

ENCYCLOPEDIA OF
ENVIRONMENTAL
ANALYSIS
AND
REMEDIATION

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Editor

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ISBN 0-471-11708-0

SOIL SAMPLING AT HAZARDOUS WASTE SITES

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The sampling of soil, or any heterogeneous media, requires an understanding of the spatial and temporal scales of interest to the decision makers. Depending on **how** the **media is sampled** and analyzed, and how the data are processed, almost any **“valid”** contaminant concentration can be obtained. In the absence of other data, data from the sampling and analysis of soil (or hazardous waste) are frequently assumed to represent the actual contaminant concentration in the vicinity of the sampling point when emphasis is placed on characterizing the distribution of a contaminant in the soil. When additional **data** are obtained in the vicinity of that sampling point, differences in contaminant concentrations almost always occur. The dilemma, then, is to try to determine the true concentration of the contaminant within a given area or volume of soil using the most efficient sampling design and minimizing the errors that occur during sample collection.

This article will provide a **basic** understanding of the processes and factors involved in the sampling of soils at a hazardous waste site. These processes and factors can be divided into several general categories, which include sample planning, documentation, quality assurance/quality control (QA/QC), sampling network design, site characterization, sample collection, and sample handling and preparation

concerns. Basic guidance on dealing with factors influencing accuracy, precision, representativeness, and comparability of data and how to develop concise statements of the sampling effort objectives will be emphasized. Interpretation of the collected data will be discussed as a means for not only answering the basic objectives of the sampling effort, but also as a means for determining whether the quality of the data is sufficient to meet the needs of the user. Information on how to answer some of the most frequently asked questions, such as, "How many samples are needed and where should they be taken?" will be provided. Regrettably, no simple answers exist. Once the planning phase is completed and the soil sampler goes to the field, discussion of site features that influence the distribution and ultimate management of the contaminants at the site and how to correctly collect the soil sample will be presented. Unfortunately, no checklists are capable of addressing all of the important issues and factors that influence the sampling and analysis of soils and heterogeneous models. Site characterization and soil sampling are multimedia, multidimensional, multidisciplinary efforts that require vigorous communications among all of the principal parties involved. A genuine grasp of the basic concepts and critical definitions is extremely important to perform efficient and effective soil sampling for characterizing hazardous waste sites.

Some of the concepts presented in this article may be new and appear to conflict with existing guidance and procedures. In this article, the basic foundation for the efficient, best scientific investigation of environmental media is the U.S. Environmental Protection Agency (EPA) new data quality objectives (DQO) process. Nothing presented in this article conflicts with that process—a process that focuses on refining the critical questions that drive the decision-making process and on the data that are necessary to provide answers to those questions. The material presented in this article may cause people to rethink how they have been characterizing soils at hazardous waste sites.

SAMPLING PLANNING

Typically, at the beginning of a site investigation, historical information is sought to allow hypotheses and conceptual models of the site to be developed. All potential pathways of contaminant movement and exposure routes to living organisms should be identified and addressed. For example, if the contamination is in the soil, one pathway of concern may be the migration of the contaminant to the groundwater. The project planners should use the limited historical information about the site to determine what additional data are needed and where to collect them to address the critical questions of how much risk the site poses to humans and the environment.

Data are collected from a variety of sources and from a variety of locations to develop a conceptual model of the site. There may be no one complete conceptual model that exists for a given site and other models may need to be developed as the site investigation progresses and new information becomes available. A variety of mathematical models, and sometimes physical models, may be employed to estimate data among collected data points and to predict data at other times, at critical receptors, and in other media.

Far too often, efficient collection of samples and data is difficult because critical definitions and well-defined problem statements are often lacking at the beginning of an investigation even though the conceptual models may have

been well defined. Usually, the controlling factors regarding the amount, quality, and type of data collected are time and money and not whether the results will adequately represent the site, fill the data gaps, or allow for a defensible decision to be made. The traditional thinking is that the more data that can be collected, the better the final conceptual model and decision-making process become.

Data that are collected in a site investigation are often viewed in a spatial context. The increasing popularity of computer-based geographical information systems (GIS) permits the rapid display of both two- and three-dimensional data. Although data from a site investigation are frequently analyzed using classical statistics, geostatistics is increasingly being used. None of these tools, however, can properly be used to answer critical questions about a site unless the critical questions have been properly highlighted and defined. Frequently, the generally accepted critical question that drives an investigation is whether an action level for one, or a variety of contaminants, is being exceeded. This question, by itself, is meaningless unless the support (1) or spatial volume that accompanies the action level is prescribed. Sampling heterogeneous materials, such as soil, can lead to a wide range of valid contaminant concentrations from which comparisons with an action level may be made depending on the volumes of material that are being represented. For example, the concentration of a contaminant in a 5-g sample may exceed the action level, whereas the concentration of contaminant in a soil core sample from which the 5-g subsample was obtained may not exceed the action level.

Terminology

A number of spatial units need to be considered and defined early in the investigation of a hazardous waste site in order to permit the efficient collection of data and the answering of critical questions (Fig. 1). All of these units are used in the development of the conceptual model for a site.

The first unit to consider is the distribution unit. The distribution unit is basically a volume of soil that has been contaminated. A large site may consist of a number of distribution units (ie, contaminated areas). Different processes may have been involved in the distribution unit for distributing

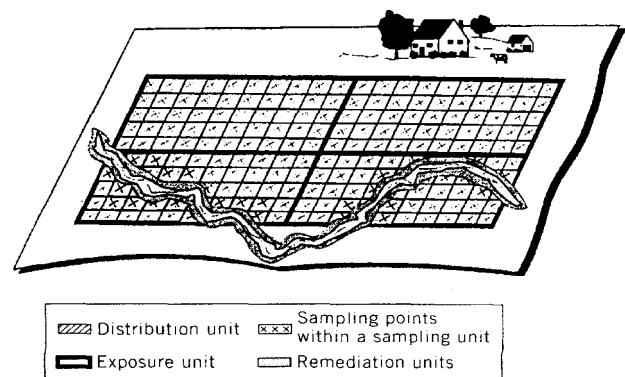


Figure 1. A site may be broken up into a variety of units to allow for critical questions to be addressed in the DQO process.

the contaminant. The contaminant may have been uniformly distributed as one might expect in a settling pond. Conversely, a contaminant may be distributed quite irregularly if leaking drums were stored in the contaminated area. It should be noted that distribution units may be described differently in different regulatory programs, eg, operable units. No matter what distribution units may be called, knowing in advance how the contaminant may have been distributed at a site should influence the decision-making process for determining how and where samples must be collected to answer critical questions about the site. The sampling procedures and number of samples needed to characterize an area where leaking drums were stored can be expected to be different than for the bottom of a settling pond. In statistical terms, the process of subdividing a site into different distribution units that may be sampled differently is called stratification.

The second unit to consider before an investigation progresses significantly is the exposure unit. The exposure unit is the volume of material, in this case soil, for which an action level or threshold concentration applies. Unfortunately, action levels are typically listed without the accompanying exposure unit. Action levels may be listed for humans for residential or industrial exposures, but usually the spatial area for which a person may be exposed to the action level (for some period of time and experience little risk) is seldomly defined precisely. Exposure units need to be defined as precisely as possible before extensive data collection begins if the exposure unit is going to serve as the foundation for the decision-making process. It may be necessary to spend some time to examine the basis and data for the action level to determine the type of exposure, underlying assumptions, and area for which actual exposure and risk of contact with the contaminant is of concern. For example, does the residential exposure unit assume 1/4 acre lots for which an individual may be exposed over a lifetime, on average, to unacceptable risks from the contaminant? Alternatively, are hot spots within the exposure unit of greater concern because the exposure and dose to children are expected to be higher? These are potential questions that must be considered when defining the exposure unit. The establishment of the exposure unit, together with the action level for a contaminant, is important in the development of the conceptual model and the associated decision-making process in a site investigation.

The third unit, the remediation unit is related to the exposure unit through the decision-making process and conceptual models in a site investigation. If the measured and estimated contamination within an exposure unit exceeds an action level, then presumably remediation needs to occur. A remediation unit may then be defined as the smallest practical volume of soil at which a cost-effective remediation can occur. The issue of the smallest practical volume is important because not only is it defined by the contaminant concentrations present but it is also operationally defined by the sampling and remediation equipment to be used during site restoration. For example, if sampling were done with tablespoons, and estimates of exposure units show that remediation is required, backhoes rather than tablespoons, would more likely be used to remediate the area. Although samples were collected with a small support and decisions were based on those data, decisions would likely be made and based on a larger support. If only a small fraction of the exposure unit were contaminated (ie, a hot spot), would it be necessary to remediate the

entire volume of the exposure unit, or can smaller areas be remediated? It may be possible to make decisions that are less costly on the basis of remediation units, eg, backhoe volumes, rather than entire exposure units. Addressing the volume of the remediation unit at the beginning stages of a site investigation allows for development of decision-rules that can guide the decision-making process and the subsequent selection of sampling procedures, analytical methods, and concentration estimation methods. (A decision-rule is described in the following section on EPA's new, revised DQO process.)

The problem of hot spot identification and the required, optimal sampling network becomes easier to address if the remediation units have been defined at an early stage. If hot spots were identified through the use of tablespoon samples at a particular grid spacing, it would not be practical to precisely define the boundaries of the hot spot at relatively small spatial scale if a backhoe were used to remove contaminated areas. High-resolution sampling for hot spots may not be cost effective if the area to be effectively and economically remediated is on a larger scale. Definition of the remediation unit prior to the collection of a significant number of samples from the field permits the costs in obtaining data at various spatial densities (to address a decision-rule) to be balanced against the costs in remediating the area with no further information or data being required.

Data Quality Objectives

All three previously defined units, the distribution, exposure, and remediation units, may be used in the DQO process to achieve an optimization of the site characterization process. The DQO process fosters communications and the development of critical decision-rules for the study of soils and hazardous waste sites (Fig. 2). A stepwise, iterative process, involving major participants in the decision-making process for a site, is used to focus attention and resources on the critical questions or hypotheses that need to be tested. A variety of questions may be answered in an investigation but there are

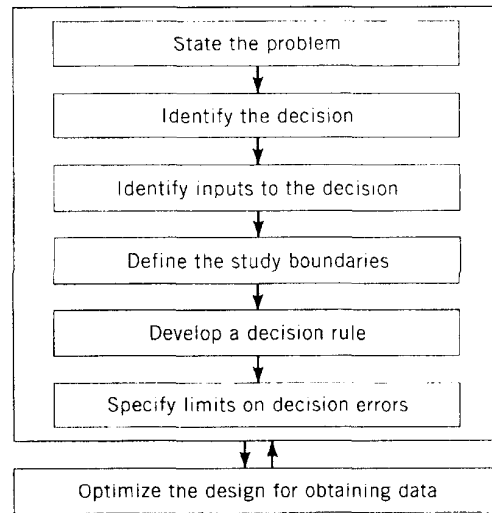


Figure 2. The data quality objective process.

usually just a few contaminants and questions that drive the decision-making process for a site investigation. An example of a decision-rule would be, if the mean concentration of arsenic exceeds the action level within the exposure unit by 10%, then the unit should be remediated by progressively removing those remediation units that contribute to the exceedance.

The DQO process has been used in the past to establish objectives for precision, accuracy, representativeness, completeness, and comparability for the reported data. The major focus of quality assurance and quality control efforts has been on the analytical phases of the sample collection and measurement process. Current DQO guidance from the EPA attempts to focus more efforts toward examining the sensitivity of the decision-making process to all key inputs, rather than just those related to sample analysis. In other words, what are the effects on the basic decision-making process (ie, the decision-rule) from imprecise, inaccurate, nonrepresentative, incomplete, and incomparable data from a variety of sources? This change in emphasis in the DQO process recognizes that there are numerous, and often significant, sources of variability outside the analytical process. There is variability in the process that is used to estimate contaminant concentrations and other physical parameters between measured points. There are errors introduced in the sample collection, subsampling, sample preparation, and sample transportation steps. All of these steps can introduce errors that are significantly greater than those encountered in the analytical process. A well-executed DQO process considers, in parallel, the tolerance for error in the basic decision-making process as well as the tolerance for error in the sample collection and measurement processes.

Further information on the DQO process may be found in the *Guidance for the Data Quality Objective Process* (2) and *The Data Quality Objectives Process for Superfund: Interim Final Guidance* (3) documents. A related document, *Guidance for Data Quality Assessment* (4) describes statistical methods for evaluating data to determine whether the DQOs have been met.

QUALITY ASSURANCE/QUALITY CONTROL

The overall goal of the QA/QC program in any investigation is to assess and ensure that the variability in the data is attributable, as much as possible, to the media and environment that is being measured and not to the measurement (including sampling) process. A properly designed and implemented QA/QC program can achieve that goal. In the field, QA/QC samples may be placed in the sample collection and preparation processing stream at several different locations. Depending on their placement, a variety of different errors can be assessed. However, the determination of the number, type, and placement of QA/QC samples within the sample stream is as problematic for a site investigation as the determination of the number, type, and location of routine samples that must be collected. All too often the number and type of samples to be used in both situations are determined by the projected budget for the site investigation and by estimated analytical costs and not by a judicious consideration of the acceptable error rates in making basic decisions (ie, false positives and false negatives).

A rationale has been developed for how to determine the number, type, and placement of QA/QC samples in soil (5). The decision-making process for determining how extensive a QA/

QC program needs to be for a particular investigation is not simple. A stepwise process needs to be developed in which data from the QA/QC effort are progressively examined and compared to the sample data and to DQOs. Basic concepts need to be understood and applied to ensure that the data collected meets the needs of the decision-maker.

Data that are collected as part of an environmental investigation are going to exhibit variability. The expectation is that variability associated with the measurement process is sufficiently low that variability in the data can be attributed to the measured parameters or population. QA/QC programs are implemented to assess and control measurement variability, but these efforts are frequently insufficient or unable to assess or control all of the sources of bias and variability throughout the measurement process. Often, the QA/QC programs are targeted toward the analytical phase of the measurement process, but considerable bias and variability may be present in other areas.

As reported in the Rationale document (5):

$$\sigma_t^2 = \sigma_m^2 + \sigma_p^2$$

where σ_t^2 = total variability, σ_m^2 = measurement variability, and σ_p^2 = population variability. Also:

$$\sigma_m^2 = \sigma_s^2 + \sigma_h^2 + \sigma_{ss}^2 + \sigma_a^2 + \sigma_b^2$$

where σ_s^2 = sampling variability (standard deviation), σ_h^2 = handling, transportation, and preparation variability, σ_{ss}^2 = preparation variability (subsampling variability), σ_a^2 = laboratory analytical variability, and σ_b^2 = between batch variability. (It is assumed that the data are normally distributed or that a normalizing data transformation has been performed.)

Bias can change over time and become intertwined with imprecision—both of these contribute to variability in the measurement process. A variety of QA/QC samples may be used in a rigorous manner to try to assess the magnitude and source of errors in the sampling of soils at hazardous waste sites. Some of the errors may never be assessed because the true distribution and concentration of contaminants in the environment can never be truly measured; they can only be estimated. The best approach is to try to minimize bias and imprecision at the outset by carefully considering and selecting the sample collection, sample preparation, subsampling, and analytical techniques that are apt to produce representative data with low measurement error.

Some of the most common QA/QC samples used during the collection of soils include field evaluation samples, field blanks, field duplicates, and preparation splits. Field evaluation samples (eg, performance evaluation materials or site-specific soil quality assurance materials) are soil samples of known concentration that are subjected to the same manipulations as routine samples. The field evaluation samples should be introduced in the field at the earliest stage possible. These samples can then be used to estimate total measurement error and, if used in duplicate, precision estimates can be made.

Field blanks provide a measure of various cross-contamination sources, decontamination efficiency, and other potential sources of error introduced from sources other than the sample. Three common types of blanks used in the field include the field rinsate blanks, preparation rinsate blanks, and trip blanks. Rinsate blanks (ie, equipment blanks) are used to assess the efficiency of the decontamination process. These blanks are obtained by pouring deionized/distilled water

over the sampling equipment (for the field rinsate blank) or preparation equipment (for the preparation rinsate blank) after it has been decontaminated and then collecting the rinsate for analysis. Trip blanks are generally used during the collection of volatile organic compounds. A trip blank consists of the sample containers filled with deionized/distilled water, solvent (eg, methanol), or evacuated vials used during headspace analyses. For some sampling efforts (depending on the DQOs), a portion of the trip blanks will be opened in the field (excluding evacuated vials) to assess if there is any contamination that may have occurred during sampling, preparation, or shipment to the analytical laboratory. The remainder of the trip blanks will not be opened in the field and, can be used to assess contamination due to incomplete sealing of volatile organic analysis (VOA) vials and sampling jars or improperly cleaned glassware.

The field duplicate sample is an additional sample taken near a routine field sample and may be used to determine total within-batch measurement variability although differences in the two samples may also be attributable to short-range spatial variability in the soil. If the data from field duplicates are significantly different from the corresponding routine sample, then there is good cause to question the representativeness of the routine sample data and to employ data from other QA/QC samples to determine whether the problem is with short-range spatial variability. If the problem seems to be associated with short-range variability, then a larger support, or compositing of the samples on a small, spatial scale, may be all that is necessary to obtain more representative samples.

Preparation split samples are soil samples that are collected after the soil has been properly homogenized and, thus, all portions of the sample have theoretically equal concentrations. The preparation split samples are used to estimate the sum of subsampling, analytical, and data-handling

error variations (ie, total within-batch error minus the error that occurs during sampling, handling, transportation, and preparation/homogenization).

SAMPLING NETWORK DESIGN

A successful sampling design accomplishes the objectives of the investigation at minimal cost. The DQO process is one approach that provides a sound framework for sampling network design. The DQO process accomplishes three critical tasks. First, the DQO process develops a clear, quantitative statement of the decision that must be made. This is generally in the form of an if-then-else decision rule: If the sample measurements exhibit characteristic X, then take action a; else, take action b. Second, by specifying tolerable error limits on the decision, the DQO process allows for the fact that the true concentration in volume v is never precisely known. Finally, the design-performance diagram can be used to evaluate the expected performance of alternate sampling design options and to select from among the acceptable designs the one with the lowest associated cost.

In the DQO approach, error limits are established across the entire range of possible contaminant concentrations, and can be plotted against true concentration to form a design-performance diagram (Fig. 3). The acceptable range of performance is the area including the gray region and the tolerable false positive and false negative regions. Each sampling scheme has an associated performance curve that shows the probability of making a correct or incorrect decision at any given true concentration. The performance curve (also known as a power curve) shown in Figure 3 represents a sampling scheme that has acceptable performance because it falls within the specified limits. This particular curve also represents a minimum cost design because it meets

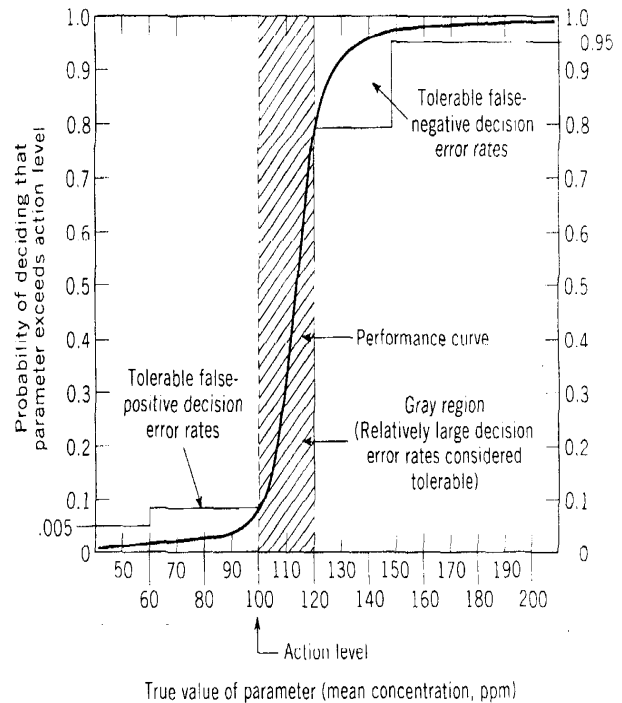


Figure 3. A typical design performance curve used to evaluate the expected performance of alternative sampling design options and to select, from among the acceptable designs, the one with the lowest associated cost.

but does not exceed the specifications. A sampling scheme with fewer samples or less accurate measurement would have higher error rates and be unacceptable, whereas a scheme with more samples or higher quality measurements would be acceptable, but unnecessarily expensive.

It is not easy to develop design performance criteria. Determining acceptable decision error rates is a subjective process that is difficult and uncomfortable for many people to perform. There is a tendency to initially set the error rates so low that they lead to unacceptably high sampling costs, in which case the process must be repeated to obtain a more reasonable sampling design with lower overall associated costs.

Basic Spatial Sampling Strategies

To illustrate some of the basic spatial sampling strategies, two common decision scenarios in soil remediation will be examined. Additionally, alternative sampling strategies under the same decision scenarios will be discussed.

The first decision scenario involves the estimation of the mean concentration of contaminant in a specified volume of soil. Assume that a single distribution unit, or stratum, has been identified, such as the sediment in an abandoned waste lagoon, and that the decision maker will have to remediate the sediments if the mean concentration exceeds a specified threshold. From a design-performance diagram it has been determined that a sampling design will be acceptable if the standard error (*se*) of the mean is less than or equal to 4, or equivalently, a variance (V_c) less than or equal to 16 ($se^2 = V_c$). Further, assume that the measurement error variance (V_m) for collecting and analyzing a soil core is 10, and the population variance (V_p) is 90 (ie, 10% of the total variance is due to measurement error including sample collection, preparation, and analysis, whereas 90% is due to natural variability at the site). The total variance (V_t), including measurement error, is:

$$V_t = V_p + V_m = 100$$

For this scenario, a soil core costs \$25 to collect in the field and \$300 to analyze in the laboratory. The question that needs to be answered is, "What is the best sampling design?"

The classic approach to estimating a population mean is by the arithmetic mean of a random sample. Random sampling is always a valid method, even though it may not always be the most efficient for spatial sampling. In the random sampling approach, it is simple to calculate the standard error of the mean and evaluate the costs of alternate sampling schemes.

For a random sample of N measurements, the variance of the error of the mean (V_c) is simply equal to V_t/N . The design process now involves nothing more than finding the smallest value of N that will meet our example design requirement, namely, $V_c \leq 16$. In this case, $N = 7$, making $V_c = 100/7 = 14.3$ which is < 16 . The final cost of the random sampling design is $7 \times \$325 = \2275 .

The bulk of the cost in the case of this random sampling design is due to laboratory analysis, so let us next consider a less expensive alternative. Assume that an alternate laboratory method is available at a cost per analysis of \$100, but that this method is less precise, with $V_m = 40$ instead of 10. If the initial study objectives were to get high quality measurements of individual soil cores, this approach would not be useful. By taking 4 separate measurements of a single core sample, V_m could be reduced to $V_m/4 = 40/4 = 10$ and equal the precision/quality of a single measurement by the

original method. But the cost of the replicated analyses would be $4 \times \$100 + \$25 = \$425$ per sample—more expensive than the original method selected. However, the original decision rule has nothing to do with measuring individual cores, only with estimating the mean of the sediments in the waste lagoon. For this purpose, $V_t = V_p + V_m = 90 + 40 = 130$. To meet the required objective, nine measurements would be needed so that $V_c = 130/9 = 14.4 < 16$. The cost of this design alternative becomes $9 \times \$125 = \1125 , a substantial financial improvement from the \$2275 cost of the initial random sampling design with the more precise (but more expensive) analytical method.

Suppose that with the initial measurement method, where $V_m = 10$, the population variance is much higher than the measurement variance. In such cases, composite sampling can be a cost-effective alternate approach. In composite sampling, individual core samples are physically combined to make a single unique sample from which one or more measurements of the sample can be made. The overall estimation error is: $V_c = V_p/N_c + V_m/N_a$, where N_c is the number of cores or increments that make up a composite sample, and N_a is the number of analyses performed on a single composite sample. There will also be an additional error component and an added cost introduced by the process of homogenizing and subsampling the composite, but for this example, it is assumed that this added error and cost are negligible. The problems associated with subsampling a composite sample are far from trivial and will be discussed in detail in the subsequent section on particulate sampling theory. In the terminology of that theory, a composite sample is a multiple-increment sample taken to reduce the effects of short- and long-range heterogeneity.

The composite sampling design process becomes one of evaluating combinations of N_c and N_a and selecting the lowest cost combination that meets the initial design requirements. In this case, the best result is obtained when $N_c = 15$ and $N_a = 1$ (ie, a single analysis of a 15-core composite sample). The error associated with the alternate approach would be: $V_c = 90/15 + 10/1 = 6 + 10 = 16$, and the associated cost is $(N_a \times \$300) + (N_c \times \$25) = \$675$. This achieves the original objectives at a lower cost than with individual samples.

From these examples, it has been shown that an improvement on the original design can be achieved by using a lower cost analytical method or by compositing. Can the project planners do even better by combining both methods and analyzing composite samples by the cheaper method? Interestingly, in this particular example, the answer turns out to be no. With a single composite sample, measurement error is again a significant factor, and the cost of reducing V_m through multiple analyses becomes cost-prohibitive. The best option to meet the original goal of $V_c \leq 16$ is with $N_c = 15$ and $N_a = 4$ (where $V_c = V_p/N_c + V_m/N_a = 90/15 + 40/4 = 6 + 10 = 16$), for a total cost of \$775 [equals $(15 \times \$25) + (4 \times \$100)$]. Although this approach costs slightly more than the single, more precise analysis (\$400 vs \$300), the four replicate analyses on the composite sample provide at least some measure of quality assurance in terms of precision (laboratory analytical repeatability).

This example was intended to illustrate the type of economic analysis of sampling alternatives that is part of the DQO process, not to provide a blanket endorsement of composite sampling or cheap but imprecise analytical methods. With different costs, variances, and objectives the conclusions could be quite different.

Estimating Variances

Although this analysis is relatively simple, the results are only strictly true if all of the assumptions are correct. The major assumptions were:

- the variance of the population distribution and the measurement error distribution for each measurement alternative are known,
- the true costs of sample collection and measurement are known, and
- all measurements are independent of each other.

In practice, these assumptions are at best only approximately true, so it is worth looking at how they affect the analysis of sampling alternatives. The relative magnitude of the variance of the population versus the variances of the alternative measurement methods and the relative costs of the alternatives are all important in determining the overall sampling strategy. Generally, the relative costs of measurement methods can be estimated reasonably accurately. The more difficult problem is estimating the error variances. The true variance of a contaminant in soil at a site or within a stratum will always remain unknown. The best way to estimate it is through the analysis of sample data, but this poses a dilemma. It is more difficult to estimate variances precisely than it is to estimate means. It is likely that by the time one collects enough samples to get a satisfactory estimate of variance, there will be far more data than needed to efficiently estimate the mean. The situation becomes worse if composite sampling or another alternate measurement method were evaluated because an estimate of the measurement variance(s), in addition to the total variance, is needed. Analytical variances are often well-known, but measurement variance includes many sampling and subsampling error components that are site-specific. Estimating measurement variance from sample data requires the analyses of multiple field replicates. Estimating these variances can easily cost many times more than simply estimating the mean.

The alternative to estimating variances from data is to estimate them through educated guesswork (ie, best professional judgement). Usually, at least a few measurements will be available from previous investigations which can in turn be used to estimate the measurement variance. Analogies can be made with data sets from other similar sites. Measurement variance can likewise be estimated from quality assurance data collected at similar sites, or it can be estimated by combining the analytical variance from laboratory studies with estimates of preparation and subsampling variances from particulate sampling theory (as described later in this article).

The question of which approach to take to estimate the variances should be addressed as part of the data quality objectives process. In addition to determining the decision to be made and how accurately it must be made, it is also necessary to know how accurately the decision maker needs to know how accurate the decision is. Unfortunately, there is no standard formula for determining these requirements. In the previous example, if the single-composite, single-analysis option is chosen, there will be no way to evaluate whether or not the desired precision is achieved. The driving factor will be the magnitude of the consequences of decision errors. Suppose in the previous example that the variances were preliminary

estimates based on prior investigations of similar sites. Suppose, also that the decision involved is relatively minor; for example, whether to leave slightly contaminated soil in place or remove it to a municipal land fill at a cost of \$5,000 to \$10,000. In this situation, the described analysis might well be sufficient, and one of the compositing schemes discussed would be chosen. However, if the decision were between disposal in the municipal landfill versus in a hazardous waste facility at a typical cost of \$100,000, it is reasonable to spend more money on sampling in order to be more confident about the quality of the decision. Again, there is no unique simple strategy for such cases; nonetheless, some form of sequential sampling may be appropriate.

In sequential sampling, an initial set of data is collected and evaluated. The decision to be made is now threefold: yes, remediation is required; no, the soil is clean and can be left *in situ*; or, collect more data to help better define the situation. The initial data is used to estimate the mean, variance, and standard error, and if the standard error is low enough, the decision is made. If the standard error is too high, the variance is used to estimate how many additional samples are needed in the second sampling phase. The process is repeated until a confident yes or no decision can be made. The number of samples collected in the initial set is determined by economics and logistics. If measurements are being made in the field, it may be practical to re-evaluate the data after each measurement. This approach minimizes the total number of samples required. However, if the samples must be sent to a laboratory for measurement, there are time delays and mobilization costs each time a sampling crew returns to the field. This may make it advantageous to make a conservative initial estimate of variance and attempt to over-sample the initial phase to avoid further sampling.

Sequential sampling can be used with either individual or composite samples. Unfortunately, economic analysis of the alternatives is very difficult, especially when the population is not normally distributed. Real-world distributions of contaminated soil measurements are often highly skewed and contain nondetects (ie, data below the instrument or method detection limit). In such cases, the only practical method for comparing the cost-effectiveness of alternative sequential designs is through rather computer simulations of the sampling process. Composite samples may well have an advantage in that the composite samples will be more normally distributed than the original population. The occurrence of a more normally distributed population makes it more likely that normality-based tests, such as Student's *t*-test, can be used with a small number of measurements.

Spatial Dependence

In the previous examples, it was assumed the data were independent. However, the fate and transport of contaminant in soil is determined by physical and chemical processes that do not operate at random. This is also true of natural soil parameters such as clay content, porosity, etc. that can influence the spatial distribution of contaminant. In general, it is expected that soil measurements taken a few centimeters apart would be more similar in concentration than when the measurements are 1&en kilometers apart. Quantifying such spatial dependence and how it affects sampling and estimation is the subject matter of geostatistics. The reader is referred to

Reference 6 for a practical introduction to geostatistics. Additionally, Reference 7 provides an excellent discussion of the specifics involved in spatial dependence during soil sampling applications. Only a brief discussion of the basic concepts and a few rules-of-thumb for sampling and estimation will be provided in the following text.

Spatial dependence can be quantified in the form of a semivariogram (Fig. 4), which shows the variance of measurements as a function of the spatial vector (distance and direction) separating the points. More precisely, the semivariogram shows the expected value of $(z(x) - z(x + h))^2/2$, where z is the analyte concentration at points x and $x + h$, and sampling location $x + h$ is separated from sampling location x by the vector h . The semivariogram incorporates all of the components of the total variance. The y-intercept at $h = 0$ includes all of the independent measurement variance components, such as field and laboratory preparation, subsampling, and analytical error. The semivariogram at very small h values (ie, very small distances from the original sampling location) would reflect the variance of collocated samples. The shape of the semivariogram curve defines the spatial dependence structure. The mean value of the semivariogram over all possible sample pairs at all possible separation vectors is simply the total population variance. The semivariogram provides a very useful model of both independent and dependent variance components, but like any of the components, creating a semivariogram entirely from data can be very expensive.

When data are spatially dependent, the arithmetic mean is no longer the best (minimum error variance) estimate of the population mean. Instead, a weighted mean should be used. In geostatistics, a weighting method called kriging is frequently used, in which the semivariogram is used to compute sample weights that minimize the estimation variance. When two or more samples are clustered very closely together (ie, sampling distances between two points is small), whether by chance or by design, they are effectively providing duplicate information. This duplication is indicated by low semivariogram values among neighboring samples and smaller weights are assigned to such samples. Conversely, samples with no near neighbors receive higher weights. By down-weighting clustered data that

are duplicative, kriging provides better estimates than simple averaging.

The most efficient sampling design, when spatial dependence exists, is one that minimizes the duplication of information; or equivalently, maximizes the distance from each sample to its nearest neighbor. The solution to this problem is simple: use a systematic sampling scheme and sample at regular sampling grids. It has been shown (7) that grid sampling can be significantly better than random sampling in some cases. They also reported that the arithmetic mean of a regular grid is essentially equivalent to the kriged mean for the same data set. This suggests that regular grids are also optimal for composite sampling where the physical mixing of the soil cores is equivalent to arithmetic averaging. Triangular grids are the most efficient (in two-dimensions) but square grids are very nearly as good and are generally more convenient in the field. Because it is usually safe to assume the existence of some form of spatial dependence in soil contamination, regular grid sampling is recommended, preferably with a random origin and orientation.

Sampling for Local Estimation

The purpose of local estimation is usually to classify a site into higher concentration areas that need some form of remedial action and lower concentration areas that need none. When it is assumed that the site contains high and low concentration areas, it is the same as saying that there is spatial dependence (ie, similar values tend to be grouped together). This spatial dependence becomes a significant factor in sampling network design. Similar to global estimation, sampling on regular grids is more efficient for local estimation.

Local estimation and decision-making can become very complex. It is critical to establish a very clear decision rule at the beginning that must include the scale of the decision. The simplest approach to local estimation is to subdivide the site into an array of discrete units. These discrete units may be as large as exposure units or as small as remediation units. Generally, high remediation costs lead to the consideration of smaller decision units in an attempt to be more selective in which units must undergo the expense of being remediated. Each unit will be classified based on an estimate of its mean concentration. Subdividing a site into smaller remediation units means that many decisions will be made instead of just one and that the consequences of error for any given decision become much smaller. The DQO process should reflect this by establishing broader error tolerances for smaller units.

Composite sampling remains an attractive option for local estimation because compositing increases the area represented by each sample and reduces the sample variance. Compositing is particularly effective when individual core concentrations vary greatly over distances that are small compared to the scale of decision, and when this short range variability is high relative to measurement error. Generally the larger the area over which a composite is taken, the greater the reduction in sample variability; however, the composite area should not be larger than the decision unit.

Local estimation differs significantly from global estimation because the local unit being estimated is a part of the larger population. As a result of spatial dependence, each unit is correlated to some extent with its neighboring units. It becomes possible to estimate the concentration of a unit by inter-

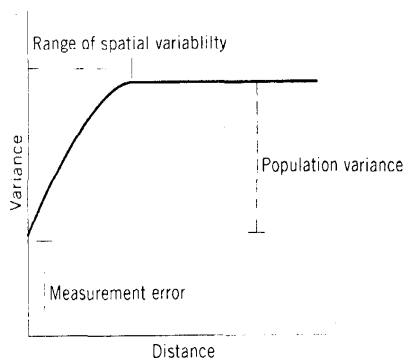


Figure 4. A typical semivariogram showing the variance of measurements as a function of the spatial vector (distance and direction) separating the data points.

polation from nearby data, even when there are no samples in the unit itself. In this case the design problem is no longer one of adequately sampling each unit, but rather one of sampling the population area in order to adequately estimate each unit.

There are two basic approaches to optimizing sampling designs for local estimation. Both require an estimate of the semivariogram. The first approach is based on the fact that when estimates of remediation units are made by kriging, estimates of the corresponding standard errors are also produced. The basic assumptions involved in kriging result in the standard error estimates being determined by only the semivariogram and the sample locations, not by the data values, so it becomes possible to calculate kriging standard errors for hypothetical sampling network designs. The performance of alternate designs can be compared to the established decision error limits. The approach has been detailed (8,9). The major problem with this approach is that kriging estimates of kriging standard errors are quite sensitive to errors in estimating the semivariogram, and to errors in the basic assumptions.

A promising alternative approach to the problem of developing complex designs for local estimation has been described (10, 11). This approach involves first creating a detailed computer model of the soil contamination distribution that is consistent with both the semivariogram and with any available data using a geostatistical technique called conditional simulation. The resulting model contains a very dense grid of data points (typically, 10,000 or more points). A few of these grid points will contain actual measured data values whereas the remainder of the grid points will contain simulated measurements that are both reasonable and realistic in detail. This approach is somewhat less sensitive to errors in estimating the semivariogram because the model is forced to fit the actual data values. After the detailed model has been constructed, it is possible to simulate the entire process of sampling, estimation, and decision-making for any particular sampling design and to compare the cost-effectiveness of alternative designs. The basic procedure for evaluating a sampling design by conditional simulation is as follows:

1. Overlay a grid of decision units over the modeled area. Each unit will contain many simulated sample values.
2. Calculate the mean concentration of all the simulated values in each unit. These are the true concentrations of the units. Comparing these means to a specified action level determines what the correct remediation decision for each unit should be.
3. Select a sampling design scheme, such as 50 samples at randomly selected locations over the model area.
4. Determine a set of sample locations and draw the simulated sample concentration values from the model.
5. Estimate the mean concentration of each decision unit, apply the decision rule, and determine whether the decision is correct by comparison with the true concentration from step 2.
6. Repeat steps 4 and 5 at least 100 times with different sample sets generated by the same sampling scheme and keep track of all decisions obtained.
7. On a design performance diagram, plot the proportion of correct decisions versus the concentration for each of the decision units. If all of the points fall within the

design performance limits, the sampling scheme is deemed acceptable.

The conditional simulation procedure is complex and clearly best suited to large, expensive remediation projects. Its primary advantages include the flexibility to evaluate alternate spatial designs, such as regular grids and composites; to tabulate detailed performance statistics, such as costs of sampling and remediation; and to quantify the amount of contaminant that would remain unremediated following the selected sampling design.

This simulation approach can also be used to evaluate the cost-effectiveness of the overall sampling/remediation strategy. In a design-performance diagram like that in Figure 3, the right-hand boundary of the acceptable performance zone limits the probability that highly contaminated soil will remain unremediated, whereas the other boundary limits the probability that relative uncontaminated soil will be unnecessarily remediated. The former is of primary interest to the regulator because it protects against risk to humans and the environment. The latter boundary, however, is primarily an economic choice on the part of the party responsible for remediation. The trade-off is between sampling and remediation costs. In practice, the actual decision level will occur at estimated concentrations near the center of the gray region. Moving the left-hand boundary farther to the left lowers the effective decision level and increases the amount of remediation to compensate for the greater uncertainty due to less sampling. Moving it to the right raises the effective decision level and increases the amount of sampling required. Simulation can be used to evaluate these trade-offs and find the most cost-effective solution that meets the regulatory requirements.

DOCUMENTATION

Accurate documentation is essential for the success of a field sampling program. Documentation should occur in all phases of a soil sampling program including: planning, sample collection, and laboratory analysis. Three documents usually required are the field sampling plan (FSP), quality assurance project plan (QAPP), and health and safety plan (HASp). The FSP provides guidance for all fieldwork by defining in detail the sampling and data-gathering methods to be used on a project (12). Topics that should be discussed in the FSP are the site background, sampling objectives, sample location and frequency by matrix (including QA/QC samples), sample designation, sampling equipment and procedures with standard operating procedures (SOPS), if available, and sample handling and analysis. The QAPP describes the policy, organization, functional activities, and QA/QC procedures necessary to achieve the DQOs defined for the project. The detail to be included in QAPPs depends on the category of investigation being undertaken. Most investigations under the Superfund program have compliance or litigational implications and thus require discussion of the following: 14 points: project description; project organization and responsibilities; quality assurance objectives; site selection and sampling procedures; sample custody; calibration procedures and frequency; analytical procedures; data reduction, validation, and reporting; internal quality control checks; performance and system audits; preventative maintenance; calculation of data quality

indicators; corrective action; and quality control reports to management (13). If both a FSP and QAPP are required for the same project, to avoid duplicative efforts for sections that contain the same information, it is recommended to simply reference the overlapping section in the one document from the other document. The HASP is prepared as a support document to protect the health and safety of the soil sampler and to meet OSHA guidelines and regulations (12). Topics to be discussed in a HASP include: identification of key health and safety personnel (eg, site health and safety officer); hazard risk analysis by task and operation; employee training requirements; personal protective equipment; medical surveillance requirements; frequency and types of air monitoring; site control measures; decontamination procedures; standard operating procedures for the site; emergency response procedures; and confined space entry should sampling be performed in a pit or hole.

During sampling and analysis, it is only through proper documentation that a soil sample can be linked with a sampling location (including depth) and the date and time of collection. Use of detailed field and laboratory logbooks to document routine information (eg, sampling date, weather conditions, sample numbers, QA/QC sample use, air monitoring data, etc), problems encountered during sampling (eg, inability to penetrate to desired depth), sampling location changes, site characterization information (to be discussed), soil characteristics, production of the site map (to be discussed), or analytical procedural changes. The use of standardized forms greatly enhances field documentation by ensuring the collection of complete information required at the site. Other required documentation associated with sample identification and shipment include: sample labels; chain-of-custody forms, custody seals, and shipping airbills (14).

SITE CHARACTERIZATION

Whenever a soil sampler, sampling team, or management team approaches a new site that needs to be evaluated, one of the initial steps performed should be site characterization (ie, site description). A good site characterization program consists of two major steps, namely, site observation and subsequent site mapping. Site characterization may be limited to a reconnaissance survey or involve a full-scale (or definitive) survey. A reconnaissance survey is performed to confirm historical data through the collection of a few selected soil samples for analysis or to identify site features that may indicate contaminant sources, pathways, affected populations, and potential monitoring/sampling locations. A full-scale survey is undertaken to obtain soil samples for analysis with their results being used in the final decision-making process (15).

For both survey types, a basic suite of characteristics should be noted and recorded during the site visit. This suite includes information and observations on climate and weather, slope, surface erosion and erodibility, surface runoff potential, vegetation, and the presence of macro- and meso-fauna. Even though these parameters are readily observed, they should be recorded because they may provide explanations for observed trends in the data and possible considerations for long-term planning and remediation efforts at the site.

In addition to the site characteristics, numerous soil characteristics should be examined and recorded in the field because they provide information on potential subsurface

transport pathways, variability of soil properties, location of contaminants, and identify features that may influence remedial operations. More complete descriptions and information on site characteristics are available (16,17) (see *SITE CHARACTERIZATION, EXPEDITED*).

Site Observations

Climate and weather are site characteristics that often influence the collection of soil samples. Climate, the average condition of the weather over a period of years, influences the time of year that sampling and site remediation efforts can occur (16). Clearly, soil sampling during the winter months in the northeastern United States may be restricted due to cold weather and frozen ground conditions, yet in contrast, winter sampling in the southwestern United States may be preferable to avoid the extreme high temperatures commonly encountered during the summer months. Weather conditions, the state of the atmosphere during the field investigation, influences the specific time of sampling. Specific conditions to note include temperature, precipitation, wind speed and direction, and humidity. While often noted, but not recorded, these conditions generally influence the health and safety of the sampler and have little direct influence on soil sample collection with the exception of precipitation. For example, when the wind is blowing, samplers should always position themselves upwind to avoid inhalation of dust and other particles that may contain the contaminants of concern. During time of high winds, sampling should be avoided to prevent the spread of the contaminants sorbed on fugitive dust particles leaving the waste site.

Noting the slope of the soil surface is important because it influences the rate and amount of runoff and erosion of water, soil, and associated contaminants (16,18). Slope features to be noted include gradient, length, shape, and topographic position. Simple terms such as flat, moderate, or steep can be used to describe the gradient. If greater accuracy is required, slope gradients may be recorded as a slope percentage (the change in elevation per horizontal distance between two points) or a slope angle (measured in degrees from horizontal). Slope length, usually measured in meters or feet, influences the behavior of water and the potential for erosion. In general, longer slopes have greater runoff and erosion potential than shorter slopes (18). Slope shape (eg, convex, concave, or flat) and topographic position (eg, summit, shoulder, sideslope, or floodplain) influence the movement of water and soil on the surface and in the subsurface. For example, steep convex shoulder positions are more likely to experience erosion than flat or concave floodplains in which soil deposition is likely to occur (19).

Site characterization of surface erosion is important to assess soil loss or deposition in the past and to assess future erosion potential. The determination of surface erosion is easily done visually by comparison of observed changes in soil texture and color between surface and subsurface horizons (16). If subsurface colors or textures are noted at or near the surface, then that area has undergone and is prone to erosion. The presence of rills, gullies, or other erosional features also indicate erosion is occurring at the site and mark potential pathways for movement of contaminated soils within and from the site. Valuable information on surface erosion and erodibility of soils at a site can be found in county soil surveys published

by the U.S. Department of Agriculture (USDA)–National Resource Conservation Service (formerly the USDA–Soil Conservation Service).

Surface runoff potential is used to evaluate the potential for transport of contaminants at the soil surface to surface streams or other water bodies (16). The runoff potential is controlled by slope and saturated hydraulic conductivity. Saturated hydraulic conductivity may be defined as the rate of movement of water through a soil or as a measure of the ability of a soil to transmit water under saturated conditions (20). Runoff is directly proportional to slope (ie, as slope increases, surface runoff potential also increases) and inversely proportional to the saturated hydraulic conductivity (ie, as the hydraulic conductivity increases, the runoff potential decreases). Similar to surface runoff, valuable information on soil hydraulic conductivities and surface runoff may be found in soil surveys published by the USDA–National Resource Conservation Service.

Vegetation serves as an indicator of site history, erosion potential, and contaminant location. During site characterization, the sampler should observe the nature (eg, hardwood forest), kind (eg, maple trees with mixed understory), extent (eg, dense, scattered, sparse, or bare), and distribution of the site vegetation. The presence of stunted vegetation, luxuriant plant growth in comparison to surrounding areas, discolored leaves, and burn spots can all indicate the toxic effects of contaminants in the soil. In areas of heavy metal contamination, sampling of vegetation along with soil sampling may be desirable to assess potential metals exposure through bioaccumulation (16).

Macro- and mesofauna, like vegetation, can provide an indication of the presence of contaminants. Additionally, during their routine daily activities, fauna create potential pathways to enhance vertical movement and leaching of contaminants to depth within a soil, mix the soil, and are capable of moving large quantities of subsurface materials to the surface. Macrofauna are those animals that can be measured in centimeters (or inches) such as burrowing rodents, earthworms, and insects (16). In contrast, mesofauna are the smaller animals including other insects, arthropods, nematodes, and smaller worms. Simple terms, such as many, common, few, and none, can be quickly recorded to indicate the presence of indigenous fauna. A lack of any visible fauna may indicate an area of high contaminant concentrations.

Site Mapping

The second step in the site-characterization process is the production of a site map. Site mapping generally occurs in two phases: initial and final mapping. The site map is important because it allows for planning sampling locations and allows for the subsequent plotting of the contaminant spatial distribution. The initial site map is a crude sketch of the sampling site. Features that should be drawn on the site map include both permanent and temporary objects. Permanent objects to be mapped include playgrounds, buildings, roads, railroads, boulders, and bodies of water (eg, streams or rivers). Temporary objects include trees, fences, power poles, and telephone poles. Drainage ways (eg, gullies or depressions) are sometimes plotted because they represent potential pathways of contaminant migration. Other features that may be included on the initial site map are bare spots, percent vegetative cover, and

unusual surface features such as soil discolorations and surface staining.

Once the first draft sketch of the site has been completed, the approximate locations of the sampling points can be plotted based on the sampling design. More exact locations of the sampling points can be made using surveying equipment or global positioning systems (GPS) units depending on the needs of the program. It will be from these marked points that the samplers will collect the soil samples. If changes in sampling location need to be made due to unmapped surface features or unforeseen subsurface obstacles (eg, large tree roots, buried rocks, etc), the sampler should indicate the type of obstacle encountered, move approximately 30 cm (12 inches) in any direction away from the obstacle, and try to recollect the sample. This process may have to be repeated several times in different directions from the initial point or at greater distances until a satisfactory location is found. The new sampling point should be marked on the site map to indicate the sampling location change.

After sampling has been completed, the final location of each sampling point needs to be accurately determined. Exact locations of the final sampling points can be made through a variety of different techniques ranging from measuring the distance and direction from a fixed reference point (or points) with tape measures, through the use of field survey equipment and GPS units. Each sampling point should be reconfirmed as to its approximate spatial location in the field and on the site map. A more formal site map may then be produced using this collected information in conjunction with, or separate from, the analytical results.

SAMPLE COLLECTION

After the site has been adequately mapped and all important site observations have been noted, the next step is to collect the soil samples. It is often quoted that it is during sample collection that a majority (80% or more) of the total measurement error occurs and yet it is during this phase of a program that the least attention to detail is paid by the decision-makers, project managers, chemists, etc. The following text provides guidance on how to properly collect a soil sample taking into account all the potential pathways for error to enter the system, provides means to minimize these errors, provides information on common concerns during the selection of sampling equipment, and discusses the advantages and disadvantages of commonly available sampling tools.

Particulate Sampling Theory

A particulate sampling theory was developed by Pierre M. Gy for the mining industry to provide a more accurate and precise means for identifying heterogeneous ore grades. The theory is based on the relationship that exists between the variability of the material, the particle sizes within the material, the distribution of the component of interest (eg, pollutant), and the size of the sample collected. Particulate sampling theory is based on sampling correctness, which in turn is based on a property intrinsic to the material itself and the equipment used to extract the sample (21). Gy defines a correct sample as "A sample in which all particles in a randomly chosen sampling unit have the same probability of being selected for inclusion in the sample". The importance of this theory to environmental soil sam-

pling is that the theory identifies various sources of error that can influence the final determined contaminant concentration and presents the means for controlling these errors. The following text is a very brief synopsis of Gy's particulate sampling theory as it pertains to soil sampling. A more complete and in-depth discussion of all aspects of the sampling theory is available (21).

Two models are described in Gy's particulate sampling theory: a continuous model which deals with variables through time and space, and a discrete model which addresses sampling populations of fragments. Because soil sampling is more closely related to sampling populations of discrete fragments, only those errors associated with the discrete model will be discussed. The errors associated with the discrete model include the fundamental error; grouping and segregation; short-range heterogeneity; increment delimitation; increment extraction; preparation; and analytical errors.

Sources of Variation and Sampling Error. One of the key concepts of Gy's particulate sampling theory and its determination of the fundamental error is that the relationship between the maximum particle size in a collected sample and the weight of the sample to be collected must be clearly defined. By following the formulae (to be presented), the sampler can select a particle size range that will reduce the relative variance in the final results to an acceptable level (as defined in the DQO process) and ensure that enough soil is collected to present an unbiased sample to the laboratory for analysis.

The fundamental error (FE) is associated with the natural variability inherent (ie, the heterogeneity that is inherent to the composition of each fragment or particles making up the lot) in the composition of every particle making up the lot to be sampled (21). A lot is defined by Gy as the batch, volume, or sampling unit from which increments and samples are collected. Lots may range in size from a bottle of soil at the laboratory, to a dump truck load of soil, through the entire hazardous waste site under investigation. The fundamental error is the only error that cannot be eliminated; however, it can be reduced by comminution of the maximum particle size or by taking larger sample sizes in the lot. Mathematically, the fundamental error is defined as:

$$s_{FE}^2 = (1/M_S - 1/M_L)IH_L$$

where s_{FE}^2 = relative variance of the fundamental error; M_S = mass of the sample (g); M_L = mass of the lot (g), and IH_L = constant factor of constitution heterogeneity. When the mass of the lot, M_L , is large in relation to the sample weight to be collected, M_S , the formula can be simplified to:

$$s_{FE}^2 = IH_L/M_S$$

IH_L can be estimated using the following relationship:

$$IH_L = fgc^3d^3$$

where f = shape factor (dimensionless); g = particle-size (or granulometric) factor (dimensionless); c = mineralogical factor (g/cm^3), ℓ = liberation factor (dimensionless), and d = diameter of the largest particle (cm).

The shape factor (f) addresses error due to all fragments not being perfect cubes and thus not fitting perfectly through the square holes in sieves used to screen the soil (21). The shape factor is influenced by the number of particles passing through two consecutive upper and lower sieves, average particle

diameter, average particle density, and the mass of the fraction to be analyzed. Mathematically,

$$f = M/pd^3\lambda$$

where M = mass of fraction collected on the lower sieve (g); p = number of individual particles; d = average diameter of particles passing through the two sieves (cm), and λ = average density of collected fraction (g/cm^3). For all practical purposes, a quick examination of the sample under a microscope is sufficient to determine the shape factor based on visual observation and comparison with the following approximate shape factor values. The shape factor used in calculating the fundamental error can be approximated by using values of 0.5 or slightly less for most minerals and soils, 0.1 for flat minerals such as micas, and ≥ 1 for acicular minerals, such as asbestos and tourmalines. Shape factors as large as 10 may be used in the presence of very long, thin, needle-shaped minerals.

The particle size distribution factor (g) addresses error due to all fragments not being exactly the same size and thus not being the coarsest fragments in the sample (21). Mathematically,

$$g = \sum M_{L\alpha}/M_L$$

where $M_{L\alpha}$ = mass of the size fraction (α) of concern (g), and M_L = mass of the lot (g). Two general assumptions for g that can be made in calculating the fundamental error are that for most soils, g is approximately equal to 0.25 and that if the soil has been screened to a particular size fraction, then g can be approximated by a value of 0.55.

The mineralogical factor (c), also known as the chemical or mineralogical composition factor, accounts for the maximum heterogeneity within the lot when the mineral (or constituent of interest, eg, a pollutant) is completely liberated from the other material (gangue). It results from differing particle densities and associated differing concentrations (21). Although the full mathematical formula accounts for the average concentration of the lot and the varying densities of the fraction of concern and the gangue or rest of the material, for most soils the following formulas may be used:

$$c = \lambda_M/a_L \text{ if } a_L < 0.1 \quad c = (1-a_L)\lambda_c \text{ if } a_L > 0.9$$

where λ_M = density of pure mineral or constituent of concern (g/cm^3), a_L = critical content (proportion of the constituent of interest in the lot) of the lot (dimensionless), and λ_c = density of gangue or background materials (g/cm^3). A particle density of 2.6–2.65 g/cm^3 is often used to represent the density of the soil in these formulae. The critical content (a_L) of the lot is usually an approximation based on historical data about the proportion (or concentration) of the contaminant of concern at the site.

The liberation factor (ℓ) is a correction factor taking into account that c is a measure of the maximum possible heterogeneity where the contamination occurs as completely separated (liberated) discrete particles within the soil matrix (21). Thus ℓ , whose values lie between 0 and 1, adjusts for heterogeneity of the sample or lot. Mathematically,

$$\ell = (a_{max} - a_L)/(1 - a_L)$$

where a_{max} = maximum proportion of the pollutant associated with the largest contaminated particles in the lot, and a_L = average proportion of the pollutant in the lot. The liberation

factor can be estimated depending on the degree of visual heterogeneity within the sample using values of 0.8, 0.4, 0.2, 0.1, and 0.05 for matrices that appear to be very heterogeneous, heterogeneous, average, homogeneous, and very homogeneous, respectively. At most hazardous waste sites where soil contamination is the result of a spill, t values range between 0.05 and 0.2. In contrast, where discrete contaminant particles have been spread out at a site (eg, ground Pb plates from used automotive batteries), then a liberation factor of between 0.8 and 1 may be appropriate.

The final and perhaps most influential factor used in the determination of the fundamental error is the diameter (d) of the opening of a square mesh retaining no more than 5% of the sample (21). The largest particles strongly influence the fundamental error because they have the greatest inherent heterogeneity among particles.

By knowing the factors that control the FE, the sampler can minimize, but never eliminate, the fundamental error. However, the fundamental error is not the only source of error or bias that must be considered during the collection of a soil sample. The sampler must be careful to collect a correct sample and thus avoid introducing the other biases into the final results. These biases are the result of the grouping and segregation, increment delimitation, increment extraction, preparation, and analytical errors. Fortunately, these errors can be minimized or eliminated through the careful collection of the soil sample.

The grouping and segregation error (GE) results from the distributional heterogeneity, the heterogeneity that is inherent to the manner in which separate and distinct particles (or aggregates) are scattered or spread out within the lot to be sampled (21). The grouping factor reflects the number of increments (ie, the group of particles extracted from the lot in a single operation of a sampling device) making up the sample compared to the number of fragments (ie, particles) making that sample. The grouping factor approaches zero as the number of increments approaches the number of fragments. The segregation factor accounts for the segregation of different types of particles and the natural range between the minimum and maximum distributional heterogeneity within a sample. The segregation factor ranges between zero and one. The major factor affecting GE is gravity. GE can occur due to gravitational separation that results from differences in:

- particle density
- size
- shape (eg, round particles tend to move or roll easier in the sample or pile than flat particles)
- magnetic properties (eg, magnetite particles will preferentially tend to stick to themselves or steel sampling utensils)
- electrostatic charge (eg, charges on the sides of plastic bottles or bags can cause the adhesion of charged clay particles)
- moisture which tends to clump soil particles together
- air turbulence during sample splitting or homogenization can cause loss of the fines and
- vibration (eg, during transport from the site, on a laboratory cart, or from a vibrospatula used to obtain subsamples) in which heavy particles will slowly settle to the bottom of the sample container.

To minimize the grouping error, the collection of as many small increments as practically possible is recommended. This process of collecting numerous small increments increases the probability of collecting all types of groups for inclusion in the sample and thus reduces the grouping error. To minimize the segregation error, homogenization of the lot before sampling is recommended taking care that the homogenization process itself is free from the factors that increase GE.

The short-range heterogeneity error (CE_1) is a random, discontinuous error that is influenced by FE and GE ($CE_1 = FE + GE$) and thus, can only be minimized (21). CE_1 is what most soil samplers are referring to when they discuss the natural variability of a soil through time or space and what geostatisticians call the nugget effect. Because CE_1 is a random and discontinuous error, it can only be minimized by performing correct sampling and, thus, minimizing the factors that influence FE and GE.

Sampling is a selection process. The selection process can either be probabilistic (ie, sampling with a random selection component) or nonprobabilistic. Nonprobabilistic sampling (eg, grab sampling) can never be strictly correct. Probabilistic sampling can be correct or incorrect. Probabilistic sampling is correct when all of the constituents of the lot to be sampled have an equal probability of being sampled and when all constituents that are not of the lot have no probability of being selected. The error associated with the selection/sampling process is known as the materialization error (ME) and is the combination of the two relative variances, the delimitation error (DE) and the extraction error (EE); that is, $ME = DE + EE$. The materialization error must be minimized to approach the notion of correct sampling. (Note: although the preparation error (to be discussed) is technically part of the materialization error, it is usually treated separately.)

The increment DE can be a major source of sampling bias and results from incorrectly defining the boundaries or limits of the volume of material to be extracted and physically collected from the sample (21). In order to define the proper shape, the dimensionality of the lot, which ranges from 0 to 3, must be defined. A zero-dimensional lot exists when the whole lot is used for analysis and, thus, no DE exists, or when the order (through time or space) of the sampling has been lost or is irrelevant (eg, a processed laboratory sample). Similar to a zero-dimensional lot, a one-dimensional lot may be defined as a thin, continuous, elongated pile or stream, except that the order of the sample is important. For zero- or one-dimensional lots, a correct sample is obtained by collecting all of the soil between two parallel planes cut across the entire sample. An example of a one-dimensional lot may be a core sample from which a slice is cut for the analytical subsample.

A two-dimensional lot is correctly sampled by a cylinder with a constant cross-section and consists of a mass with an upper and lower boundary. The third dimension, although existent, for a two-dimensional lot should be insignificant when compared to the other two dimensions. Typical examples of two-dimensional lots in a soil is a horizon (or a designated sampling layer) in which the thickness of the horizon is negligible compared to its horizontal distribution or a core sample collected through the entire soil horizon.

A three-dimensional lot is a sample in which all three dimensions are important and significant. Examples of three-dimensional lots include: large waste piles, truck loads of soil, or an entire waste site. Three-dimensional lots are

nearly impossible to sample correctly because the proper shape of the sample is a sphere. Three-dimensional samples are typically reduced to two-dimensional samples for correct subsampling purposes. The one- and two-dimensional lots are most frequently encountered during soil sampling. The two-dimensional lot is collected in the field, whereas the one-dimensional lot is sampled during sample preparation and analysis.

The other important aspect of DE is that the sampling device must be able to physically collect all particles (or portions of particles) that lie within the edges of the sampling device (Fig. 5). If a particle or fraction of particle lies outside the sampling device, then it must be excluded from the collected sample. However, collection of only those portions of particles that lie within the edges of the sampling device is neither practical nor possible because the sampling device cannot cut soil particles. To account for the cutting error associated with the sampling device as it delimits the sample, the increment extraction error (EE) has been defined. To properly extract a sample, the rule of the center-of-gravity must be respected (21). The rule of the center-of-gravity simply states that if the center-of-gravity for a particle lies within the sampling device boundaries, then the fragment must be included with high probability in the sample (Fig. 6). Conversely, if the particle's center-of-gravity lies outside the sampling device boundaries, yet part of the particle lies within the sampling device's edges, then the particle must be excluded with high probability from the collected sample (Fig. 7). Factors that can affect the center-of-gravity rule during soil sampling are the:

- Straightness of the cutting edge: The tips of the cutting edge should be superposable to each other by simple lateral translation (ie, both edges of equal length).
- Shape of the cutting edge: The cutting edge should form an angle between the outer wall of the sampler, and the line parallel to the cutting edge should be less than 45° or equal to 90° (a flat cutting edge). If the cutting edge has two beveled edges from the tip, then a right angle should be formed at the cutter tip between the two sides.

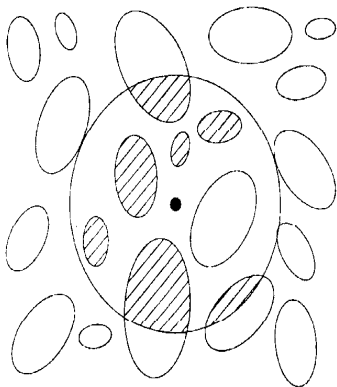


Figure 5. Illustration of proper increment delimitation. Reprinted with permission from Ref. 21. Copyright 1993. CRC Press.

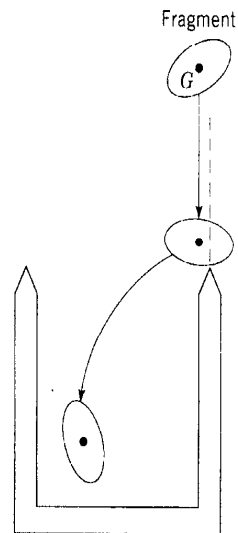


Figure 6. Illustration of the rule of the center of gravity: G - the center of gravity of the fragment or soil particle. Reprinted with permission from Ref. 21. Copyright 1993. CRC Press.

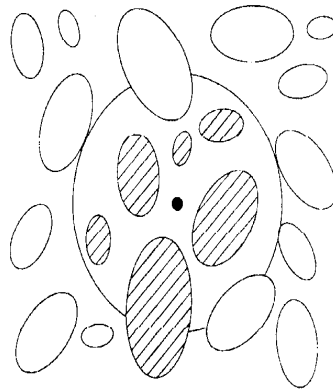


Figure 7. Illustration of proper increment extraction. Reprinted with permission from Ref. 21. Copyright 1993. CRC Press.

- Width of the cutting mouth: For $d \geq 3$ mm, the width of the cutting mouth should be at least $3 \times d$. If $d < 3$ mm, then the width should be at least 10 mm in diameter.

One possible means to minimize the extraction error is to select a larger diameter coring device.

The last two errors, the preparation (PE) and analytical errors (AE), are the result of physical sample manipulation or from noise in the system (21). The major sources of preparation error include contamination, alteration of chemical form (eg, volatilization or redox reactions), human error, and loss of the sample via misplacement or spillage. Preparation errors may be unintentional and occur during sample grinding, sieving, and storage, or through other mistakes such as mislabelling or improper sample handling. Occasionally, intentional error (eg, fraud or sabotage) may be encountered

such that pollutants of concern are intentionally lost during preparation or sampling. Analytical error is the error of which most analysts are cognizant. Unfortunately, it is in reducing, controlling, and quantifying analytical error that most quality assurance/quality control time, effort, and budget is expended. As presented throughout the discussion of particulate sampling theory, without giving proper attention to the actual soil sample collection, the sample received at the analytical laboratory may have already been so severely biased that controlling the analytical error may hardly be worth the time and expense.

Application of Particulate Sampling Theory to Soil Sampling.

One of the keys to Gy's theory is that it provides a means to determine the quantity of soil to collect and submit a representative sample with an acceptable and set relative variance of the fundamental error to the analytical laboratory based on the maximum particle diameter. Additionally, if a large sample is submitted, it provides a comminution (ie, particle-size reduction) protocol to reduce the sample size yet maintain the fundamental error below the acceptable relative variance as defined in the DQO process (22). Of course, this assumes that the sample is collected correctly using all possible means to control and minimize the GE, DE, EE, PE, and AE so that these errors are negligible compared to FE. Two approaches used to determine the required sample mass are to calculate the mass by solving for M_S in the fundamental error equation or to create a sampling nomograph (Fig. 8). Following the calculational pathway, for example, based on the assumptions of an allowable percent relative standard deviation for FE of 15% (or a relative variance of FE of 0.0225); $M_L \gg M_S$; $d = 2$ mm; $f = 0.5$; $g = 0.25$; $\ell = 0.2$; $\lambda = 2.65$ g/cm³; and contaminant concentrations (a_L) = 50, 100, 250, 500, and 1000 ppm, then the required sample weights are approximately 471, 236, 94, 47, and 24 g, respectively. By changing the allowable relative standard deviation of FE to 5% and following the same assumptions, the required sample weights would be 4240, 2120, 848, 424, and 212 g, respectively.

A sampling nomograph also allows the sampler to control sampling and sample processing such that no step exceeds the DQO established for allowable fundamental error. The nomo-

graph provides a visual interpretation of steps that may be necessary to reduce particle and sample size as well as providing the required sample mass for a given maximum particle size. The construction of a nomograph simply involves solving and plotting the equation for the fundamental error. The fundamental error was defined as:

$$s_{FE}^2 = (1/M_S - 1/M_L)fgctd^3$$

This equation can be rearranged to identify a sampling constant, C , by assuming constant values for f , g , c , and ℓ and assuming M_L is much larger (at least 10-fold) than M_S (21). The formula then becomes:

$$s_{FE}^2 = Cd^3/M_S$$

where $C = fgc\ell$. In order to put all the pertinent information on a single nomograph, a logarithmic coordinate system is used and thus the formula becomes:

$$\log s_{FE}^2 = \log C + 3 \log d - \log M_S$$

For a given particle size d and a sampling constant C , the value of $\log s_{FE}^2$ is directly proportional to $-\log M_S$ (21). The derivative of $\log s_{FE}^2$ with respect to M_S equals -1 . Then, the line representing the $\log s_{FE}^2$ as a function of $-\log M_S$ for a given particle size d and a sampling constant C , has a slope of -1 on the nomograph. Therefore, to plot the nomograph for a given d with a constant C , the $\log s_{FE}^2$ is plotted on the y-axis, $\log M_S$ is plotted on the x-axis, and $\log d$ is plotted as a family of parallel lines with a slope of -1 (see Fig. 8).

To identify a minimum required sample size given the largest particle diameter and acceptable relative variance limit, the sampler can follow the diagonal particle size line to the point where it intersects the horizontal variance line. At the point of intersection, the value on the x-axis equals the minimum sample size required to collect a representative sample.

Alternately, if a large sample is collected whose mass must be reduced to a smaller size (eg, to the size required for an extraction procedure), the nomograph provides information on how to reduce the sample mass without exceeding the acceptable DQO relative variance level. To identify the steps

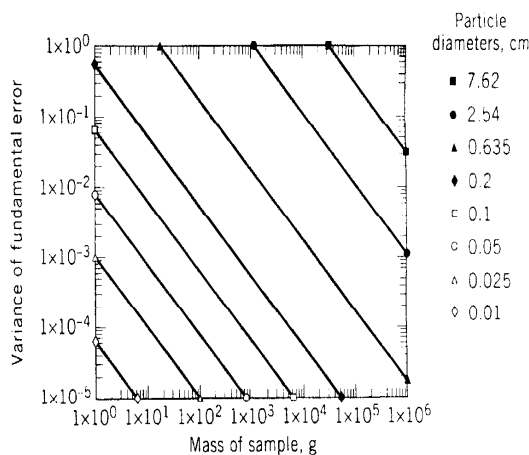


Figure 8. Typical sampling nomograph for soils where $g = 0.25$, $f = 0.5$, $\ell = 0.2$, $\lambda_M = 2.65$ g/cm³, and $a_L = 1000$ μ g/g (ppm). See text.

necessary (if any) to properly reduce the sample mass, the sampler needs to know the acceptable relative variance limit as well as the starting and final sample weights. Following the same process for determining the required sample size, if the intersection of the particle size and variance lines results in a sample size smaller than the final required weight, then no particle size reduction is necessary, and the sample can be collected bearing in mind to minimize the other sampling errors. If not, then some form of particle size reduction (ie, comminution) must be taken. The reduction in particle size can be performed by grinding, correct splitting, or by screening the sample to remove larger particle sizes (22). The removal of the coarsest particle sizes should be based on the assumption that the largest particles do not significantly contribute to the contaminant concentration. To select an appropriate particle reduction scheme, simply follow the maximum particle diameter line from its intersection with the starting weight line until it intersects the acceptable variance line. At this point, a vertical line should be drawn until it intersects a smaller particle diameter line that falls below the variance line for the final required sample size. Once an appropriate smaller particle diameter line has been identified, then the whole sample needs to be reduced to have the coarsest particles equal to or smaller than that selected diameter. Several steps may be required to properly reduce the maximum particle sizes but the key is to not go above the acceptable relative variance limit.

Selection of Sampling Equipment

The selection of sampling equipment should be performed with care since the sampling equipment must be able to collect a representative and correct sample and because the sampling equipment comes in direct contact with the soil. Criteria that should be considered during the selection of the appropriate sampling tool are: chemical and physical compatibility; matrix effects; volume capacity; physical requirements for equipment use; ease of operation; time requirement; decontamination and reuse potential; cost; and soil type to be sampled (15,23,24). Further, the appropriate sampling devices selected must be correct with respect to delimitation and extraction errors previously discussed. Each of these factors will be discussed in greater detail in the following text.

Because the soil comes into direct contact with the sampling tool, the sampling tool must be compatible both chemically and physically with the soil and the contaminants present in the soil (23). Chemical compatibility is a concern primarily when organic contaminants are present. The sampling tool should not be a source of additional sample contamination and should be free from (or at least resistant to) chemical degradation that may occur due to interactions with the samples. For example, plastic or acetate sleeves commonly used as liners in coring equipment should be avoided when sampling for organics because they can bleed phthalates and other organic compounds into the sample as well as degrade due to the solvent effect of some organics that may be present in the contaminated soil. Conversely, certain organic contaminants may partition into the plastic resulting in lower contaminant concentrations and cross contamination, if the plastic sampler is used to obtain another sample. Physical compatibility is a consideration in terms of the physical strength of the sampler to resist deformation when collecting the soil and for the possibility of sample contamination due to physical abrasion of the

sampler with the separated particles becoming part of the analytical sample. For example, the use of stainless steel sampling equipment for sampling chromium-contaminated soils should be avoided because chromium is a major elemental component in stainless steel.

The effect of the sampling device on the soil matrix is an important concern because the design of the device influences the representativeness of the collected sample (23). As previously discussed, if the sampling device excludes a given particle-size fraction (Gy's increment extraction error) or improperly collects the sample by influencing which part of the whole sample is collected (Gy's increment delineation error), then major sources of error enter into the sampling and analytical stream. Further, sample disturbance is a primary consideration when sampling soil for VOCs. An intact core is highly preferred (versus a disaggregated sample obtained from devices such as augers) during collection of VOC-contaminated soils because the minimization of atmospheric exposure of the sample and consequent VOC loss from the sample is imperative.

Most sampling devices provide adequate sample volume (23). However, sampling device volumes should be compared to the volume necessary for all required analyses, quality assurance purposes, and provide sufficient excess sample for archiving and re-analyses purposes. If the device does not provide adequate volume, the following options should be considered: collecting multiple increments/samples in very close proximity to the original location, using a similar device with increased capacity, using an alternate device with increased capacity, or modifying the existing device.

The physical requirements, ease of operation, and time required for use of the sampling equipment all relate to the transport and operation of the selected sampling device (23). Physical requirements to be considered are the device's size and weight because the sampler may have to carry the device to the sampling location and manually collect the sample. If the device is power driven, then the power source and ancillary equipment (eg, drill rigs, trucks, etc) are a concern in selecting these devices. Ease of operation concerns involve the training of personnel to effectively and properly use the sampling device. Fortunately, most manually operated sampling equipment is relatively simple to use, and representative samples can be collected as long as proper care is taken during sample collection. A factor influencing both the ease of operation and time requirement for sampling is the labor requirement. Most power-driven sampling tools require multiple-person teams to effectively and safely use the equipment but have the advantage of shorter sample collection times, especially when depths exceed one meter (39 inches). In contrast, manual sampling tools, although allowing a single sampler to quickly and efficiently collect surface samples, require much longer times to collect samples at depth than power-driven tools.

Decontamination, reuse potential, and cost are concerns when sampling equipment may be used for multiple sampling programs (23). Three aspects of decontamination, and thus the device's reuse potential, to consider are the procedure's ease, success (ie, ability to eliminate or minimize the potential for sample cross-contamination), and time involved. Ideally, the decontamination procedure should be simple, quick, and successful. Most soil sampling equipment can be effectively decontaminated with a simple soap and water wash followed by water rinse. However, where certain organic compounds are of concern, a solvent rinse may also be necessary. (Note: caution

should be taken during the selection and use of solvents to rinse sampling equipment to avoid cross-contamination of the samples, health and safety issues, and spillage such that the solvents become part of the problem at the site.) If successful decontamination procedures are not possible due to the soil matrix or contaminant (eg, soil contaminated with heavy oils or tars), the use of disposable sampling tools should be considered to avoid sample cross-contamination. Cost considerations should be contemplated and compared to the life expectancy of the equipment and use expectancy (ie, will the equipment be used for multiple projects within the organization or should it be leased for a short one-time project).

Several soil types make the routine collection of soil samples difficult and thus require specialized (or modified) sampling tools. These soil types include stony soils, noncohesive soils, and saturated (or nearly saturated) soils (15,23). Sampling difficulties are encountered in stony soils due to the blocking or plugging of the device's opening thus preventing the collection of the sample. Large stones may also prevent the penetration of the sampling device to any significant depth. Noncohesive soils, such as loose sands, require special sampling equipment that retains the soil within the sampler and thus prevents the soil from flowing out the open end of the sampling device. In contrast, saturated soils (eg, mucks and muds) are generally difficult to collect and remove from the sampling tool especially if the soils have high clay contents. Under these circumstances, specialized sampling tools are required that will allow for the simple removal of the soil from the sampling device.

Soil Sampling Tools

Soil sampling tools can be divided into two main categories based on the depth to which the samples can be collected. Surface samplers are designed to generally collect the sample within the upper 30 cm (12 in.) with a single pass (ie, single insertion into the soil) of the sampling device. Subsurface devices are generally designed to sample to a depth of 1.5 m (60 in.) in a single pass but can usually collect deeper samples with multiple passes. Table 1 presents commonly available sampling tools and information on the selection criteria previously discussed. Project-specific parameters, such as compatibility, matrix effects, decontamination requirements, and cost will need to be determined on a per-project basis.

Although spoons, scoops, trowels, and shovels (or backhoes) are perhaps the most readily available and widely used sampling tools for the collection of surface samples, they also tend to introduce the greatest sampling error. The most common cause of the sampling error is the improper increment delimitation in which part of the sample is unintentionally excluded from the sample to be analyzed (21). For example, when using a shovel, rarely is the hole dug such that the sidewalls are parallel to each other and perpendicular to the surface. The natural tendency is to dig a hole with sidewalls at an angle to the surface (Fig. 9, C). If the sample collected from the soil is removed from the hole, the lower portion of the sample is under-represented because less sample is collected at that depth than at the wider surface mouth. Under these circumstances, if the contaminant has been leached to a depth within a given sampling increment, a lower contaminant concentration may be obtained due to sample dilution with the cleaner overlying ma-

terial. Conversely, if the contaminant is present at or near the surface, then the reported concentrations may overestimate the actual concentration due to inclusion of a greater proportion of surface versus subsurface soil. To overcome this problem, it is recommended to dig a hole to the required depth and collect the sample from the walls of the hole being careful to collect a uniform sample thickness from the entire length of the sampling stratum. Alternately, care must be taken to dig the hole properly to include equal proportions of all fractions within the sampling increment (Figs. 9, A and B).

The same general principle is the basis for the error introduced when using spoons, scoops, and trowels. Most of these samplers have a rounded bottom which when used to collect soil preferentially collects less of the lower portion of the sample than the surface and thus biases the results (Fig. 10, A). Additionally, because none of these sampling utensils has sidewalls capable of holding the entire sample within the sampler, as the sample is removed and transported to the sample container, portions of the sample may be lost by falling off the sampler (Fig. 11, A). In some cases, both the upper and lower portion of the sample may be lost with the greatest sampled portion being collected from the middle of the sampling stratum. To properly collect a sample with these types of tools would require equipment modification to have parallel side walls and a flat bottom capable of holding the entire sampling increment (Fig. 10, B and Fig. 11, B).

Tube-type samplers, including probes, punches, corers, tube samplers, barrel samplers, continuous samplers, and zero contamination samplers, tend to have a distinct advantage over other forms of samplers because an intact soil core is collected. The cylindrical shape of the intact core is the correct shape for a two-dimensional lot and, thus, reduces the increment delimitation error as long as care is taken to collect the entire layer or depth defined as the sampling increment (Figs. 9, A and B) (21).

When more sample is collected than required for the sampling increment (eg, a 25-cm (10-in.) core is collected and subsampled to collect the top 10 cm (4 in.)), care must be taken to delineate the proper increment by cutting the new boundary parallel to the surface of the sample at the desired depth. For the thinner diameter tube-type samplers (<2.5 cm ID; 1 in.), the increment extraction error may be a concern due to exclusion larger particles at the mouth of the sampling tube.

Augers, whether used to collect surface or subsurface samples, with the exception of the hollow stem auger, collect samples that are disaggregated by the cutting bit or within the auger threads. This cutting action destroys natural soil structure and makes it difficult to accurately delineate soil horizon boundaries or sampling depths. This disaggregating property of augers also makes them unsuitable for the collection of soils contaminated with VOCs because the sample disturbance allows for the rapid loss of VOCs to the atmosphere. Increment delimitation error may also occur with augers because the lowest portion of the sample (ie, that portion where threads do not overlap and have an upper and lower thread to hold the sample) cannot be collected.

Thief and triers are two types of sampling tools that were not designed for the collection of soils but can be used when necessary. Thiefs are usually pushed into the sample and rotated to open windows and allow the sample to flow into the central chamber/barrel to be collected (23). This type of filling

Table 1. Guide to Sampling Equipment Selection^a

Type of Sampler	Typical Lengths/ ID ^b	Relative Sample Size	Liners	Core Samples	Suitable Soil Types			Manual or Power	Labor
					Cohesive	Moisture	Stony		
<i>Surface Samplers</i>									
Spoons, trowels, and shovels	Var	Var	No	No	All	All	Yes	Manual	Single
Soil punches and sampling tubes	6-10/1-3	Small	Yes	Yes	Coh	All	No	Manual	Single
Probes and tube samplers	6-24/1	Small	No	Yes	Coh	All	No	Manual	Single
Soil corers	2-12/1-3	Small	Yes	Yes	Coh	All	No	Manual	Single
Split core/barrel samplers	6-12/1-2	Small	Yes	Yes	Coh	Int	No	Manual	Single
Screw augers	6-12/1-2	Small	No	No	Coh	Int	No	Manual	Single
Soil recovery augers	8-12/2-3	Large	Yes	Yes	Coh	Int	No	Manual	Single
<i>Subsurface Samplers</i>									
<i>Augers</i>									
Power augers	36-60/2-6	Large	No	No	All	Int	No	Power	Multi
Solid stem flight augers	36-60/2-6	Large	No	No	All	All	Yes	Power	Multi
Hollow stem augers	36-60/2-4	Large	No	Yes	All	All	Yes	Power	Multi
Regular bucket augers	6/2-4	Large	No	No	Coh	Int	No	Manual	Single
Sand bucket augers	6/2-4	Large	No	No	Non	Int	No	Manual	Single
Mud bucket augers	6/2-4	Large	No	No	Coh	Wet	No	Manual	Single
Dutch augers	6/3	Large	No	No	Coh	Wet	No	Manual	Single
Screw augers	12/1-2	Small	No	No	Coh	Int	No	Manual	Single
Post-hole auger	6/2-8	Large	No	No	Coh	Wet	No	Manual	Single
<i>Tube-type samplers</i>									
Shelby/thin wall tube sampler	30/2-4	Large	No	Yes	Coh	Int	No	Both	Multi
Subsoil probes	36-48/1	Small	No	Yes	Coh	All	No	Manual	Single
Split spoon/tube samplers	12-60/2-4	Large	Yes	Yes	Coh	Int	No	Both	Multi
Continuous sampler	48-60/2-5	Large	Yes	Yes	Coh	All	No	Power	Multi
Veilmeyer sampling tubes	48-72/1	Large	No	Yes	Coh	Int	Yes	Manual	Single
Zero contamination sampler	12-24/1	Small	Yes	Yes	Coh	Wet-Int	No	Both	Multi
Ring-lined barrel sampler	24-60/2-4	Large	Yes	Yes	All	Wet-Int	Yes	Both	Multi
<i>Miscellaneous Samplers</i>									
Triers	6-60/1-3	Var	No	No	Coh	Int	No	Manual	Single
Thiefs	6-72/1	Var	No	No	Non	Dry	No	Manual	Single
Backhoes	Var	Large	No	No	All	All	Yes	Power	Multi

^a This table does not purport to be exhaustive. Only the general names of the sampler types are presented. Varying names for the same sampler or same type of sampler with slight modifications are commonly identified in the literature and sales catalogs.

^b Approximate lengths and internal diameters (ID) are presented in inches. To convert inches to meters, multiply by 2.54×10^{-2} .

^c Var = variable depending on length of sampler or size of hole dug; Coh = cohesive; Non = noncohesive; Int = intermediate wetness (ie, not dry nor wet); Multi = two or more persons required to operate effectively.

^d Manual auger lengths given are just for portion of the sampler where the actual sample is held. Most bucket augers have handles and extensions that allow for greater sampling depths with multiple passes.

^e Manually operated versions of these samplers can be operated by one person.

action leads to the concerns about incorrect sample delimitation because: (1) the sample below the windows, although truly a part of the sample, cannot be collected; (2) only those particles that flow easily are collected; (3) particles larger than the window diameter are excluded, thus biasing the collected sample to the finer particle sizes; and (4) the proper cylindrical shape for a two-dimensional lot is not guaranteed. Triers, on the other hand, although nearly capable of collecting the proper sample cylinder, do not have sidewalls to hold the sample once collected. This lack of sidewalls can lead to sample loss during sample removal and transfer operations resulting in potentially biased analytical results.

SAMPLE HANDLING AND PREPARATION

Once the sample has been collected, the next steps that occur prior to preparing subsamples for analysis, are sample handling and preparation. Various concerns and steps are involved which are briefly presented here to remind the sampler of their final responsibilities. These steps include sample compositing, homogenization, preservation, and storage.

Sample compositing is the process of combining several distinct subsamples to create a single sample for analysis (25). The combined subsamples are then homogenized to make up the final sample submitted for analysis. The ad-

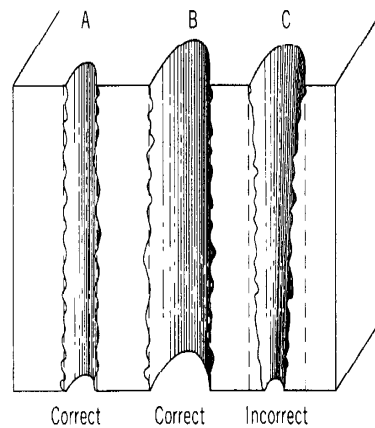


Figure 9. Illustration of correct and incorrect sample delimitation using a coring device (A) and shovels (B and C). Reprinted with permission from Ref. 21. Copyright 1993. CRC Press.

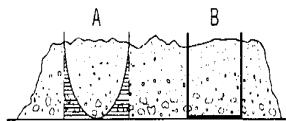


Figure 10. Incorrect increment delimitation introduced by a scoop with round bottom. Reprinted with permission from Ref. 21. Copyright 1993. CRC Press.

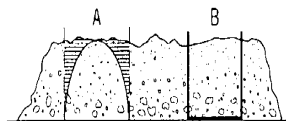


Figure 11. Incorrect increment delimitation introduced by scoop with no side walls to prevent material from falling. Reprinted with permission from Ref. 21. Copyright 1993. CRC Press.

vantages of composite sampling are that it reduces the cost of analysis at a waste site and provides an estimate of the mean concentration. If the sampler follows the principles of Gy's particulate sampling theory, then sample compositing after the collection of numerous small increments is an excellent way to reduce GE. The disadvantages of sample compositing are a loss of information about variation within the sampling area (26), loss of sensitivity because of sample dilution (27), and the time required to homogenize the samples prior to laboratory subsampling. In situations where rapid, field-portable methods are used (eg, field portable X-ray fluorescence spectrometry), compositing may not be necessary because individual samples can be analyzed in real time to provide a more definitive answer concerning the distribution of a contaminant at the waste site.

Numerous techniques have been used to homogenize soils prior to the removal of the analytical subsample (28,29). Some of these techniques include bottle shaking, stirring, sheet mixing or rolling, tumbling, mechanical mixing (eg, cement mixers or V-blenders), riffing, cone-and-quartering, and sectorial splitting. Homogenization is performed to diminish GE within

the sample or lot. When performing sample homogenization, perhaps the most frequently asked question is "When has homogeneity been reached?" Although there is no patented answer to this question, most investigators will homogenize their samples for a fixed time, a fixed number of passes, or until visual homogeneity is reached without ever testing the degree of sample homogeneity. Investigators should always test their homogenization technique prior to full-scale usage especially because some homogenizers will actually lead to resegregation of the sample.

In one experiment to determine the effectiveness (degree of homogeneity) and the efficiency (time consumption) of riffing versus cone-and-quartering as homogenization techniques, it was found (29) that using a closed-bin riffle splitter (which also reduced visual fine particle loss) was more consistently effective and efficient than cone-and-quartering, open-bin riffle splitting, or random sampling, when the sample was passed through the riffle splitter five times. In this experiment, homogenization was performed on bulk soils (approximately 5 kg each with textures ranging from sand through clay) by either pouring the soil collected in both bins back through the riffle splitter, or by forming a new cone as the individual quarters are removed during cone-and-quartering. Although homogenization can help overcome grouping and segregation problems that occur during shipment and handling, properly performed incremental sampling can also produce the same benefits without the added time and expense (22).

Sample preservation for most soil samples is simply to cool the sample to 4°C or colder (30). For soils contaminated with VOCs, preservatives such as methanol or other biocides are commonly used, in conjunction with cooling, to help reduce biological degradation and volatilization of the VOCs. Sample storage in either plastic or glass containers is generally acceptable for soils contaminated with inorganics. In contrast, organically contaminated soils should be stored in glass containers (with Teflon-lined caps for VOC containing soils). It should be noted that some compounds are light-sensitive, and those samples should be stored in the dark to avoid any photochemical reactions.

ACKNOWLEDGMENT

The U.S. Environmental Protection Agency, through its Office of Research and Development (ORD), funded and performed the research described here. It has been subjected to the Agency's peer review and has been approved as an EPA publication. The U.S. government has the right to retain a nonexclusive, royalty-free license in and to any copyright covering this article. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

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