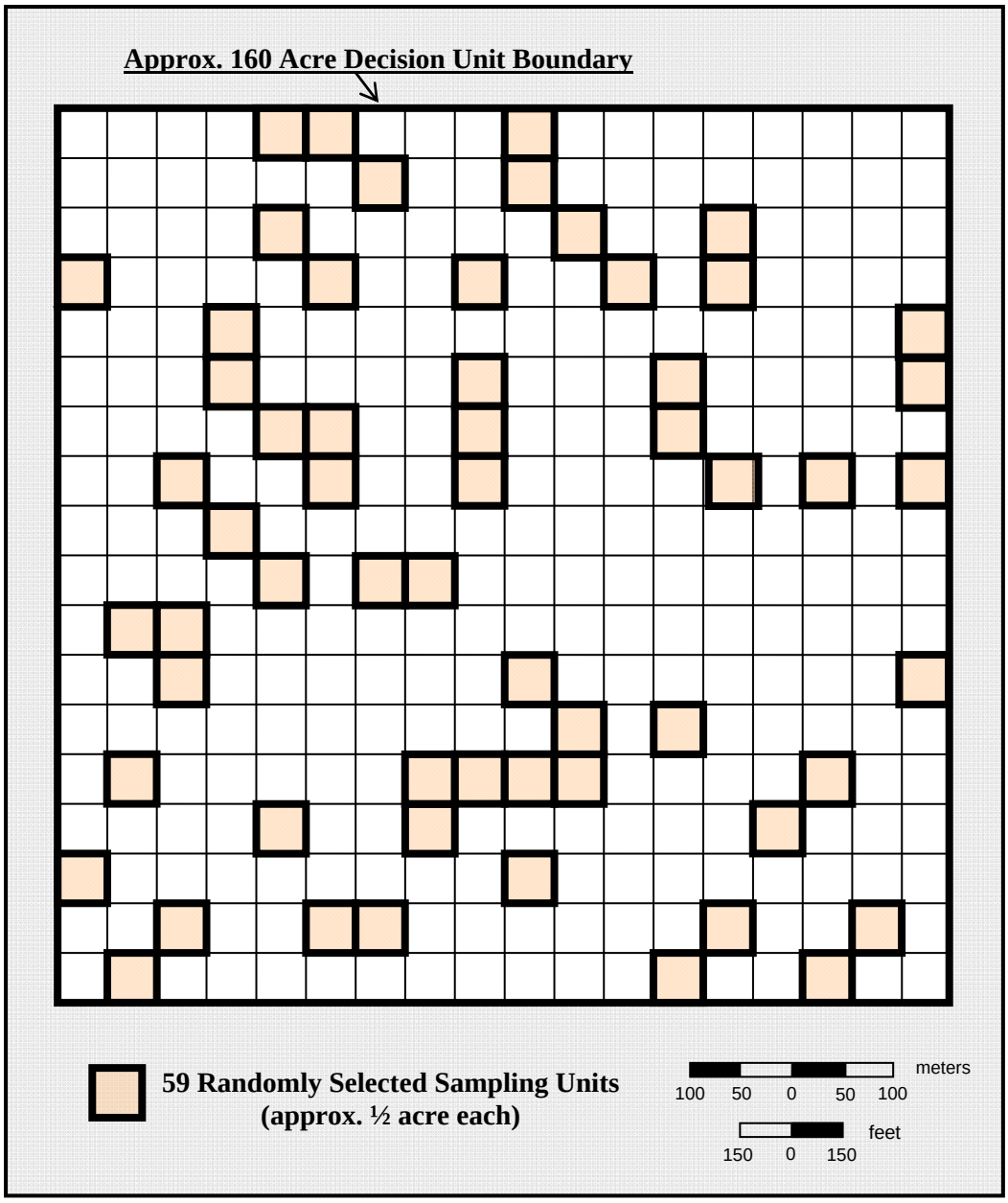


Figure 7-2 – Example of Probabilistic Sampling of a Large Decision Unit

In the example shown in Figure 7-2, if all 59 of the randomly located, half acre Sampling Units within the 160 acre Decision Unit have mean analyte concentrations less than an action level, there is 95% confidence that at least 95% of all of the half acre areas within the Decision Unit will have mean concentrations less than that level. In contrast, if the entire 160 acre Decision Unit is sampled as a single Sampling Unit, the number of half-

acre areas that may have mean concentrations greater than the action level is completely unknown. Properly designed, this approach can provide a probabilistic evaluation of large areas using Sampling Units of an appropriate size for comparison to regulatory action levels intended for application to residential or other scenarios.

The subset of Sampling Units that actually are sampled can be selected in a systematic grid pattern (e.g. a "checkerboard" pattern), or in a random pattern, as shown. The fundamental requirement is that the entire Decision Unit be represented in a uniform and unbiased manner. Data from the initial Sampling Units can be included in subsequent sampling phases if more detailed investigation is required.

Table 7-1 – Number of Sampling Units Required to Achieve a Specified Confidence Level (Binomial Distribution).

p=0.05	Confidence Levels				
Number of Failures	60%	80%	90%	**95%**	99%
0	18	32	46	**59**	90
1	40	60	77	94	130
2	62	85	106	125	166
3	84	110	133	154	198
4	105	134	159	180	228
5	126	157	184	208	258
6	147	180	209	235	288
7	168	204	234	260	317
8	189	227	258	286	344
9	209	249	282	310	370
10	230	272	306	336	397

Table 7-1 uses the binomial distribution to show the number of Sampling Units that must be sampled to achieve specified confidence levels that all Sampling Units of a large Decision Unit will be below an action level. P = 0.05 indicates that, for this table, the portion of Sampling Units that could have a mean concentration greater than the action level (i.e. the "defective" portion), may be up to five percent.

For example, if 59 Sampling Units are sampled and all have mean concentrations less than the action level (Number of Failures = 0), there is 95% confidence that 95% or more of all the Sampling Units of that Decision Unit have mean concentrations less than the action level (USEPA, 1989a, Table 7.1 and related sections; USEPA, 1999, Table 5). If

90 Sampling Units are sampled and all are "clean", there is 99% confidence that the remaining unsampled Sampling Units are "clean". If fewer than 59 unbiased Sampling Units are sampled, the sampling plan is inadequate to infer with 95% confidence that 95% of all Sampling Units of the Decision Unit will be below the action level. Binomial confidence level tables for different probabilities and confidences can be downloaded from http://src.alionscience.com/toolbox/src_oneshot.html.)

The values in Table 7-1 may be regarded as conservative regardless of the total number of equal size Sampling Units (sampled plus unsampled) within the Decision Unit. The binomial distribution used to generate Table 7-1 assumes that there is an infinite total number of Sampling Units within the Decision Unit. The number of Sampling Units required to be sampled to achieve a specified level of probability and confidence is independent of the total number of Sampling Units.

If a significant portion of the total number of Sampling Units is to be sampled, the number required to achieve specified probabilities and confidence can be more accurately calculated by using the hypergeometric distribution. The hypergeometric distribution takes into consideration that the total number of Sampling Units within a Decision Unit is finite, and corrects the proportion of those actually required to be sampled, relative to those unsampled.

For example, applying the hypergeometric distribution to the example in Figure 7-2, which has a total of 324 half-acre Sampling Units, shows that only 54 Sampling Units actually need to be sampled, as long as none exceed the action level, to demonstrate that there is 95% confidence that 95% or more of the unsampled units do not exceed the action level. As the total number of Sampling Units in the Decision Unit decreases and approaches the number that actually will be sampled, the values from the binomial distribution shown in Table 7-1 become increasingly more conservative than indicated.

Visual Sampling Plan (VSP) provides a hypergeometric calculator that can be used to calculate the number of "clean" samples needed to calculate the probability that a Decision Unit is clean at a specified level of confidence. Hypergeometric calculators also are available on the internet. Reports and work plans should state which type of distribution was used for such calculations.

One important point that neither the hypergeometric distribution nor the binomial distribution takes into account is the uncertainty associated with determining whether an individual Sampling Unit exceeds an action level. Both assume that the determination of "clean" or "dirty" is made with 100% confidence. This is not necessarily the case. If the IS estimate of the mean concentration of a Sampling Unit is very close to the action level, there may be considerable uncertainty. If replicate IS samples were collected from the Sampling Unit, comparison of the 95% UCL of the mean to the action level would provide 95% (not 100%) confidence of a correct decision for that particular unit. Having even a single exceedance of the action level significantly lowers the confidence that the unsampled units will have the specified probability of being below the action level.

If one or more of the Sampling Units has a mean concentration that exceeds a specified action level, the significance of the exceedance(s) should be evaluated. Depending on project objectives, additional sampling, and/or possibly remediation, may be required. This is analogous to the situation where a discrete sample exceeds an action level. A course of action contingent upon such a possibility should be identified in the work plan.

7.3 Sampling Units based on Current or Anticipated Future Land Use

If the future use of a site is known, Sampling Units should be designed to provide information relevant to that future use.

7.3.1 Sampling Units for Comparison to Risk-Based SSLs

Risk-based soil screening values are based on average or upper percentile exposure factors and durations. They are optimally applied across a specific area - the Exposure Unit. A receptor is assumed to be exposed equally to all parts of an exposure unit. The mean soil contaminant concentration across the exposure unit best represents the potential exposure of a receptor to site contaminants over a long period of time.

> ***"The most appropriate Decision Unit for evaluation of direct-exposure to soil contaminants having toxicity-based hazards is the assumed exposure area." (Hawaii Department of Health, 2008).***

Comparison of results to a risk-based SSL implies that the smallest area that should be of concern is the area of the Exposure Unit to which the SSL is intended to be applied. Many times default risk-based values used for site screening are based on residential exposures. In these cases the area of exposure (i.e. the Exposure Unit) is assumed to be a suburban residential lot size, typically about one quarter to one half acre (USEPA, 1996a; USEPA, 1996b, Section 4.1.4; USEPA, 2009). Exposure Units for industrial based screening levels may be intended to be applied to somewhat larger areas. Trade-offs should be considered before comparing the mean concentration of a Sampling Unit larger than the Exposure Unit to risk-based SSLs. If the Sampling Unit size is significantly larger than the exposure unit that forms the basis of the risk-based value (e.g. 0.5 acre), there may be significant areas (i.e. exposure unit size) within the Sampling Unit that have mean concentrations greater than the screening value, but which may not be recognized because large areas of uncontaminated soil also would be represented in the sample. If the IS results are to be compared directly to SSLs for direct exposure, Sampling Units should be about the same size as the exposure unit upon which the SSL is based (USEPA, 1996b).

7.3.2 Sampling Units to Assess Human Health Risk

For conventional screening level assessments (SI stage), the maximum detected concentration (MDC) of a given chemical in an area often has been used for risk screening. This is a very conservative screening approach used to limit cost at this early

project phase, and to decrease the likelihood of underestimating the significance of releases. Such a conservative approach may be unnecessary if IS is used. The use of IS, properly applied, can provide data that are more representative of realistic exposure concentrations by providing unbiased and reproducible estimates of average concentrations over the exposure area, using relatively few samples and within a project budget appropriate for a typical SI.

For baseline risk assessments at the RI stage, it is necessary to define nature and extent of contamination, and to establish exposure point concentrations (EPCs). Per USEPA (1989b) the EPCs should be based on a 95% UCL, to ensure that the mean is not underestimated. Average concentration is most representative of the concentration that would be contacted at a site, over time (USEPA, 1992a). IS gives a confident estimate of the true mean concentration, and may be substituted for a 95% UCL. If, however, project requirements dictate that a 95% UCL must be generated, this may be done using replicate IS samples. A minimum of three to five replicate IS samples from a Sampling Unit can reduce and quantify the uncertainty of the estimated mean concentration.

In an RI, where Sampling Units will provide the basis for exposure point concentrations for a baseline risk assessment, the cost of such sampling effort would need to be balanced with other project factors and appropriately documented in project planning documents. Depending on specific project factors, not all portions of a Decision Unit would necessarily need to be sampled. For example, a "checkerboard approach" could be used, and statistically based inference made regarding the unsampled areas of the Decision Unit (see Section 4.2.2.1). The "acceptable level of effort" must be determined in the systematic planning process. Implications and uncertainty of sampling only a portion of a Decision Unit, and the size of Sampling Units upon which a decision will be based, should be identified and explained in project documents.

7.3.3 Sampling Units to Assess Ecological Risk

As noted above relative to assessing human health risks, the ecological risk assessment conventionally follows the same pattern. At the SI stage, the screening-level ecological risk assessment (SLERA) compares the MDC to conservative ecological screening levels to insure that the potential for risks is not underestimated. As long as the on-site samples are biased to areas most likely to contain the highest concentrations of contaminants, IS should provide adequate assurance that conservatism has been maintained, and use of the MDC may not be required.

For the RI, establishment of nature and extent and calculation of appropriate EPCs for ecological receptors follows the same procedures as for human health assessments. The benefits of IS and the ability to calculate a 95% UCL applies equally to the baseline ecological risk assessment (BERA) as it does to human health. Typically, however, the size and depth of the sampling units required will be different, sometimes significantly so.

The BERA will establish assessment endpoints (valued resources that require protection), which will help determine appropriate Sampling Unit sizes (US Army Biological Technical Assistance Group, 2002). Home range size, feeding and nesting patterns, and burrowing activities should all be considered in establishing appropriate Sampling Units to assess ecological risk. Note that not all ecological entities necessarily require protection and, therefore, the smallest home range of all organisms that could be present may not necessarily be the proper focus. Sampling Units for ecological risk assessment may need to focus on areas of preferred habitat within a site, and those receptors that require and utilize that habitat. Management goal establishment will dictate what areas need to be sampled, and which do not (US Army Biological Technical Assistance Group, 2005).

7.4 Sampling Units for Comparison to SSLs Protective of Groundwater

SSLs developed for protection of groundwater from contaminants leaching from soil are model-based mean contaminant concentrations. Among the required model input parameters, is a defined source area or volume of soil. Soil concentrations deemed protective of groundwater may be site-specific, or default values such as those published in the EPA Regional Screening Level tables and many state regulatory agencies. The models assume that contamination is uniformly distributed throughout the source area (i.e. a mean concentration). A default 0.5 acre source area is used to calculate the generic EPA SSLs protective of groundwater (US EPA, 1996b, Section 1.3.4). The source areas upon which different default screening levels are based typically range from an area of half an acre (e.g. the default EPA Regional Screening Levels (USEPA, 2009) and Dilution Attenuation Factor (DAF)) to as much as 30 acres or more (e.g. Texas Tier 1 Residential Soil to Groundwater Protective Concentration Level (PCL)). If IS results are to be compared directly to SSLs for evaluation of threat of leaching to groundwater, the Sampling Unit size should consider the source area/volume of soil upon which the SSL mean concentration value is based. In the absence of other sound rationale, a Sampling Unit size of half an acre would not be unreasonable.

7.5 Units to Determine Background Concentrations

In some locations, background concentrations of metals or other naturally occurring or anthropogenic analytes may exceed risk-based screening levels or regulatory limits used for response action decisions. Mean background concentrations are required if analytes of interest include naturally occurring or anthropogenic analytes unrelated to the potential releases being investigated (e.g. metals, perchlorate, PAH compounds). IS is well suited to determine accurate, site-specific mean background or ambient concentrations. Published regional background data are not intended to reflect, nor can they always be confidently assumed to be representative of, specific locations or sites within a region. Most published regional background studies (e.g. USGS) include a statement to this effect.

Site specific mean background concentration data set a standard against which all other site data will be compared. Unlike regulatory or risk-based concentration criteria which are very clearly and precisely defined by a single number (for comparative purposes they are infinitely precise), empirical estimates of site specific mean background concentrations have some degree of uncertainty. Comparison of means may be performed between IS data sets using standard statistical tests. Comparison of IS means to UTLs of data sets of discrete samples is inappropriate (see Section 2.2). Thus, the uncertainty in estimates of site-specific mean background concentrations, particularly in the RI phase, needs to be as small and well defined as reasonably possible to provide a reasonable degree of confidence in decisions regarding other site data. Large uncertainty in background concentrations due to inadequate sampling (or unknown uncertainty due to a lack of QC replicate samples) needlessly increases uncertainty. Because of the importance and utility of having well defined estimates of site-specific background concentrations, site-specific background Sampling Units are recommended to be sampled at least in triplicate. The rationale for the number and acceptable degree of uncertainty in replicate site-specific background values should be explicitly discussed and documented during the DQO process.

Ideally, Background Units (i.e. the areas sampled to estimate site-specific mean background or ambient concentrations) should be of the same size and have the same increment density as the Sampling Units for results that will be evaluated with respect to the background data. This will ensure the most defensible direct comparisons. However, because background analytes may tend to have a more uniform spatial distribution (i.e. lower distributional heterogeneity) across a site than MC analytes released by past site activities, it may be possible that sufficiently accurate estimates of the mean background concentrations could be obtained from areas smaller than those used for the field samples. Nonetheless, Background Units should be large enough to ensure that the natural variability of soil composition across the site or area of interest is captured.

As discussed previously (Section 7.1.1, Sampling Unit Size), if the background concentration itself is to be evaluated against, or understood in the context of a regulatory standard or risk-based screening value, the Background Units should be of the same size as areas upon which those values are based. Whether smaller areas should be sampled to obtain estimates of site-specific mean background should be discussed during project planning, and any rationale to do so clearly documented.

Because background analytes may tend to have a more uniform spatial distribution (i.e. lower distributional heterogeneity) across a site than MC analytes released by past site activities, it may be tempting to reduce the number of increments collected for site-specific incremental background samples. However, reducing the number of increments will increase the uncertainty in the mean background concentration. Because of the importance and potential benefit from having accurate and precise site-specific background concentrations, marginal cost reduction in field sampling effort by reducing the number of increments for Background Units will be negligible relative to potential overall cost benefit to the project by reducing decision error.

Caution should be used if site-specific background determined by IS is being compared to site-specific mean background determined from discrete sampling (see Section 2.2, above). If the IS samples have been ground to reduce particle size for improved precision, it may be possible that the mean from the IS may be elevated relative to a mean calculated from unground discrete samples due to the difference in laboratory processing. Also, using an insufficient number of discrete samples to estimate the background mean, will tend to underestimate the true mean. Both of these considerations will tend to make the IS sample mean appear higher than the discrete sample mean, even though it is likely to be a better estimate of the true population mean.

If there are different soil types, or soils derived from different parent material or processes across a site or study area, background soil samples should be collected from each type. For example, if some portion of a site is composed of dredged material and some portion is composed of native soil, characterizing the two types separately may be necessary. Avoid delineating background sample areas that encompass more than a single soil type.

8 Sample Collection

An unbiased sampling scheme must be developed and carefully followed to uniformly sample the volume of soil within the boundaries of the Sampling Unit. The fundamental requirements for increment collection are:

- an unbiased pattern throughout the entire Sampling Unit
- complete and uniform sampling across the specified depth interval
- uniform size/mass of increments

A sampling methodology is considered unbiased and correct if all of the particles in the Sampling Unit have the same probability of being included in the sample (Gy, 1998). To obtain a sample that is representative of the population in terms of particle type, size, and proportion, the volume of soil in each increment must be constant.

Coring devices that assure a uniform diameter core through the entire sampled interval are preferred for increment collection. Most devices such as a garden trowel or hand auger do not control the amount of material per increment, or ensure representative proportions of material from throughout a specific depth interval. They are likely to introduce bias into the sampling. Although their use may be unavoidable for some material, such tools are not recommended (Pitard, 1993).

A variety of hand operated coring devices designed for surface sampling (e.g. < 6 inches) are widely available from a variety of vendors (e.g. forestry supply catalogs, etc.). "Pogo-stick" type coring devices patterned after prototypes designed by CRREL (Figure 8-1) are available from GPL Laboratories, LLP. Where suitable, cohesive soils are present, a coring device greatly facilitates the rapid collection of uniform, representative increments from a consistent depth interval. For highly compacted or cemented soils, split barrel samplers with a drive shoe can be driven manually using a slide hammer, or used with a direct push drill rig. They may work well for deeper samples. Graduated plunger devices or coring devices such as an Encore sampler will provide a consistent volume for obtaining increments from conventional deeper cores (e.g. split barrel, Shelby tube, etc.). The diameter of the cores should be adjusted to obtain a total, dry weight sample mass of at least 500 grams (preferably 1 kg to 2 kg) from the prescribed number of increments.

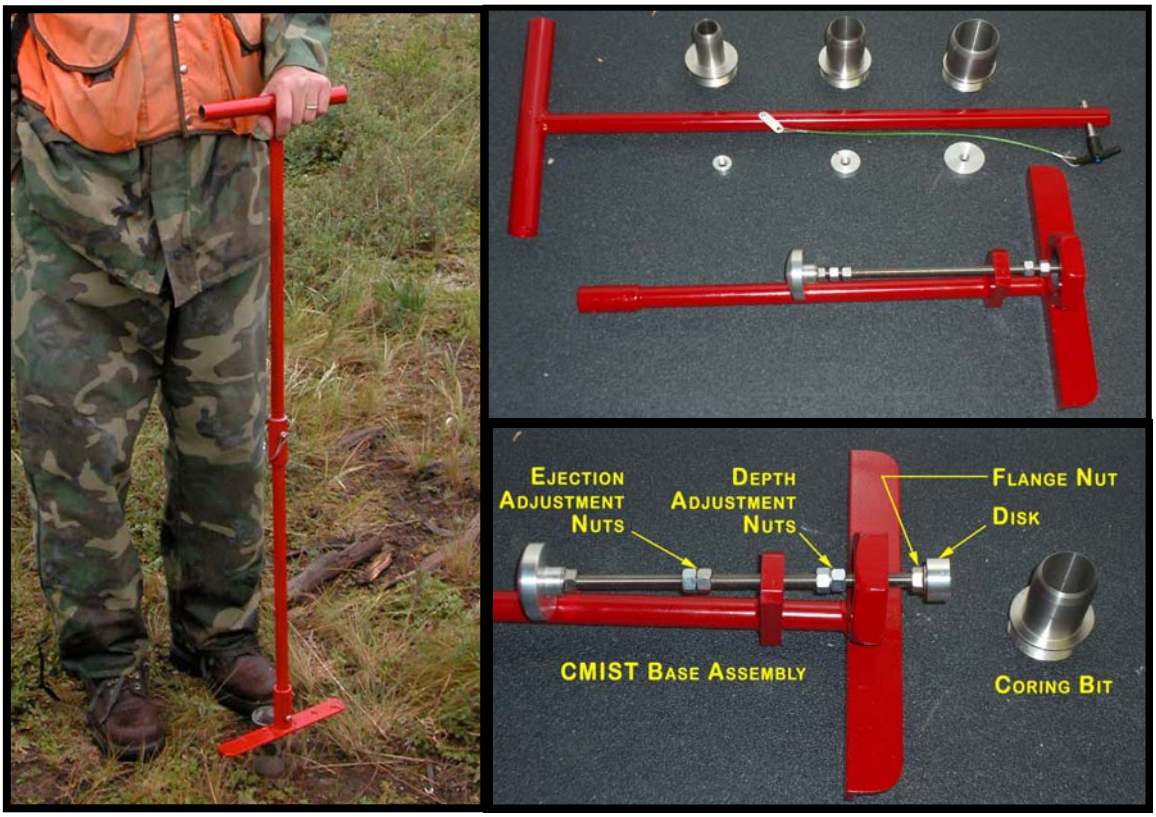


Figure 8-1 - CRREL Coring Device
(CRREL, TN-04-1, 2004a; CRREL, SR-09-1, 2009). Note various size coring shoes. Increment cores from a single Sampling Unit should be of the same size.

Project planning should provide discussion of sample collection and identify contingency actions in case sample collection difficulties are encountered. Taking a wide variety of implements into the field may help ensure the greatest likelihood of successful sample collection.

Field sampling equipment should be decontaminated by standard procedures before moving to a new Sampling Unit. Because individual increments from a single Sampling Unit are combined, it is not necessary to decontaminate sampling equipment while collecting increments from within a single Sampling Unit. Rinseate blanks are unnecessary for soil and sediment analyses because the contamination in these blanks is typically negligible.

The location of Sampling Unit boundaries should be accurately determined and marked by stakes, flagging, or other means of clear visual reference in the field. Use of a Global Positioning System (GPS) for locating boundaries is recommended. It is not necessary to record the locations of individual increments.

8.1 Determining Sample Size and Number of Increments Required

To ensure that the IS sample will not "miss" contamination of concern within the Sampling Unit, collecting a sufficient number of increments is essential. The number of increments required to ensure a representative IS sample, and to meet the required level of reproducibility specified in the DQOs (i.e. data precision), depends on the distributional heterogeneity of analytes within the Sampling Unit. The number of increments required to represent a Sampling Unit is not directly related to the size of the Sampling Unit, but depends only on the degree of the variability within the Sampling Unit. (In statistics, the number of measurements required to characterize a population doesn't depend on the size of the population, but on the variability of the population.) There is an indirect general correlation between size and variability, however, because a larger Sampling Unit potentially encompasses greater variability.

Investigations conducted by CRREL show that the minimum number of increments typically required to achieve good reproducibility (e.g. %RSD <30) between replicates at active firing ranges, where distributional heterogeneity is high, is empirically demonstrated to be 30 to 100 for energetic compounds. Statistical investigations also support this range as a reasonable minimum number of increments for acceptable reproducibility (USEPA, 2003b). Just as increasing the number of discrete samples analyzed from a given area reduces the variability of the estimated mean concentrations of the area, increasing the number of increments for a IS sample reduces the variability of the estimated mean concentrations. However, increasing the number of increments above 100 provides only marginal improvement in precision.

The number of increments must be balanced with the mass of each individual increment to yield a total sample mass to sufficiently average the compositional heterogeneity of particles (Table 5-1). Adequate total sample mass for typical soil-size particles (< 2 mm) is empirically demonstrated to be 1 to 2 kg (based on analyses of explosives).

Table 8-1 – Example Mass-Increment Table for Coring Devices

Corer Diameter (cm)	Desired Total Incremental Sample Mass* (g)				
	500	750	1,000	1,500	2,000
	Number of increments to reach desired total sample mass (g)				
1.00	170	255	340	509	679
1.25	109	163	217	326	435
1.50	75	113	151	226	302
1.75	55	83	111	166	222
2.00	**42**	**64**	**85**	**127**	**170**
2.25	34	50	67	101	134
2.50	27	41	54	81	109
2.75	22	34	45	67	90

3.00	**19**	**28**	**38**	**57**	**75**
3.25	16	24	32	48	64
3.50	14	21	28	42	55
3.75	12	18	24	36	48
4.00	**11**	**16**	**21**	**32**	**42**
4.25	9	14	19	28	38
4.50	8	13	17	25	34
4.75	**8**	**11**	**15**	**23**	**30**
5.00	7	10	14	20	27

* Assumed dry bulk soil density = 1.50 g/cc
* Assumed increment core length = 1 inch

The number of increments per unit area should be the same for Sampling Units that will be compared directly to each other, or to the same decision criteria. This will help assure that the results being compared have the same precision from the different Sampling Units. It also will support application of the precision determined by replicate sampling of one Sampling Unit to similar units that were sampled at the same increment spacing.

8.2 Increment Collection Designs

Several probabilistic sampling designs that may be used for IS are briefly summarized below. Because all have a statistical basis, uncertainty can be quantified by collection of replicate samples. Additional probabilistic sampling schemes are described in the EPA guidance document QA/G-5S (USEPA, 2002b).

8.2.1 Systematic Random Sampling

The most commonly used and most reproducible sampling pattern is the systematic random approach. The key steps for collection of a systematic random sample are:

- Sub-divide the Sampling Unit into uniform grid cells, one per increment.
- Randomly select a single increment sampling point in an initial grid cell.
- Collect increments from the same relative location within each of the other grid cells.

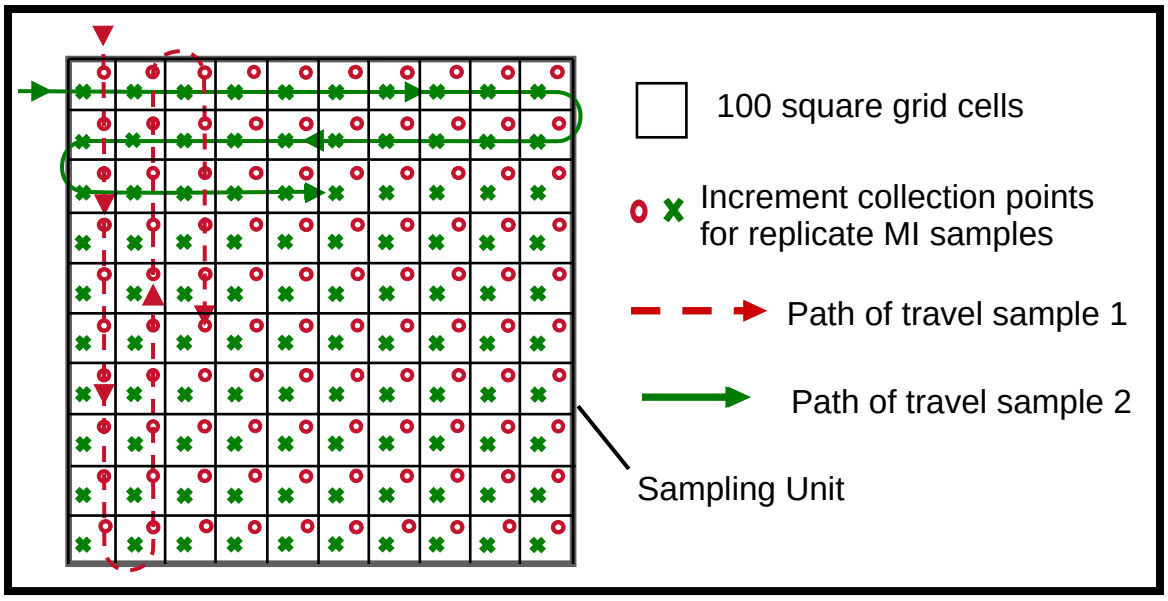


Figure 8-2 - Systematic Random Sampling[3]
for collection of two (replicate) 100-increment samples. Figure after CRREL, 2007.

8.2.2 Stratified Random Sampling

In stratified random sampling, the Sampling Unit is sub-divided by a uniform grid and one increment is collected from a location chosen randomly in each grid cell.

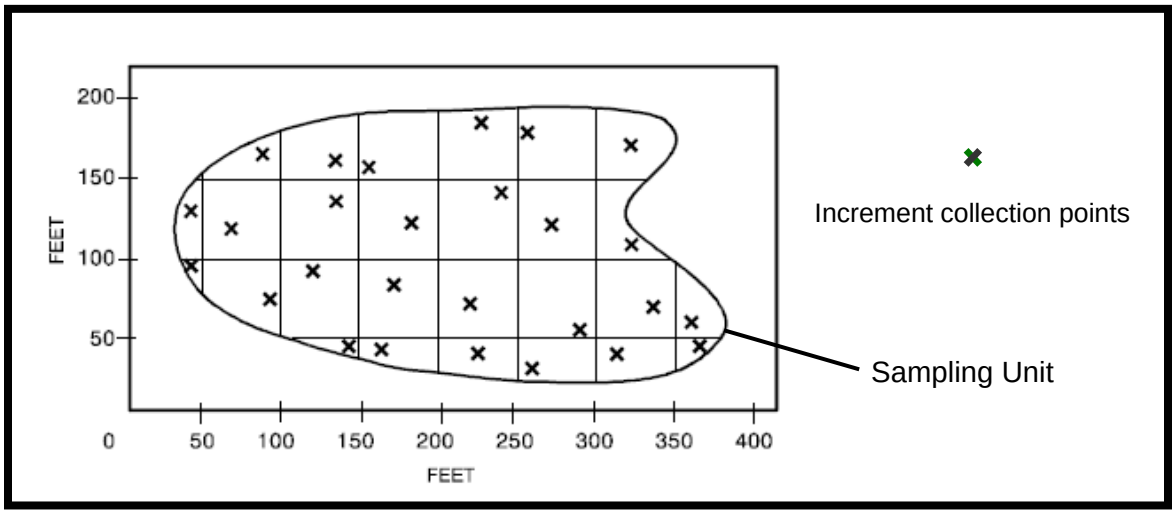


Figure 8-3 – Stratified Random Sampling
Figure after USEPA, 1995 (540/R-95/141, Figure 5). Note that as illustrated, this Sampling Unit encompasses an area of about 1 acre, represented by only 27 increments.

[3] Nomenclature per Pitard, (1993, Figure 21.8); CRREL; and EnviroStat, Inc. There are nomenclatural differences in increment collection schemes between those and USEPA,1995 (540/R-95/141) and USEPA 1989 (EPA/230/02-89/042).

8.2.3 Simple Random Sampling

Purely random sampling provides statistically uniform coverage and meets the requirement that every point within the Sampling Unit has an equal probability of being included in the IS sample. Although statistically valid, simple random sampling tends to require a larger number of increments than other schemes to achieve a specified level of precision. Depending on site specific conditions and objectives, it may be slightly more costly than other statistically based collection schemes. Purely random sampling ignores information that may be available to develop more efficient or cost effective sampling designs. Also, it may be difficult to locate truly random sampling points within the Sampling Unit.

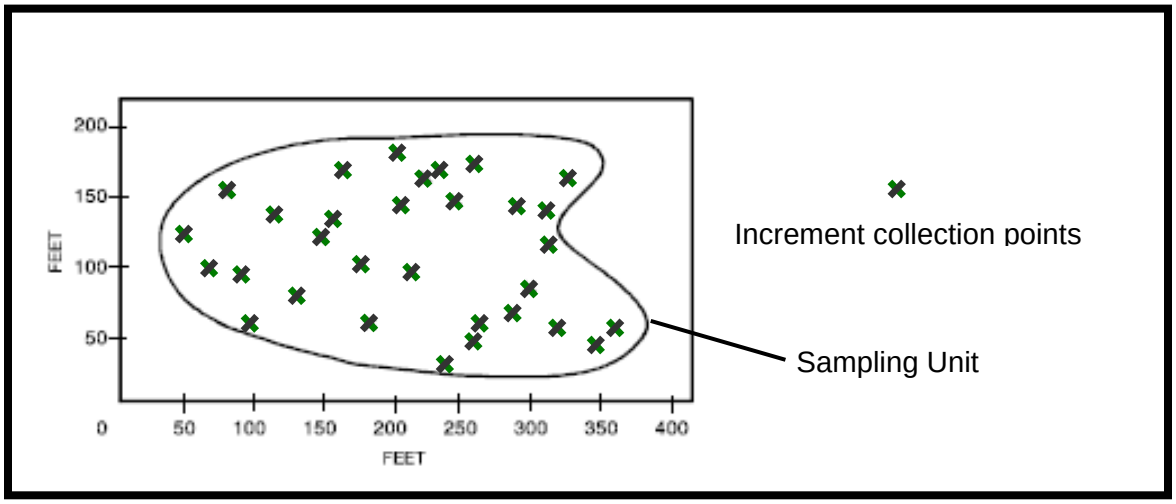


Figure 8-4 – Random Sampling
Figure after USEPA, 1995 (540/R-95/141, Figure 2). As illustrated, this Sampling Unit encompasses about 1 acre, to be represented by 31 increments.

9 Laboratory Considerations

9.1.1 Laboratory Approval

The large field sample mass, laboratory space for drying, suitable grinding equipment, representative sub-sampling procedures, decontamination, and dust control measures are some of the challenges faced by the laboratory in implementing 8330B-compliant or other IS processing. The laboratory's equipment, procedures (including decontamination), and accreditation should be determined to be adequate for project requirements.

To date, the Department of Defense (DoD) has evaluated and approved few laboratories for the sample preparation and processing procedures recommended in Method 8330B for explosive and propellant compounds, or for adequacy of SOPs for IS processing in general. However, increasing demand for IS laboratory processing is resulting in a relatively rapid increase in the number of commercial laboratories that are developing this capability.

Laboratories must demonstrate compliance with the DoD Quality Systems Manual (QSM) through the DoD Environmental Laboratory Accreditation Program (ELAP), and the SOPs should be in accordance with the EPA Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples (USEPA, 2003b). Assessment by the DoD ELAP according to the DoD QSM version 4.1 (Department of Defense, 2009) will cover laboratory procedures for Incremental Sampling for explosives analysis. Laboratories proposed for analysis of parameters other than explosives should be assessed and approved for IS sample processing in accordance with USACE Incremental Sampling ("MIS")-Based Laboratory Requirements for the Analysis of Explosives (Method 8330B) and Metals in Solid Matrices (USACE, 2008b). ASTM D6323 Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities (ASTM, 2003) gives guidance on sample splitting, particle size reduction, and the mass of subsample necessary to reduce the fundamental error to <15%.

9.1.2 Laboratory Sample Processing Considerations

All IS processing should occur under controlled conditions in a laboratory. Splitting, sub-sampling, or other processing in the field should be avoided. It is very difficult, if not impossible, to avoid introducing bias and imprecision using typical field mixing methods.

Method 8330B requires that the entire 1 to 2 kg sample be air dried, then sieved to remove particles larger than 2 mm (i.e. very coarse sand size, #10 sieve). This sieving requirement comes from a definition of soil as being particles < 2 mm. Samples must be thoroughly disaggregated before sieving in the laboratory to ensure that all soil-size particles pass through the sieve. The entire sieved sample is then ground to reduce

particle size further, typically to an average particle size of less than 75 micrometers (very fine sand and smaller). The smaller the particles, the lower the variability between sub-samples from a given mass of sample (USEPA, 1992b). Using the incremental approach, a large number of small aliquots (typically at least 30) are taken in an unbiased manner from the processed sample and composited until a sufficient sample mass is obtained for extraction and analysis. EPA Method 8330B recommends processing 10 grams of soil for solvent extraction and analysis, rather than the 1 to 2 grams typically analyzed by other methods. The recommendation to analyze a 10-gram aliquot should be implemented for all propellant/explosive analyses. The particle size reduction (grinding) was determined to be necessary to improve precision to acceptable levels (e.g. %RSD < 15%) for replicate laboratory sub-samples for some explosive/propellant compounds (Walsh, et al., 2002) (see also USEPA, 2003b).

Grinding to reduce particle size may not be feasible for all situations (see Section 9.1.4.1). Sieving through sieves finer than the 2-mm (#10) recommended by Method 8330B may be another option to reduce particle size and improve precision in laboratory sub-sampling for some analytes or project objectives (see Section 9.1.4.1, below).

9.1.3 Laboratory QA/QC Considerations

Some portion of the IS field samples should be sub-sampled and analyzed in triplicate to demonstrate that laboratory sub-sampling procedures are correct and adequate to control both compositional and distributional heterogeneity. The %RSD for laboratory triplicates typically can be < 15%. However, as with the suggested %RSD of 30% to 35% for results from field replicates (which reflect total error), this suggested maximum RSD of 15% for laboratory reproducibility is a general guide for what can be expected. Whether it is appropriate for a specific data set depends on the precision required to meet DQOs, and how close to a decision limit a concentration result might be. It does not mean that data should be automatically rejected if the %RSD is greater. Because %RSD is a relative measure, it tends to become larger for concentrations near the analytical quantitation limits.

The replicate laboratory analyses should be done using IS samples that are expected to contain contaminant concentrations near the chosen action level or below this value, but above detection. This will provide the most demanding test of reproducibility while minimizing the chance of non-detect results which would be of little value in evaluating data quality.

Laboratory Control Samples (LCS) and MS/MSD samples spiked prior to grinding may be problematic. Spiking a large volume of sample requires a relatively large amount of standard. It is typically not practical to add surrogates or target analyte spikes to 1 – 2-kg samples. However, reference materials for Performance Evaluation (PE) for explosives are now commercially available. See the Environmental Data Quality Working Group white paper Guide for Implementing EPA SW 846 Method 8330B (EDQW, 2008) for further discussion of laboratory QC. The DoD QSM Version 4.1 (DoD, 2009) also has

laboratory QC requirements for analysis of explosives by EPA Method 8330B. Laboratory QC requirements should be discussed with laboratory personnel during project planning.

9.1.4 Analyte-Specific Considerations

Sample collection and laboratory processing protocols may vary depending on the analytes of interest in a particular investigation or Sampling Unit. There are no published procedures for laboratory processing of environmental IS samples for analytes other than energetic compounds. Laboratory procedures of Method 8330B, developed specifically for explosive and propellant compounds, may need to be modified for other analytes. In particular, the grinding process may be unnecessary or even inadvisable for certain analytes and objectives (e.g., drying, sieving, and grinding would be inappropriate for volatile organic compounds).

Depending on the nature of the analytes of interest (e.g. some metals), grinding can introduce a bias into the analytical results, either from loss of analyte from the sample, or addition of spurious contaminants to the sample. However, because grinding increases precision, relatively small biases may be more than outweighed by improvements in precision. Project-specific measurement quality objectives and the potential effects of grinding should be considered and addressed by the laboratory and project team during the planning process.

9.1.4.1 Metals

Developing protocols for laboratory processing of IS samples for metals and other analytes is the subject of current research. Nonetheless, bearing in mind certain potential effects discussed below, a laboratory sub-sample suitable for metals analysis should be attainable.

Preliminary studies on metal concentrations using IS samples of uncontaminated natural soils have shown that grinding greatly improves reproducibility (precision), but consistently imparts a small, but for most purposes negligible, positive bias (increase) in median metals concentrations (Felt and others, 2008). However, the reduction in total measurement uncertainty (error) compensated for the slight positive bias, improving the overall accuracy of the measurements.

Most commercial crushing or grinding equipment has working surfaces composed of metal alloys containing iron, chromium, tungsten (carbide), etc. These grinding surfaces have been demonstrated to contribute metal contamination during sample processing. This spurious contamination may be significant for environmental samples if the metals contained in the grinding equipment are analytes of interest. Grinding devices having agate or ceramic surfaces are available and may need to be considered.

Naturally occurring metals in surface soils are almost always found as crystalline silicate, oxide/hydroxide, or carbonate minerals that are amenable to grinding. Mechanical crushing and grinding procedures have long been used in traditional geochemical studies. However, some MC metals in elemental form (e.g. lead, copper, tin) are malleable and not amenable to grinding. Effects of grinding on soil samples containing malleable metal particles (e.g. projectile fragments in a firing range berm) have not been well studied, but are the subject of current research. Malleable particles tend to smear during grinding, perhaps being lost from the sample to equipment surfaces and biasing sample results low from analyte losses. Decontamination of malleable metals from grinding equipment may be problematic and could result in high bias in subsequent samples from carry over (cross-contamination).

If significant quantities of malleable metal particles are suspected to be present in the soil, grinding may be undesirable. Instead of particle size reduction by grinding, increasing the sub-sample mass for extraction and analysis of the unground sample can improve precision. However, the amount of mass required to achieve a specific precision would be specific to the material being analyzed. Alternatively, dividing a sample prior to processing to apply different processing protocols to the splits may be acceptable if this done in a manner that is consistent with Gy's sampling theory on sub-sampling (e.g., ASTM D6323 -98 (ASTM, 2003) and EPA 600/R-03/027 (USEPA, 2003b). An unbiased split is best achieved using a sectorial (rotary) splitter in the laboratory. Again, because of the difficulties in obtaining unbiased splits, we emphasize that *the field team should not attempt to homogenize and split an IS sample in the field.*

As another example, lead in replicate unground IS samples from small arms firing ranges may have unacceptably large variability (>100% RSD), even after air-drying, sieving through a #10 sieve (< 2 mm) and subsampling using a sectorial splitter. The large variability for lead may be due to single particles of lead between 1 and 2 mm diameter being present in only some of the replicate splits (i.e. the compositional heterogeneity has not been adequately addressed). However, if the end use of the data is to assess risk of incidental ingestion of lead, precision for the concentration of lead contained in larger particles may be of less interest than lead contained in the finer (< 0.25 mm) fraction (USEPA, 2000; USEPA, 2003a; ITRC, 2003). Using a finer mesh sieve (e.g. 0.25 mm rather than 2 mm) prior to subsampling may improve precision and reproducibility that will meet data quality requirements without grinding. Material retained on the sieve may be reserved for possible analysis. It is important to note, however that sieving an unground sample through sieves finer than 2 mm is not an appropriate protocol for high explosives or for propellants. Much of the mass of the energetic analytes is in particles greater than 0.59 mm (30-mesh sieve) (Walsh and others, 2007).

9.1.4.2 PAHs, Perchlorate, White Phosphorous

There are no published procedures specific to the laboratory processing of environmental IS samples for Polycyclic Aromatic Hydrocarbons (PAHs). Collection and laboratory processing of samples per EPA Method 8330B is recommended. Methods SW3540 and

SW3550 require extraction of 10 grams and 30 grams of soil, respectively, so increasing the analytical method aliquot size is unnecessary.

There are no published procedures specifically for laboratory processing of environmental IS samples for perchlorate. Collection and laboratory processing of samples per EPA Method 8330B is recommended. Methods 6850 and 6860 require extraction of 1 gram of soil in 10 mL of reagent water; however, the recommendation in Method 8330B to analyze a minimum 10-gram aliquot for all propellant and explosive analyses should be implemented. IS sampling has been conducted successfully at Hill AFB to investigate perchlorate concentrations.

There are no published procedures specifically for laboratory processing of environmental IS samples for white phosphorus. Due to the nature of white phosphorus, according to SW7580, the laboratory should carefully homogenize the soil/sediment sample in its original container but not dry it or grind it prior to extraction. SW7580 requires extraction of 40 grams of wet soil. IS sampling has been conducted successfully by the USACE at Eagle River Flats to document white phosphorus concentrations reproducibly (e.g. Walsh and others, 1997).

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