5 OPTIMIZING THE DESIGN FOR OBTAINING THE DATA

This section describes DQO Process Step 7, the last step in the DQO Process. The purpose of this step is to identify an optimal design for obtaining the data. An optimal sampling design is one that obtains the requisite information from the samples for the lowest cost and still satisfies the DQOs.

You can optimize the sampling design by performing five activities that are described in detail in this section. These activities are based on those described in *Guidance for the Data Quality Objectives Process EPA QA/G-4* (USEPA 2000b), but they have been modified to more specifically address RCRA waste-related studies.

In this final planning step, combine the data collection design information with the other outputs of the DQO Process and document the approach in a planning document such as a QAPP, WAP, or similar planning document. As part of this step, it may be necessary to work through Step 7 more than once after revisiting the first six steps of the DQO Process.

Step 7: Optimize the Design for Collecting the Data

Purpose
To identify a resource-effective data collection design for generating data that are expected to satisfy the DQOs.

Activities
- Review the outputs of the first six steps of the DQO Process (see Section 5.1).
- Consider various data collection design options, including sampling and analytical design alternatives (see Section 5.2), and composite sampling options (see Section 5.3).
- For each data collection design alternative, determine the appropriate number of samples (see Section 5.4 or 5.5).
- Select the most resource-effective design that satisfies all of the data needs for the least costs (see Section 5.6).
- Prepare a QAPP, WAP, or similar planning document as needed to satisfy the project and regulatory requirement (see Section 5.7).

5.1 Review the Outputs of the First Six Steps of the DQO Process

Each of the steps in the DQO Process has a series of outputs that include qualitative and quantitative information about the study. The outputs of the first six steps of the DQO Process, as described in Section 4, serve as inputs to DQO Step 7.

Review the existing information and DQO outputs (see Table 5). Determine if any data gaps exist and determine whether filling those gaps is critical to completion of the project. Data gaps can be filled by means of a “preliminary study” or “pilot study.” A preliminary study or pilot can include collection of samples to obtain preliminary estimates of the mean and standard deviation. In addition, a preliminary study can help you verify waste or site conditions, identify unexpected conditions or materials present, gain familiarization with the waste and facility operations, identify how the waste can be accessed, check and document the physical state of the material to be sampled, and identify potential health and safety hazards that may be present.

Review the potential sources of variability and bias (“error”) that might be introduced in the sampling design and measurement processes. See Section 6 for a discussion of sources of error in sampling and analysis.

5.2 Consider Data Collection Design Options

Data collection design incorporates two interdependent activities -- the sample collection design and analytical design.

> **Sampling Design:** In developing a sampling design, you consider various strategies for selecting the locations, times, and components for sampling, and you define appropriate sample support. Examples of sampling designs include simple random, stratified random, systematic, and judgmental sampling. In addition to sampling designs, make sure your organization has documented standard operation procedures (SOPs) that describe the steps to be followed when implementing a sampling activity (e.g., equipment preparation, sample collection, decontamination). For guidance on suggested content and format for SOPs, refer to *Guidance for the Preparing Standard Operating Procedures (SOPs) EPA QA/G-6* (USEPA 2001c). Sampling QA/QC activities also should be part of sampling design. Activities used to document, measure, and control data quality include project-specific quality controls (e.g., duplicate samples, equipment blanks, field blanks, and trip blanks) and the associated quality assessments (e.g., audits, reviews) and assurances (e.g., corrective actions, reports to management). These activities typically are documented in the QAPP (see Section 5.7 and USEPA 1998a).
>
> **Analytical Design:** In DQO Steps 3 and 5, an Action Level and candidate analytical methods were identified. The information should be used to develop analytical options in terms of cost, method performance, available turnaround times, and QA/QC requirements. The analytical options can be used as the basis for designing a performance-based cost-effective analytical plan (e.g., deciding between lower-cost field analytical methods and/or higher cost laboratory methods). Candidate laboratories should have adequate SOPs that describe the steps to be followed when implementing an analytical activity (e.g., sample receipt procedures, subsampling, sample preparation, cleanup, instrumental analysis, data generation and handling). If field analytical techniques are used, hard copies of the analytical methods or SOPs should be available in the field. Refer to Chapter Two of SW-846 for guidance on the selection of analytical methods.

The goal of this step is to find cost-effective design alternatives that balance the number of samples and the measurement performance, given the feasible choices for sample designs and measurement methods.

Sampling design is the “where, when, and how” component of the planning process. In the context of waste sampling under RCRA, there are two categories of sampling designs: (1) **probability** sampling and (2) **authoritative** (nonprobability) sampling. The choice of a sampling design should be made after consideration of the DQOs and the regulatory requirements.

Probability sampling refers to sampling designs in which all parts of the waste or media under study have a known probability of being included in the sample. In cases in which all parts of the waste or media are not accessible for sampling, the situation should be documented so its potential impacts can be addressed in the assessment phase. Probability samples can be of various types, but in some way, they all make use of randomization, which allows probability statements to be made about the quality of estimates derived from the resultant data.

Probability sampling designs provide the ability to reliably estimate variability, the reproducibility of the study (within limits), and the ability to make valid statistical inferences. Five types of probability sampling designs are described in Sections 5.2.1 through 5.2.5:

- Simple random sampling
- Stratified random sampling
- Systematic sampling
- Ranked set sampling
- Sequential sampling.

A strategy that can be used to improve the precision (reproducibility) of most sampling designs is **composite** sampling. Composite sampling is not a sampling design in and of itself, rather composite sampling is a *strategy* used as part of a probability sampling design or an authoritative sampling design. Composite sampling is discussed in Section 5.3.

One common misconception of probability sampling procedures is that these procedures preclude the use of important prior information. Indeed, just the opposite is true. An efficient sampling design is one that uses all available prior information to help design the study. Information obtained during DQO Step 3 (“Identify Inputs to the Decision”) and DQO Step 4 (“Define the Study Boundaries”) should prove useful at this stage. One of the activities suggested in DQO Step 4 is to segregate the waste stream or media into less heterogeneous subpopulations as a means of segregating variability. To determine if this activity is appropriate, it is critical to have an understanding of the various kinds of heterogeneity the constituent of concern exhibits within the waste or media (Pitard 1993). Making assumptions that a waste stream is homogeneous can result in serious sampling errors. In fact, some authors suggest the word “homogeneous” be removed from our sampling vocabulary (Pitard 1993, Myers 1997).

Sampling Over Time or Space?

An important feature of probability sampling designs is that they can be applied along a line of time or in space (see Figure 13) or both (Gilbert 1987):

Time
Sampling designs applied over time can be described by a *one-dimensional* model that corresponds to flowing streams such as the following:

- Solid materials on a conveyor belt
- A liquid stream, pulp, or slurry moving in a pipe or from a discharge point (e.g., from the point of waste generation)
- Continuous elongated piles (Pitard 1993).

Space
For practical reasons, sampling of material over a *three-dimensional* space is best addressed as though the material consists of a series of overlapping *two-dimensional* planes of more-or-less uniform thickness (Pitard 1993, Gy 1998). This is the case for obtaining samples from units such as the following:

- Drums, tanks, or impoundments containing single or multi-phasic liquid wastes
- Roll-off bins, relatively flat piles, or other storage units
- Landfills, soil at a land treatment unit, or a SWMU.

Table 6 provides a summary of sampling designs discussed in this guidance along with conditions for their use, their advantages, and their disadvantages. Figure 13 provides a graphical representation of the probability sampling designs described in this guidance. A number of other sampling designs are available that might perform better for your particular situation. Examples include cluster sampling and double sampling. If an alternative sampling design is required, review other publications such as Cochran (1977), Gilbert (1987), USEPA (2000c) and consult a professional statistician.

Table 6. Guidance for Selection of Sampling Designs

Sampling Design	*Appropriate Conditions for Use*	*Advantages*	*Limitations*
Probability Sampling			
Simple Random Sampling (Section 5.2.1)	Useful when the population of interest is relatively homogeneous (i.e., there are no major patterns or "hot spots" expected).	• Provides statistically unbiased estimates of the mean, proportions, and the variability. • Easy to understand and implement.	• Least preferred if patterns or trends are known to exist and are identifiable. • Localized clustering of sample points can occur by random chance.
Stratified Random Sampling (Section 5.2.2)	Most useful for estimating a parameter (e.g., the mean) of wastes exhibiting high heterogeneity (e.g., there are distinct portions or components of the waste with high and low constituent concentrations or characteristics).	• Ensures more uniform coverage of the entire target population. • Potential for achieving greater precision in estimates of the mean and variance. • May reduce costs over simple random and systematic sampling designs because fewer samples may be required. • Enables computation of reliable estimates for population subgroups of special interest.	• Requires some prior knowledge of the waste or media to define strata and to obtain a more precise estimate of the mean. • Statistical procedures for calculating the number of samples, the mean, and the variance are more complicated than for simple random sampling.
Systematic Sampling (Section 5.2.3)	Useful for estimating spatial patterns or trends over time.	• Preferred over simple random when sample locations are random within each systematic block or interval. • Practical and easy method for designating sample locations. • Ensures uniform coverage of site, unit, or process. • May be lower cost than simple random sampling because it is easier to implement.	• May be misleading if the sampling interval is aligned with the pattern of contamination, which could happen inadvertently if there is inadequate prior knowledge of the pattern of contamination. • Not truly random, but can be modified through use of the "random within blocks" design.

Table 6. Guidance for Selection of Sampling Designs (Continued)

Sampling Design	***Appropriate Conditions for Use***	***Advantages***	***Limitations***
Probability Sampling (continued)			
Ranked Set Sampling (Section 5.2.4)	• Useful for reducing the number of samples required. • Useful when the cost of analysis is much greater than the cost of collecting samples. • Inexpensive auxiliary variable (based on expert knowledge or measurement) is needed and can be used to rank randomly selected population units with respect to the variable of interest. • Useful if the ranking method has a strong relationship with accurate measurements.	• Can reduce analytical costs.	• Requires expert knowledge of waste or process or use of auxiliary quantitative measurements to rank population units.
Sequential Sampling (Section 5.2.5)	• Applicable when sampling and/or analysis are quite expensive, when information concerning sampling and/or measurement variability is lacking, when the waste and site characteristics of interest are stable over the time frame of the sampling effort, or when the objective of the sampling effort is to test a specific hypothesis. • May not be especially useful if multiple waste characteristics are of interest or if rapid decision making is necessary.	• Can reduce the number of samples required to make a decision. • Allows a decision to be made with less sampling if there is a large difference between the two populations or between the true value of the parameter of interest and the standard.	• If the concentration of the constituent of concern is only marginally different from the action level, sequential procedures will require an increasing number of samples approaching that required for other designs such as simple random or systematic sampling.

Table 6. Guidance for Selection of Sampling Designs (Continued)

Sampling Design	*Appropriate Conditions for Use*	*Advantages*	*Limitations*
Authoritative Sampling			
Judgmental (Section 5.2.6.1)	• Useful for generating rough estimates of the average concentration or typical property. • To obtain preliminary information about a waste stream or site to facilitate planning or to gain familiarity with the waste matrix for analytical purposes. • To assess the usefulness of samples drawn from a small portion of the waste or site. • To screen samples in the field to identify "hot" samples for subsequent analysis in a laboratory.	• Can be very efficient with sufficient knowledge of the site or waste generation process. • Easy to do and explain.	• The utility of the sampling design is highly dependent on expert knowledge of waste. • Nonprobability-based so inference to the general population is difficult. • Cannot determine reliable estimates of variability.
Biased (Section 5.2.6.2)	• Useful to estimate "worst-case" or "best-case" conditions (e.g., to identify the composition of a leak, spill, or waste of unknown composition).		

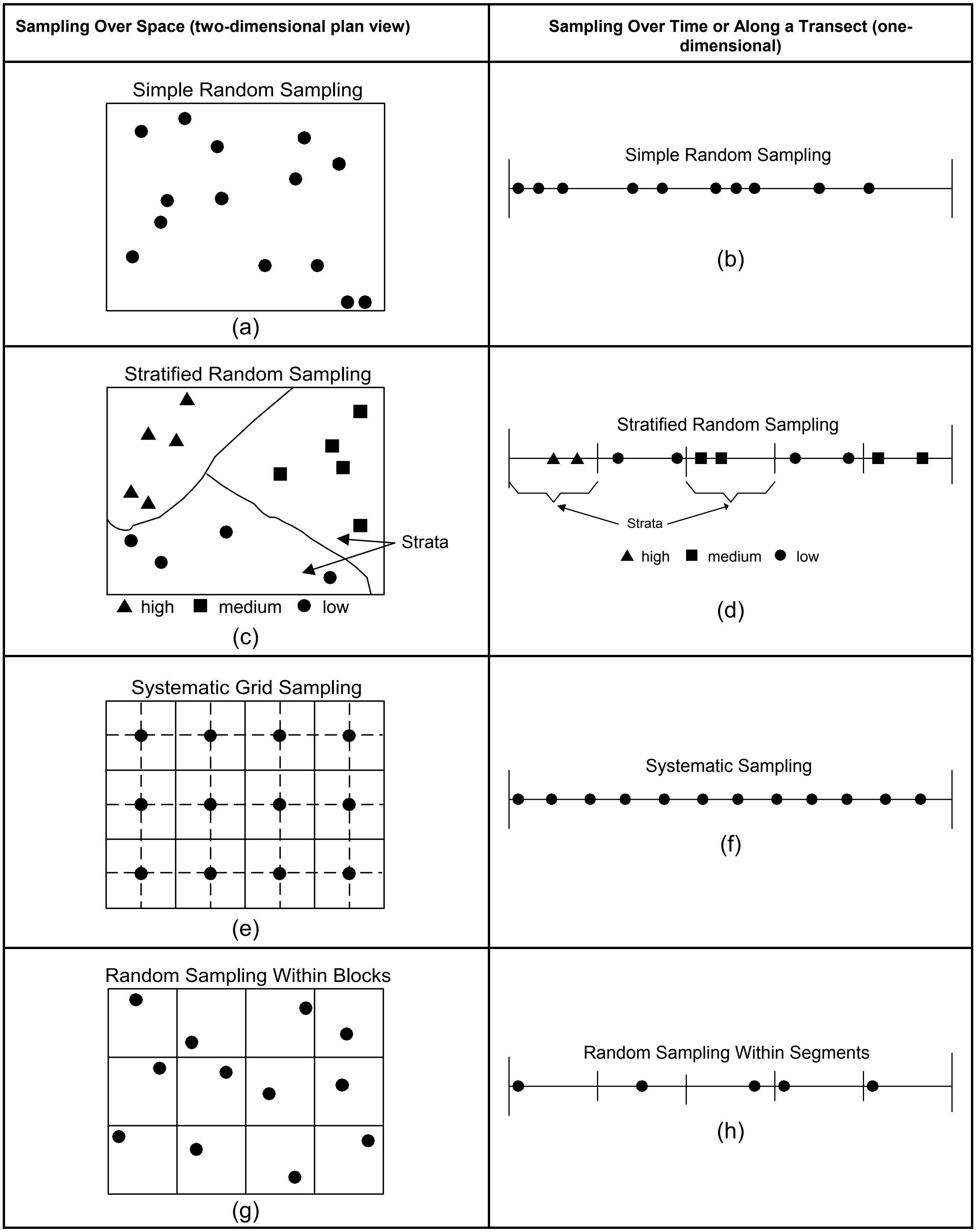

Figure 13. Probability sampling designs over space or along an interval (modified after Cochran 1977 and Gilbert 1987)

5.2.1 Simple Random Sampling

The simplest type of probability sampling is simple random sampling (without replacement), in which every possible sampling unit in the target population has an equal chance of being selected. Simple random samples, like the other samples, can be either samples in space (Figure 13(a)) or in time (Figure 13(b)) and are often appropriate at an early stage of an investigation in which little is known about nonrandom variation within the waste generation process or the site. All of the sampling units should have equal volume or mass, and ideally be of the same shape and orientation if applicable (i.e., they should have the same "sample support").

Box 3. Simple Random Sampling: Procedure

1. Divide the area of the study into N equal-size grids, intervals (if sampling over time), or other units. The spacing between adjacent sampling locations should be established in the DQOs, but the length should be measurable in the field with reasonable accuracy. The total number of possible sampling locations (N) should be much larger than n (the number of samples to be collected).*
2. Assign a series of consecutive numbers to each location between 1 and N.
3. Draw n integers between 1 and N from a random number table or use the random number function on a hand-held calculator (i.e., generate a random number between 0 and 1 and multiply the number by N).
4. Collect samples at each of the n locations or intervals.

* For additional guidance on calculating spacing between sampling locations, see *Methods for Evaluating the Attainment of Cleanup Standards, Volume I: Soil and Solid Media* (USEPA 1989a).

With a simple random sample, the term "random" should not be interpreted to mean haphazard; rather, it has the explicit meaning of equiprobable selection. Simple random samples are generally developed through use of a random number table (found in many statistical text books), a random number function on a hand-held calculator, or by a computer.

One possible disadvantage of pure random sampling is that localized clustering of sample points can occur. If this occurs, one option is to select a new random time or location for the sample. Spatial or temporal biases could result if unknown trends, patterns, or correlations are present. In such situations, stratified random sampling or systematic sampling are better options.

5.2.2 Stratified Random Sampling

In stratified random sampling, a heterogeneous unit, site, or process is divided into nonoverlapping groups called **strata**. Each stratum should be defined so that internally it is relatively homogeneous (that is, the variability within each stratum is less than the variability observed over the entire population) (Gilbert 1987). After each stratum is defined, then simple random sampling is used within each stratum (see Figure 13(c) and 15(d)). For very heterogeneous wastes, stratified random sampling can be used to obtain a more efficient estimate of the parameter of interest (such as the mean) than can be obtained from simple random sampling.

It is important to note that stratified random sampling, as described in this guidance, can be used when the objective is to make a decision about the *whole* population or decision unit. If the objective is to determine of a solid waste is a hazardous waste or to measure attainment of a treatment standard for a hazardous waste, then any obvious "hot spots" or high concentration wastes should be characterized separately from low concentration wastes to minimize mixing of hazardous waste with nonhazardous wastes and to prevent impermissible dilution (see also Appendix C). If the objective of the sampling effort is to identify nonrandom spatial patterns (for example, to create a map of contamination in shallow soils), then consider the use of a geostatistical technique to evaluate the site.

Box 4. Stratified Random Sampling: Procedure

1. Use prior knowledge of the waste stream or site to divide the target population into L nonoverlapping strata such that the variability within stratum is less than the variability of the entire population (for example, see Figure 13c and Figure 13d). The strata can represent area, volume, mass, or time intervals.
2. Assign a weight W_h to each hth stratum. The value of each W_h should be determined based on its relative importance to the data user, or it can be the proportion of the volume, mass, or area of the waste that is in stratum h.
3. Conduct random sampling within each stratum.

In stratified random sampling it is usually necessary to incorporate prior knowledge and professional judgment into a probabilistic sampling design. Generally, wastes or units that are “alike” or anticipated to be “alike” are placed together in the same stratum. Units that are contiguous in space (e.g., similar depths) or time are often grouped together into the same stratum, but characteristics other than spatial or temporal proximity can be employed. For example, you could stratify a waste based on particle size (such that relatively large pieces of contaminated debris are assigned to one stratum and unconsolidated fines assigned to a separate stratum). This is called *stratification by component*. See Appendix C of this guidance for additional information on stratification, especially as a strategy for sampling heterogeneous wastes, such as debris.

In stratified random sampling a decision must be made regarding the allocation of samples among strata. When chemical variation within each stratum is known, samples can be allocated among strata using *optimum allocation* in which more samples are allocated to strata that are large, more variable internally, or cheaper to sample (Cochran 1977, Gilbert 1987). An alternative is to use *proportional allocation*. In proportional allocation, the sampling effort in each stratum is directly proportional to the size (for example, the mass) of the stratum. See Section 5.4.2 for guidance on determining optimum and proportional allocation of samples to strata.

There are several advantages to stratified random sampling. Stratified random sampling:

- Ensures more uniform coverage of the entire target population
- Ensures that subareas that contribute to overall variability are included in the sample
- Achieves greater precision in certain estimation problems
- Generally will be more cost-effective than simple random sampling even when imperfect information is used to form the strata.

There are also some disadvantages to stratified random sampling. Stratified random sampling is slightly more difficult to implement in the field and statistical calculations for stratified sampling are more complex than for simple random sampling (e.g., due to the use of weighting factors and more complex equations for the appropriate number of samples).

5.2.3 Systematic Sampling

Systematic sampling entails taking samples at a preset interval of time or in space and using a randomly selected time or location as the first sampling point (Gilbert 1987).

Systematic sampling over space involves establishing a two-dimensional grid of the unit or waste under investigation (Figure 13(e)). The orientation of the grid is sometimes chosen randomly and various types of systematic samples are possible. For example, points may be arranged in a pattern of squares (rectangular grid sampling) or a pattern of equilateral triangles (triangular grid sampling). The result of either approach is a simple pattern of equally spaced points at which sampling is to be performed. As shown in Figure 13(f), systematic sampling also can be conducted along a transect (every five feet, for example), along time intervals (every hour, for example), or by flow or batches (every 10,000 gallons, for example) (King 1993).

The systematic sampling approach is attractive because it can be easily implemented in the field, but it has some limitations such as not being truly random. You can improve on this sampling design by using random sampling within each grid block (Figure 13(g)) or within each time interval (Figure 13(h)). This approach maintains the condition of equiprobability during the sampling event (Myers 1997) and can be considered a form of *stratified random sampling* in which each of the boundaries of the strata are arbitrarily defined (rather than using prior information) and only one random sample is taken per stratum (Gilbert 1987). This approach is advantageous because it avoids potential problems caused by cycles or trends.

Box 5: Systematic Sampling: Procedure

Sampling Over Space

1. Determine the size of the area to be sampled.
2. Denote the surface area of the sample area by A.
3. Assuming a square grid is used, calculate the length of spacing between grid nodes (L)

$$L = \sqrt{\frac{A}{n}}$$

   where n is the number of samples. The distance L should be rounded to the nearest unit that can be easily measured in the field.
4. To determine the sampling locations, randomly select an initial sampling point within the area to be sampled. Using this location as one intersection of two gridlines, construct gridlines parallel to the original grid and separated by distance L.
5. Collect samples *at* each grid node (line intersection) (see Figure 13e). Alternatively, randomly select a sampling point *within* each grid block (see Figure 13g).

Sampling Along a Line (e.g., Over Time)

1. Determine the start time and point and the total length of time (N) over which the samples will be collected.
2. Decide how many samples (n) will be collected over the sampling period.
3. Calculate a sampling interval where $k = \frac{N}{n}$.
4. Randomly select a start time and collect a sample every kth interval until n samples have been obtained (see Figure 13f). Alternatively, randomly select a sampling point *within* each interval (Figure 13h).

Systematic sampling also is preferred when one of the objectives is to locate “hot spots” within a site or otherwise map the pattern of concentrations over an area (e.g., using geostatistical techniques). Even without using geostatistical methods, “hot spots” or other patterns could be identified by using a systematic design (see “ELIPGRID” software in Appendix H and Gilbert 1987, page 119). On the other hand, the systematic sampling design should be used with caution whenever there is a possibility of some type of cyclical pattern in the waste unit or process that might match the sampling frequency, especially processes being measured over time (such as discharges from a pipe or material on a conveyor).

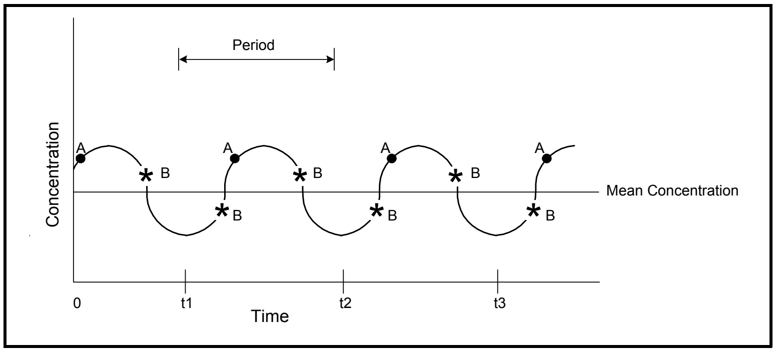

Figure 14. Potential pitfall of systematic sampling over time: cyclic trend combined with a systematic sampling design (after Cochran 1977 and Gilbert 1987)

Figure 14 illustrates the potential disadvantage of using systematic sampling when cyclic trends are present. When there is a cyclic trend in a waste generation process, using a uniform pattern of sampling points can result in samples with very unusual properties. The sets of points labeled “A” and “B” are systematic samples for which the sampling intervals are one period and one-half period, respectively. The points labeled “A” would result in a biased estimate of the mean but a sampling variance of zero. The points labeled “B” would result in an unbiased estimate of the mean with very small variance, even a zero variance if the starting point happened to be aligned exactly with the mean.

5.2.4 Ranked Set Sampling

Ranked set sampling (RSS) (McIntyre 1952) can create a set of samples that at a minimum is equivalent to a simple random sample, but can be as much as two to three times more efficient than simple random sampling. This is because RSS uses the availability of expert knowledge or an inexpensive surrogate measurement or auxiliary variable that is correlated with the more expensive measurement of interest. The auxiliary variable can be a qualitative measure, such as visual inspection for color or an inexpensive quantitative (or semi-quantitative) measure that can be obtained from a field instrument such as a photoionization detector for volatile organics or an X-ray fluorescence analyzer for elemental analysis. RSS exploits this correlation to obtain a sample that is more representative of the population than would be obtained by random sampling, thereby leading to more precise estimates of the population parameters than random sampling. RSS is similar to other probabilistic sampling designs such as simple random sampling in that sampling points are identified and samples are collected. In RSS, however, only a subset of the samples are selected for analysis.

RSS consists of creating m groups, each of size m (for a total of “m x m” initial samples), then ranking the surrogate from largest to smallest within each group. One sample from each group is then selected according to a specified procedure and these m samples are analyzed for the more expensive measurement of interest (see Box 6 and Figure 15).

The true mean concentration of the characteristic of interest is estimated by the arithmetic sample mean of the measured samples (e.g., by Equation 1). The population variance and standard deviation also are estimated by the traditional equations (e.g., by Equations 2 and 3). For additional information on RSS, see USEPA 1995b, USEPA 2000c, and ASTM D 6582 *Standard Guide for Ranked Set Sampling: Efficient Estimation of a Mean Concentration in Environmental Sampling*.

Box 6. Ranked Set Sampling: Procedure

1. Identify some auxiliary characteristic by which samples can be ranked in order from lowest to highest (e.g., by use of a low-cost field screening method).
2. Randomly select $m \times m$ samples from the population (e.g., by using simple random sampling).
3. Arrange these samples into m sets of size m .
4. Within each set, rank the samples by using only the auxiliary information on the samples.
5. Select the samples to be analyzed as follows (see Figure 17):
   - In Set 1, select the sample with rank 1
   - In Set 2, select the sample with rank 2, etc ...
   - In Set m , select the unit with rank m .
6. Repeat Steps 1 through 5 for r cycles to obtain a total of $n = m \cdot r$ samples for analysis.

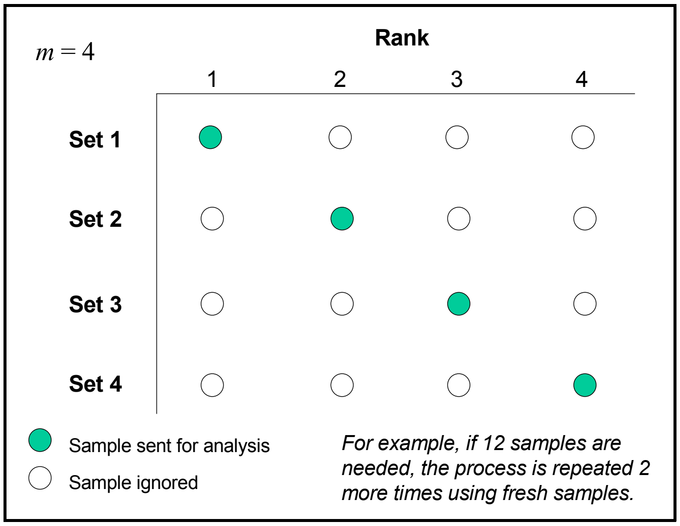

Figure 15. Ranked set sampling. After the samples are ranked in order from lowest to highest, a sample is selected for analysis from Set 1 with Rank 1, from Set 2 with Rank 2, etc.

5.2.5 Sequential Sampling

In sequential testing procedures (Wald 1973), sampling is performed by analyzing one (or more) sample(s) at a time until enough data have been collected to meet the statistical confidence level that the material does not exceed the critical level. The expected sample size, using this sequential procedure, can be approximately 30- to 60-percent lower than a corresponding fixed sample size test with the same power. The sequential procedure is especially helpful in situations in which the contamination is very high or very low relative to the action level. In these situations, the sequential procedure will quickly accumulate enough evidence to conclude that the waste or site either meets or fails to meet the standard.

Figure 16 shows how the procedure operates in a simple example for determining the mean concentration of a constituent of concern in soil. This particular example involves clean closure of a waste management unit, however, the approach could be used for other situations in which the mean is the parameter of interest. The procedure consists of analyzing groups of samples and calculating the mean and 80-percent confidence interval (or upper 90-percent confidence limit) for the mean after analysis of each group of samples. The horizontal axis represents the number of sample units evaluated. The vertical axis represents the concentration of the contaminant; plotted are the mean and 80-percent confidence interval after analysis of n samples. The AL, against which the sample is to be judged, is shown as a horizontal line.

The sampled units are analyzed first in a small lot (e.g., five samples). After each evaluation the mean and confidence interval on the mean are determined (point “a”). If the 90-percent UCL on the mean value stays above the critical value, AL, after successive increments are analyzed, the soil in the unit cannot be judged to attain the action level (point “b”). If the UCL goes below the critical value line, it may be concluded that the soil attains the standard. In the figure, the total number of samples is successively increased until the 90-percent UCL falls below the critical level (points “c” and “d”).

A sequential sampling approach also can be used to test a percentile against a standard. A detailed description of this method is given in Chapter 8 of *Methods for Evaluating the Attainment of Cleanup Standards Volume 1: Soil and Solid Media* (USEPA 1989a).

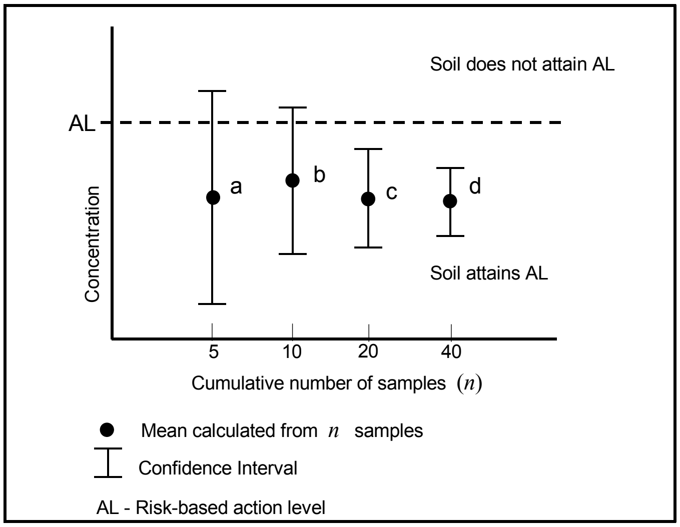

Figure 16. Example of sequential testing for determining if concentrations of a constituent of concern in soil at a closed waste management unit are below a risk-based action level (AL).

In sequential sampling, the number of samples is not fixed *a priori*; rather, a statistical test is performed after each analysis to arrive at one of three possible decisions: reject the hypothesis, accept the hypothesis, or perform another analysis. This strategy is applicable when sampling and/or analyses are quite expensive, when information concerning sampling and/or measurement variability is lacking, when the waste and site characteristics of interest are stable over the time frame of the sampling effort, or when the objective of the sampling effort is to test a specific hypothesis. It may not be especially useful if multiple waste characteristics are of interest or if rapid decision making is necessary.

In planning for a sequential sampling program, the following considerations are important:

- Pre-planning the effort between the field and laboratory, including developing a system of pre-planned paperwork and sample containers
- Arranging for a system of rapid delivery of samples to the laboratory
- Providing rapid turnaround in the laboratory
- Rapidly returning data to the planners, supervisors, and others responsible for decision making.

If the sequential sampling program is carried out using field methods (e.g., portable detectors), much of the inconvenience involved with shipping and return of results can be avoided.

5.2.6 Authoritative Sampling

Authoritative sampling is a nonstatistical sampling design because it does not assign an equal probability of being sampled to all portions of the population. This type of sampling should be considered only when the objectives of the investigation do not include the estimation of a population parameter. For example, authoritative sampling might be appropriate when the objective of a study is to identify specific locations of leaks, or when the study is focused solely on the sampling locations themselves. The validity of the data gathered with authoritative sampling is dependent on the knowledge of the sampler and, although valid data sometimes can be obtained, it is not recommended for the chemical characterization of wastes when the parameter of interest (such as the mean) is near the action level.

Authoritative sampling (also known as judgmental sampling, biased sampling, nonprobability sampling, nonstatistical sampling, purposive sampling, or subjective sampling) may be appropriate under circumstances such as the following:

- You need preliminary information about a waste stream or site to facilitate planning or to gain familiarity with the waste matrix for analytical purposes.
- You are conducting sampling for a RCRA Facility Assessment (RFA) to identify a potential or actual release to the environment.
- You have encountered a spill of an unknown chemical and need to determine the chemical makeup of the spilled material.
- You have access to only small portions of the population and judgment is applied to assess the usefulness of samples drawn from the small portion.
- You are screening samples in the field, using an appropriate field method, to identify “hot” samples for subsequent analysis in a laboratory.
- You are sampling to support case development for an enforcement agency or to “prove the positive” (see also Section 2.2.4).

With authoritative sampling, it is not possible to accurately estimate the population variance. Also, due to its subjective nature, the use of authoritative sampling by the regulated community to demonstrate compliance with regulatory standards generally is not advisable except in those cases in which a small volume of waste is in question or where the concentration is either well above or well below the regulatory threshold.

The ASTM recognizes two types of authoritative sampling: judgmental sampling and biased sampling (ASTM D 6311).

5.2.6.1 Judgmental Sampling

Judgmental sampling is a type of authoritative sampling. The goal of judgmental sampling is to use process or site knowledge to choose one or more sampling locations to represent the “average” concentration or “typical” property.

Judgmental sampling designs can be cost-effective *if* the people choosing the sampling locations have sufficient knowledge of the waste. If the people choosing the sampling locations intentionally distort the sampling by a prejudiced selection, or if their knowledge is wanting, judgmental sampling can lead to incorrect and sometimes very costly decisions. Accurate and useful data can be generated from judgmental sampling more easily if the population is relatively homogeneous and the existence of any strata and their boundaries is known.
The disadvantages of judgmental sampling designs follow:

- It can be difficult to demonstrate that prejudice was not employed in sampling location selection

- Variances calculated from judgmental samples may be poor estimates of the actual population variance

- Population statistics cannot be generated from the data due to the lack of randomness.

An example application of judgement sampling is given in Appendix C of *Guidance for the Data Quality Objectives Process for Hazardous Waste Site Operations* (USEPA 2000a).

5.2.6.2 Biased Sampling

Biased sampling is the type of authoritative sampling that intends not to estimate average concentrations or typical properties, but to estimate "worst" or "best" cases (ASTM D 6051-96). The term "biased," as used here, refers to the collection of samples with expected very high or very low concentrations. For example, a sample taken at the source of a release could serve as an estimate of the "worst-case" concentration found in the affected media. This information would be useful in identifying the constituent of concern and estimating the maximum level of contamination likely to be encountered during a cleanup.

At times, it may be helpful to employ a "best case" or both a "best-case" and "worst-case" biased sampling approach. For example, if there is a range of wastes and process knowledge can be used to identify the wastes likely to have the lowest and highest contamination levels, then these two extremes could be sampled to help define the extent of the problem.

Biased sampling, while having the ability to cost-effectively generate information, has similar disadvantages to that of judgmental sampling.

5.3 Composite Sampling

Composite sampling is a strategy in which multiple individual or "grab" samples (from different locations or times) are physically combined and mixed into a single sample so that a physical, rather than a mathematical, averaging takes place.[1] Figure 17 illustrates the concept of composite samples. For a well-formed composite, a single measured value should be similar to the mean of measurements of the individual components of the composite (Fabrizio, et al. 1995). Collection of multiple composite samples can provide improved sampling precision and reduce the total number of analyses required compared to noncomposite sampling. This strategy is sometimes employed to reduce analysis costs when analysis costs are large relative to sampling costs. The appropriateness of using composite sampling will be highly dependent on the DQOs (Myers 1997), the constituent of concern, and the regulatory requirements. *To realize the full benefits of composite sampling, field and laboratory personnel must carefully*

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[1] Some authors use the term "discrete sample" to refer to an individual sample that is used to form a composite sample. The RCRA regulations often use the term "grab sample." For the purpose of this guidance, the terms "discrete," "grab," and "individual" sample have the same meaning.

follow correct procedures for sample collection, mixing, and subsampling (see Sections 6 and 7).

5.3.1 Advantages and Limitations of Composite Sampling

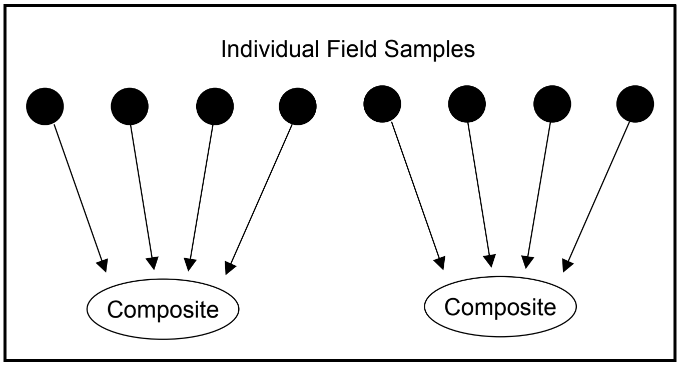

Figure 17. Forming composite samples from individual samples (from USEPA 1995c).

A detailed discussion of the advantages and limitations of composite sampling is presented in the *Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities* (ASTM D 6051-96) and EPA's *Guidance for Choosing a Sampling Design for Environmental Data Collection, EPA QA/G-5S* (USEPA 2000c). Additional information on composite sampling can be found in Edland and van Belle (1994), Gilbert (1987), Garner, et al. (1988 and 1989), Jenkins, et al. (1996 and 1997), Myers (1997), and USEPA (1995c).

Advantages

Three principal advantages to using composite sampling (see ASTM D 6051-96) follow:

- It can improve the precision (i.e., reduce between-sample variance) of the estimate of the mean concentration of a constituent in a waste or media (see Section 5.3.5)
- It can reduce the cost of estimating a mean concentration, especially in cases in which analytical costs greatly exceed sampling costs or in which analytical capacity is limited
- A "local" composite sample, formed from several increments obtained from a localized area, is an effective way to increase the sample support, which reduces grouping and segregation errors (see also Section 6.2.2.2)
- It can be used to determine whether the concentration of a constituent in one or more individual samples used to form a composite might exceed a fixed standard (i.e., is there a "hot spot"?) (see Section 5.3.6).

Limitations

Composite sampling should not be used if the integrity of the individual sample values changes because of the physical mixing of samples (USEPA 1995c). The integrity of individual sample values could be affected by chemical precipitation, exsolvation, or volatilization during the pooling and mixing of samples. For example, volatile constituents can be lost upon mixing of samples or interactions can occur among sample constituents. In the case of volatile constituents, compositing of individual sample extracts within a laboratory environment may be a reasonable alternative to mixing individual samples as they are collected.

Listed below are some additional conditions under which compositing usually is *not* advantageous:

- When regulations *require* the use of discrete or grab samples. For example, compliance with the LDR numeric treatment standards for non-wastewaters typically is to be determined using “grab” samples rather than composite samples. Grab samples processed, analyzed, and evaluated individually normally reflect maximum process variability, and thus reasonably characterize the range of treatment system performance. Typically, grab samples are used to evaluate LDR non-wastewaters and composite samples are used to evaluate LDR wastewaters, except when evaluating wastewaters for metals (D004 through D011) for which grab samples are required [40 CFR 268.40(b)].

- When data users require specific data points to generate high-end estimates or to calculate upper percentiles

- When sampling costs are much greater than analytical costs

- When analytical imprecision outweighs sampling imprecision and population heterogeneity

- When individual samples are incompatible and may react when mixed

- When properties of discrete samples, such as pH or flash point, may change qualitatively upon mixing. (Compositing of individual samples from different locations to be tested for hazardous waste characteristic properties, such as corrosivity, reactivity, ignitability, and toxicity, is not recommended)

- When analytical holding times are too short to allow for analysis of individual samples, *if* testing of individual samples is required later (for example, to identify a “hot” sample) (see Section 5.3.6)

- When the sample matrix impedes correct homogenization and/or subsampling

- When there is a need to evaluate whether the concentrations of different contaminants are correlated in time or space.

5.3.2 Basic Approach To Composite Sampling

The basic approach to composite sampling involves the following steps:

- Identify the boundaries of the waste or unit. The boundaries may be spatial, temporal, or based on different components or strata in the waste (such as battery casings and soil)

- Conduct sampling in accordance with the selected sampling design and collect a set of n x g individual samples where g is the number of individual samples used to form each composite and n is the number of such composites

- Group either randomly or systematically the set of n x g individual samples into n composite samples and thoroughly mix and homogenize each composite sample

- Take one or more subsamples from each composite

- Analyze each subsample for the constituent(s) of concern.

The n composite samples can then be used to estimate the mean and variance (see Section 5.3.5) or identify "hot spots" in the waste (see Section 5.3.6).

5.3.3 Composite Sampling Designs

Composite sampling can be implemented as part of a statistical sampling design, such as simple random sampling and systematic sampling. The choice of a sampling design to use with compositing will depend upon the study objectives.

5.3.3.1 Simple Random Composite Sampling

Figure 18 shows how composite sampling can be integrated into a simple random sampling design. In this figure, the decision unit could represent any waste or media about which a decision must be made (such as a block of contaminated soil at a SWMU). Randomly positioned field samples are randomly grouped together into composite samples. The set of composite samples can then be used to estimate the mean and the variance.

Because the compositing process is a mechanical way of averaging out variabilities in concentrations from location to location over a unit, the resulting concentration data should tend to be more normally distributed than individual samples (Exner, et al. 1985). This is especially advantageous because the assumption of many statistical tests is that the underlying data exhibit an approximately normal distribution.[2]

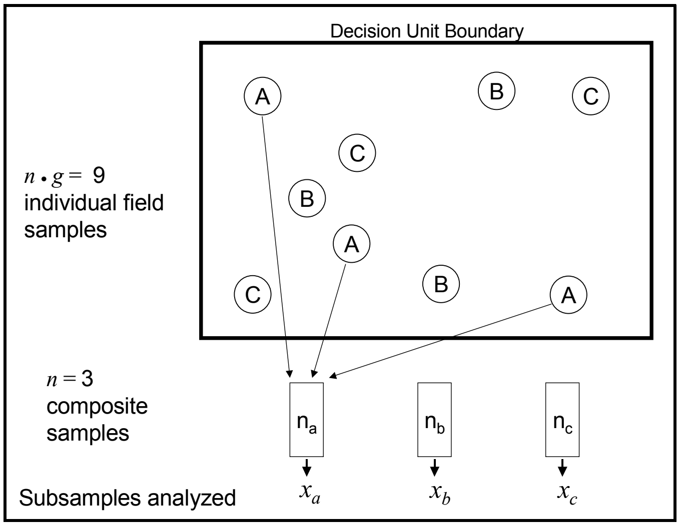

Figure 18. A basic approach to composite sampling. The figure shows how composite sampling can be integrated into a simple random sampling design. Random samples with the same letter are randomly grouped into composite samples to obtain an estimate of the unit-wide mean.

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[2] By the Central Limit Theorem (CLT), we expect composite samples to generate normally distributed data. The CLT states that if a population is repeatedly sampled, the means of all the sampling events will tend to form a normal distribution, regardless of the shape of the underlying distribution.

5.3.3.2 *Systematic Composite Sampling*

A systematic composite sampling design is shown in Figure 19. The design can be used to estimate the mean concentration because each composite sample is formed from field samples obtained across the entire unit. For example, each field sample collected at the "A" locations is pooled and mixed into one composite sample. The process is then repeated for the "B," "C," and "D" locations. The relative location of each individual field sample (such as "A") should be the same within each block.

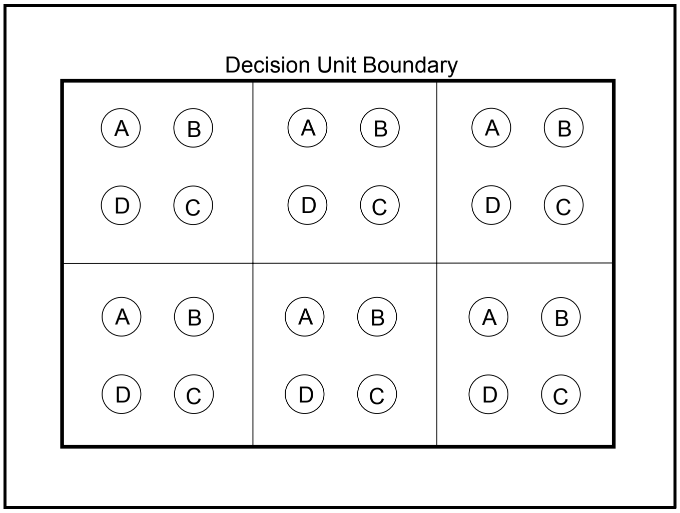

Figure 19. Systematic composite sampling across a unit or site. Samples with the same letter are pooled into composites.

This design is particularly advantageous because it is easy to implement and explain and it provides even coverage of the unit. Exner, et al. (1985) demonstrated how this design was used to make cleanup decisions for blocks of soil contaminated with tetrachlorodibenzo-p-dioxin.

A second type of systematic composite involves collecting and pooling samples from *within* grid blocks, time intervals, or batches of waste grouped together (see Figure 20).

If there is spatial correlation between the grid blocks, compositing within grids can be used to estimate block-to-block variability (Myers 1997) or improve the estimate of the mean within a block or interval (if multiple composite samples are collected within each block). In fact, compositing samples collected from localized areas is an effective means to control "short-range" (small-scale) heterogeneity (Pitard 1993). When this type of compositing is used on localized areas in lieu of "grab" sampling, it is an attractive option to improve representativeness of individual samples (Jenkins, et al. 1996).

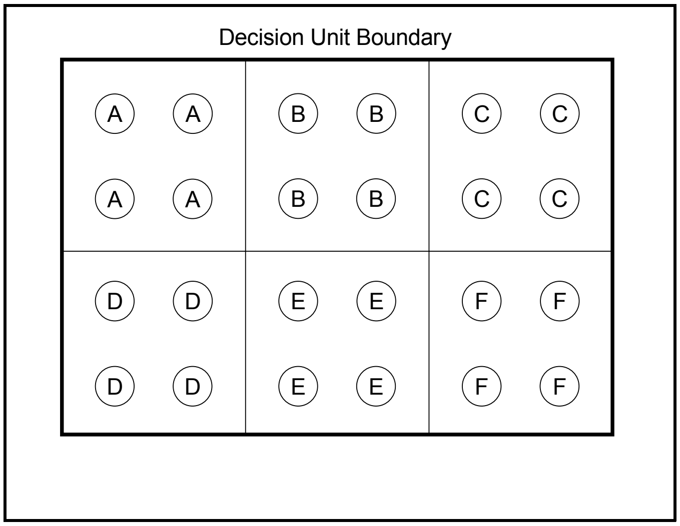

Figure 20. Systematic sampling within grid blocks or intervals. Samples with the same letter are pooled into a composite sample.

Systematic sampling within time intervals could be used in cases in which compositing occurs as part of sample collection (such as sampling of liquid effluent with an autosampling device into a single sample container over a specified time period).

If the individual field sample locations are independent (that is, they have no temporal or spatial correlation), then compositing within blocks can be an efficient strategy for estimating the population mean. If the assumption of sample independence cannot be supported, then an alternative design should be selected if the objective is to estimate the mean.

5.3.4 Practical Considerations for Composite Sampling

In creating composite samples from individual field samples, it is possible that a relatively large volume of material will need to be physically mixed at some point -- either in the field or in the laboratory. Thorough mixing is especially important when the individual samples exhibit a high degree of heterogeneity.

Once the individual samples are mixed, one or more subsamples must be taken because the entire composite sample usually cannot be analyzed directly. A decision must be made as to *where* the individual samples will be combined into the composite samples. Because large samples (e.g., several kilograms or more) may pose increased difficulties to the field team for containerization and shipping and pose storage problems for the laboratory due to limited storage space, there may be a distinct advantage to performing mixing or homogenization in the field. There are, however, some disadvantages to forming the composite samples in the field. As pointed out by Mason (1992), the benefits of homogenization may be temporary because gravity induced segregation can occur during shipment of the samples. Unless homogenization (mixing), particle size reduction, and subsampling are carried out immediately prior to analysis, the benefits of these actions may be lost. Therefore, if practical, it may be best to leave the mixing and subsampling operations to laboratory personnel.

See Section 7.3 of this document and ASTM standards D 6051 and D 6323 for guidance on homogenization, particle size reduction, and subsampling.

5.3.5 Using Composite Sampling To Obtain a More Precise Estimate of the Mean

When analytical error is minor compared to sampling error, then composite sampling can be a resource-efficient mechanism for increasing the precision of estimates of the population mean. If composite sampling is to be used to estimate the mean with a specified level of confidence, then multiple composite samples can be used to estimate the mean and variance. Alternately, confidence limits can be constructed around the sample analysis result for a single composite sample if an estimate of the variance of the fundamental error is available (see Gy 1998, page 73).[3] See Section 6.2.2.1 for a discussion of fundamental error.

The population mean (μ) can be estimated from the analysis of n composite samples (each made from g individual samples). The population mean (μ) is estimated by the sample mean ($\bar{x}$) by

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i \qquad \textbf{Equation 6}$$

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[3] ASTM D 6051, *Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities*, also provides a procedure for estimating the precision of a single composite sample.

The sample variance (s^2) can then be calculated by

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2 \qquad \textbf{Equation 7}$$

Note that Equations 6 and 7 are the same as Equations 1 and 2, respectively, for the mean and variance. When the equations are used for composite sampling, x_i is the measurement value from a subsample taken from each n composite sample rather than each individual sample. Use of these equations assumes equal numbers of individual field samples (g) are used to form each composite, and equal numbers of subsamples are taken from each composite sample and analyzed. If these assumptions are not correct, an alternative approach described in Gilbert (1987, page 79) can be used.

By increasing the number of individual field samples (g) per composite sample, there will be a corresponding decrease in the standard error ($s_{\bar{x}}$), thus improving the precision of the estimate of the mean. Edland and van Belle (1994) show that by doubling the number of individual samples per composite (or laboratory) sample, the expected size of the confidence interval around the mean decreases by a factor of $1/\sqrt{2}$, which is a 29-percent decrease in the expected width of the confidence interval. One of the key assumptions underlying the above discussion is that variances between the samples greatly exceed the random error variance of the analytical method (Garner, et al. 1988).

Williams, et al. (1989) demonstrated the benefits of using composite sampling to obtain a more precise estimate of the mean. One of their objectives was to study the efficiency of using composite sampling as compared to collecting individual samples for the purpose of estimating the mean concentration at a site. Five sites known to have radium contamination in shallow soils were extensively sampled. At each site, shallow soil samples were collected at approximately uniformly spaced points over the entire site. Three types of samples were taken: (1) individual 500-gram samples, (2) composite samples consisting of ten 50-gram aliquots uniformly spaced over the site, and (3) composite samples consisting of twenty 25-gram aliquots uniformly spaced over the site. The samples were measured for ^{226}Ra. The results indicated the individual samples yielded the least precision, even when more than twice as many individual samples were collected. Sixty-six individual samples produced a standard error of 1.35, while the thirty 10-aliquot composites and the thirty 20-aliquot composite samples produced standard errors of 0.76 and 0.51 respectively. The results demonstrate that composite sampling can produce more precise estimates of the mean with fewer analytical samples.

Box 7 provides an example of how a mean and variance can be estimated using composite sampling combined with systematic sampling.

Box 7. Example of How To Estimate the Mean and Variance Using Systematic Composite Sampling (Assume Samples Are Independent)

Under 40 CFR 261.38, a generator of hazardous waste-derived fuel is seeking an exclusion from the definition of solid and hazardous-waste. To prepare the one-time notice under 40 CFR 261.38(c), the generator requires information on the mean and variance of the concentrations of constituents of concern in the waste as generated. The generator elects to use composite samples to estimate the mean and variance of the nonvolatile constituents of concern.

Using a systematic sampling design, a composite sample is prepared by taking an individual (grab) sample at regular time intervals t_1 through t_4. The set of four grab samples are thoroughly mixed to form a composite, and one subsample is taken from each composite for analysis. The process is repeated until five composite samples are formed (see Figure 21). (*Note*: If the assumption of independent samples cannot be supported, then a simple random design should be used in which the 20 grab samples are randomly grouped to form the five composites).

The analytical results for one of the constituents of concern, in ppm, are summarized as follows for the composite samples (n_1 through n_5):
2.75, 3.71, 3.28, 1.95, and 5.10.

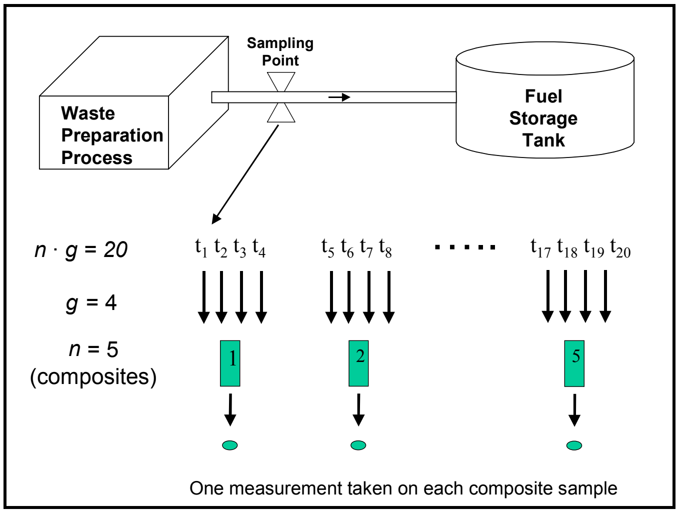

Figure 21. Example of systematic composite sampling

Using Equations 6 and 7 for the mean and variance of composite samples, the following results are obtained:

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i = \frac{16.79}{5} = 3.36\,ppm$$

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2 = \frac{1}{4}\left[0.3721 + 0.1225 + 0.0064 + 1.99 + 3.03\right] = 1.38$$

The standard error is obtained as follows:

$$s_{\bar{x}} = \frac{s}{\sqrt{n}} = \frac{1.17}{\sqrt{5}} = 0.52\,ppm$$

5.3.6 Using Composite Sampling To Locate Extreme Values or "Hot Spots"

One disadvantage of composite sampling is the possibility that one or more of the individual samples making up the composite could be "hot" (exceed a fixed standard), but remain undetected due to dilution that results from the pooling process. If the sampling objective is to determine if any one or more individual samples is "hot," composite sampling can still be used.

A procedure for detecting hot spots using composite sampling is given below. The approach assumes the underlying distribution is normal and the composite samples were formed from equal-sized individual samples.

Let AL be some "action level" or regulatory threshold that cannot be exceeded in an individual sample. Note that AL must be large relative to the quantitation limit for the constituent of concern. For a measurement x_i from a composite sample formed from g individual samples, the following rules apply, assuming analytical and sampling error are negligible:

- If $x_i < \frac{AL}{g}$, then no single individual sample can be $> AL$
- If $x_i > AL$, then at least one *must*, and as many as all individual samples *may*, be $> AL$
- If $x_i > \frac{AL}{g}$, then at least one of the g individual samples must be $> AL$.

As a general rule, we can say that no more than $\frac{g \cdot x_i}{AL}$ individual samples can be $> AL$.

If one or more of the composites are "hot" (i.e., $> AL$), then it might be desirable to go back and analyze the individual samples used to form the composite. Consider saving splits of each individual field sampling so individual samples can be analyzed later, if needed.

If compositing is used to identify a hot spot, then the number of samples that make up the composite should be limited to avoid overall dilution below the analytical limit. It is possible for a composite sample to be diluted to a concentration below the quantitation limit if many of the individual samples have concentrations near zero and a single individual sample has a concentration just above the action level. Mason (1992) and Skalski and Thomas (1984) suggest the maximum number of identically sized individual samples (g) that can be used to form such a composite should not exceed the action level (AL) divided by the quantitation limit (QL). But the relationship of $g \le AL / QL$ indicates that the theoretical maximum number of samples to form a composite can be quite high, especially given a very low quantitation limit. As a practical matter, the number of individual samples used to form a composite should be kept to a minimum (usually between 2 and 10).

An example of the above procedure, provided in Box 8, demonstrates how a "hot" drum can be identified through the analysis of just nine samples (five composites plus four individual analyses), resulting in considerable savings in analytical costs over analysis of individual samples from each of the 20 drums.

Box 8. How To Locate a “Hot Spot” Using Composite Sampling - Hypothetical Example

A secondary lead smelter produces a slag that under some operating conditions exhibits the Toxicity Characteristic (TC) for lead. At the point of generation, a grab sample of the slag is taken as the slag is placed in each drum. A composite sample is formed from the four grab samples representing a set of four drums per pallet. The process is repeated until five composite samples representing five sets of four drums (20 drums total) have been prepared (see Figure 22).

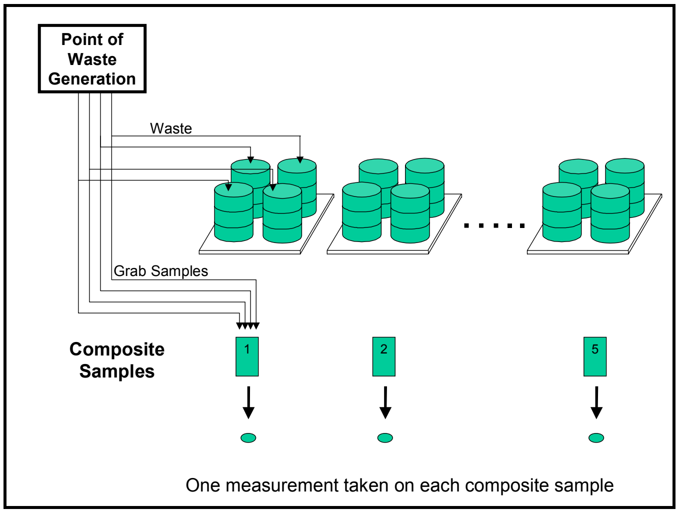

Figure 22. Composite sampling strategy for locating a “hot” drum

The generator needs to know if the waste in any single drum in a given set of four drums contains lead at a total concentration exceeding 100 ppm. If the waste in any single drum exceeds 100 ppm, then its maximum theoretical TCLP leachate concentration could exceed the regulatory limit of 5 mg/L. Waste in drums exceeding 100 ppm total lead will be tested using the TCLP to determine if the total leachable lead equals or exceeds the TC regulatory limit.

The sample analysis results for total lead are measured as follows (in ppm) in composite samples n_1 through n_5:
6, 9, 18, 20, and 45.

Using the approach for locating a “hot spot” in a composite sample, we observe that all of the composite samples except for n_5 are less than AL / g or 100 ppm/4 (i.e., 25 ppm). The result for n_5 (45 ppm) is greater than 25 ppm, indicating a potential exceedance of the TC regulatory level. A decision about the set of drums represented by n_5 can be made as follows:

No more than $\frac{g \cdot x_i}{AL}$ individual samples can be $> AL$, or no more than $\frac{(4)45\,ppm}{100\,ppm} = 1.8$ or 1 (round *down*) individual sample exceeds 100 ppm total lead.

We now know that it is possible that one of the four drums on the fifth palette exceeds 100 ppm, but we do not know *which* one. As a practical matter, analysis of all four of the individual samples should reveal the identity of the “hot” drum (if, indeed, one exists); however, the above process of elimination could be repeated on two new composite samples formed from samples taken from just the four drums in question.

5.4 Determining the Appropriate Number of Samples Needed To Estimate the Mean

This section provides guidance for determining the appropriate number of samples (n) needed to estimate the mean. The procedures can be used when the objective is to calculate a confidence limit on the mean. If the objective is to estimate a percentile, see Section 5.5.

To calculate the appropriate number of samples, it is necessary to assemble existing data identified in DQO Step 3 (“Identify Inputs to the Decision”) and Step 6 (“Specify Limits on Decision Errors”). If the parameter of interest is the mean, you can calculate n using equations presented in the following sections or by using EPA’s DEFT software (USEPA 2001a).

Alternative equations can be found in the statistical literature and guidance, including ASTM (Standard D 6311), Cochran (1977), Gilbert (1987), and USEPA (2000a, 2000b, and 2000d).

The equations presented here should yield the approximate minimum number of samples needed to estimate the mean within the precision and confidence levels established in the DQO Process; however, *it is prudent to collect a somewhat greater number of samples than indicated by the equations*.[4] This is recommended to protect against poor preliminary estimates of the mean and standard deviation, which could result in an underestimate of the appropriate number of samples to collect. For analytes with long holding times (e.g., 6 months), it may be possible to process and store extra samples appropriately until analysis of the initially identified samples is completed and it can be determined if analysis of the additional samples is warranted.

It is important to note that the sample size equations do not account for the number or type of control samples (or quality assessment samples) required to support the QC program associated with your project. Control samples may include blanks (e.g., trip, equipment, and laboratory), field duplicates, spikes, and other samples used throughout the data collection process. Refer to Chapter One of SW-846 for recommendations on the type and number of control samples needed to support your project. It is best to first determine how each type of control sample is to be used, then to determine the number of that type based on their use (van Ee, et al. 1990).

A key assumption for use of the sample size equations is that you have some prior estimate of the total study error, measured as the sample standard deviation (s) or sample variance (s^2). Since total study error includes variability associated with the sampling and measurement methods (see Section 6), it is important to understand the relative contributions that sampling and analysis activities make to the overall estimate of variability. Lack of prior information regarding population and measurement variability is one of the most frequently encountered difficulties in sampling. It quickly resembles a "chicken-and-the-egg" question for investigators – you need an estimate of the standard deviation to calculate how many samples you need, yet you cannot derive that estimate without any samples. To resolve this seemingly paradoxical question, two options are available:

Option 1. *Conduct a pilot study*. A pilot study (sometimes called an exploratory or preliminary study) is the preferred method for obtaining estimates of the mean and standard deviation, as well as other relevant information. The pilot study is simply phase one of a multi-phase sampling effort (Barth, et al. 1989). For some pilot studies, a relatively small number of samples (e.g., four or five or more) may provide a suitable *preliminary estimate* of the standard deviation.

Option 2. *Use data from a study of a similar site or waste stream*. In some cases, you might be able to use sampling and analysis data from another facility or similar operation that generates the same waste stream and uses the same process.

If neither of the above options can provide a suitable estimate of the standard deviation (s), a crude approximation of s still can be obtained using the following approach adopted from

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[4] One exception is when sequential sampling is used in which the number of samples is not fixed *a priori*; rather, the statistical test is performed after each round of sampling and analysis (see Section 5.2.5).

USEPA 1989a (page 6-6). The approximation is based on the *judgment* of a person knowledgeable of the waste and his or her *estimate* of the range within which constituent concentrations are likely to fall. Given a range of constituent concentrations in a waste, but lacking the individual data points, an *approximate* value for s may be computed by dividing the *range* (the estimated maximum concentration minus the minimum concentration) by 6, or $s \approx Range / 6$. This approximation method should be used only if no other alternative is available. The approach is based on the assumption that more than 99 percent of all normally distributed measurements will fall within three standard deviations of the mean; therefore, the length of this interval is $6s$.

5.4.1 Number of Samples to Estimate the Mean: Simple Random Sampling

In Step 6 of the DQO Process ("Specify Limits on Decision Errors"), you established the width of the gray region (Δ) and acceptable probabilities for making a decision error (α and β). Using this information, along with an estimate of the standard deviation (s), calculate the appropriate number of samples (n) for simple random sampling using

$$n = \frac{(z_{1-\alpha} + z_{1-\beta})^2 s^2}{\Delta^2} + \frac{z_{1-\alpha}^2}{2} \qquad \textbf{Equation 8}$$

where

$z_{1-\alpha}$ = the $p\text{th}$ quantile of the standard normal distribution (from the last row of Table G-1, Appendix G), where α is the probability of making a Type I set in DQO Step 6 (Section 4.6.4).

$z_{1-\beta}$ = the $p\text{th}$ quantile of the standard normal distribution (from the last row of Table G-1, Appendix G), where β is the probability of making a Type II error set in DQO Step 6 (Section 4.6.4).

s = an estimate of the standard deviation.

Δ = the width of the gray region from DQO Step 6.

An example application of Equation 8 is presented in Box 9.

Two assumptions underlie the use of Equation 8. First, it is assumed that data are drawn from an approximately normal distribution. Second, it is assumed the data are uncorrelated. In correlated data, two or more samples taken close to each other (in time or in space) will have similar concentrations (Gilbert 1987). In situations in which spatial or temporal correlation is expected, some form of systematic sampling is preferred.

If the underlying population appears to exhibit a lognormal distribution, normal theory sample size equations (such as Equation 8) still can be used though they will tend to *underestimate* the minimum number of samples when the geometric standard deviation ($\exp(s_y)$) is low (e.g., $\leq$ 2). If the underlying distribution is known to be lognormal, the method given by Land (1971, 1975) and Gilbert (1987) for calculating confidence limits for a lognormal mean can be solved "in reverse" to obtain n. (A software tool for performing the calculation, MTCA*Stat* 3.0, is published by the Washington Department of Ecology. See Appendix H). Also, techniques described by Perez and Lefante (1996 and 1997) can be used to estimate the sample sizes needed to estimate the mean of a lognormal distribution. Otherwise, consult a professional statistician for assistance.

Box 9. Number of Samples Required to Estimate the Mean Using Simple Random Sampling: Hypothetical Example

Under 40 CFR 261.38, a generator of hazardous waste-derived fuel is seeking an exclusion from the definition of solid and hazardous-waste. To prepare the one-time notice under 40 CFR 261.38(c), the generator plans to conduct waste sampling and analysis to support the exclusion. The output of the first six steps of the DQO Process are summarized below:

Step 1: State the Problem: The planning team reviewed the applicable regulations, historical analyses, and process chemistry information. The problem is to determine whether Appendix VIII constituents present in the waste are at concentration levels less than those specified in Table 1 of §261.38.

Step 2: Identify the Decision: If the waste attains the specification levels, then it will be judged eligible for the exclusion from the definition of hazardous and solid waste.

Step 3: Identify Inputs to the Decision: Sample analysis results are required for a large number of constituents present in the waste, however, most constituents are believed to be present at concentrations well below the specification levels. Historically, benzene concentrations have been most variable, therefore, the planning team will estimate the number of samples required to determine if the specification level for benzene is attained.

Step 4: Define the Boundaries: The DQO decision unit is defined as the batch of waste generated over a one-week period. Samples will be taken as the waste exits the preparation process and prior to storage in a fuel tank (i.e., at the point of generation).

Step 5: Develop a Decision Rule: The RCRA regulations at 40 CFR 261.38(c)(8)(iii)(A) specify the mean as the parameter of interest. The "Action Level" for benzene is specified in Table 1 of §268.38 as 4,100 ppm. If the mean concentration of benzene within the DQO decision unit is less than or equal to 4,100 ppm, then the waste will be considered eligible for the exclusion (for benzene). Otherwise, the waste will not be eligible for the exclusion for benzene. (Note that the demonstration must be made for all Appendix VIII constituents known to be present in the waste).

Step 6: Specify Limits on Decision Errors: In the interest of being protective of the environment, the null hypothesis was established as "the mean concentration of benzene within the decision unit boundary exceeds 4,100 ppm," or H_o: mean (benzene) > 4,100 ppm. The boundaries of the gray region were set at the Action Level (4,100 ppm) and at a value less than the Action Level at 3000 ppm. The regulations at §261.38(c)(8)(iii)(A) specify a Type I (false rejection) error rate (α) of 0.05. The regulations do not specify a Type II (false acceptance) error rate (β), but the planning team deemed a false acceptance as of lesser concern than a false rejection, and set the false acceptance rate at 0.25. Sample analysis results from previous sampling and analyses indicate the standard deviation (s) of benzene concentrations is about 1,200 ppm.

What is the appropriate number of samples to collect and analyze for a simple random sampling design?

Solution: Using Equation 8 and the outputs of the first six steps of the DQO Process, the number of samples is determined as:

$$n = \frac{(z_{1-\alpha} + z_{1-\beta})^2 s^2}{\Delta^2} + \frac{z_{1-\alpha}^2}{2}$$

$$= \frac{(1.645 + 0.674)^2 (1200)^2}{(4100 - 3000)^2} + \frac{(1.645)^2}{2} = 7.75 \approx 8 \text{ (round up)}$$

where the values for $z_{1-\alpha}$ and $z_{1-\beta}$ are obtained from the last row of Table G-1 in Appendix G.

5.4.2 Number of Samples to Estimate the Mean: Stratified Random Sampling

An important aspect of a stratified random sampling plan is deciding how many samples to collect within each of the strata (Gilbert 1987). There are many ways to design a stratified random sampling plan; the development here makes the following assumptions (refer to Section 5.2.2 for a description of terms and symbols used below):

- Weights for each stratum (W_h) are known in advance. One possible way to assign weights to each stratum is to calculate the ratio between the waste volume classified as the $h\text{th}$ stratum and the total waste volume.
- The number of *possible* sample units (i.e., physical samples) of a certain physical size is much larger than the number of sample units that will be collected and analyzed. As a general guide, this assumption should be reasonable as long as the ratio between the stratum waste volume and the volume of the individual samples is at least 100. Otherwise, you may need to consider formulas that include the finite population correction (see Cochran 1977, page 24).
- The number of sample units to be collected and analyzed in each stratum, due to analytical costs and other considerations, generally will be fairly small.
- A preliminary estimate of variability (s_h^2) is available for each stratum. If this is not the case, one can use an estimate of the overall variability (s^2) as a substitute for the separate stratum estimates. By ignoring possible differences in the variance characteristics of separate strata, the sample size formulas given below may tend to underestimate the necessary number of samples for each strata (n_h).

Given a set of stratum weights and sample measurements in each stratum, the *overall* mean ($\bar{x}_{st}$) and *overall* standard error of the mean ($s_{\bar{x}_{st}}$) (i.e., for the entire waste under study) are computed as follows for a stratified random sample:

$$\bar{x}_{st} = \sum_{h=1}^{L} W_h \bar{x}_h$$ **Equation 9**

and

$$s_{\bar{x}_{st}} = \sqrt{\sum_{h=1}^{L} W_h^2 \frac{s_h^2}{n_h}}$$ **Equation 10**

Note that $\bar{x}_h$ and s_h^2 in these formulas represent the arithmetic mean and sample variance for the measurements taken within each stratum.

In general, there are two approaches for determining the number of samples to take when stratified random sampling is used: *optimal allocation* and *proportional allocation*.

5.4.2.1 Optimal Allocation

In optimal allocation, the number of samples assigned to a stratum (n_h) is proportional to the relative variability within each stratum and the relative cost of obtaining samples from each stratum. The number of samples can be determined to minimize the variance for a fixed cost or to minimize the cost for a prespecified variance.

Optimal allocation requires considerable advance knowledge about the relative variability within each stratum and the costs associated with obtaining samples from each stratum; therefore, we recommend the use of proportional allocation (see below) as an alternative. For more complex situations in which optimal allocation is preferred, consult a statistician or see Cochran (1977, page 96), Gilbert (1987, page 50), or USEPA (1989a (page 6-13)).

5.4.2.2 Proportional Allocation

In proportional allocation, the number of samples assigned to a stratum (n_h) is proportional to the stratum size, that is, $n_h = nW_h$. To determine the total number of samples (n) so that a true difference (Δ) between the mean waste concentration and the Action Level can be detected with Type I error rate α and Type II error rate β, use the following equation:

$$n = \frac{\left[t_{1-\alpha,df} + t_{1-\beta,df}\right]^2}{\Delta^2} \sum_{h=1}^{L} W_h s_h^2$$

Equation 11

To use this formula correctly, the degrees of freedom (df) connected with each t -quantile (from Table G-1, Appendix G) in the above equation must be computed as follows:

$$df = \left(\sum_{h=1}^{L} W_h s^2 \right)^2 \Big/ \sum_{h=1}^{L} \frac{W_h^2 s_h^4}{nW_h - 1}$$

Equation 12

Because the degrees of freedom also depend on n, the final number of samples must be computed iteratively. Then, once the final total number of samples is computed, the number of samples for each stratum is determined by multiplying the total number of samples by the stratum weight. An example of this approach is presented in Box 10.

If only an *overall* estimate of s^2 is available in the preliminary data, Equation 11 reduces to:

$$n = \frac{\left[t_{1-\alpha,df} + t_{1-\beta,df}\right]^2 s^2}{\Delta^2}$$

Equation 13

and Equation 12 reduces to

$$df = 1 \Big/ \sum_{h=1}^{L} \frac{W_h^2}{nW_h - 1}$$

Equation 14

Box 10. Number of Samples Required to Estimate the Mean Using Stratified Random Sampling – Proportional Allocation: Hypothetical Example

Under the RCRA Corrective Action program, a facility owner has conducted a cleanup of a solid waste management unit (SWMU) in which the contaminant of concern is benzene. The cleanup involved removal of all waste residues, contaminated subsoils, and structures. The facility owner needs to conduct sampling and analysis to confirm that the remaining soils comply with the cleanup standard.

Step 1: State the Problem: The planning team needs to confirm that soils remaining in place contain benzene at concentrations below the risk-based levels established by the authorized state as part of the cleanup.

Step 2: Identify the Decision: If the soils attain the cleanup standard, then the land will be used for industrial purposes. Otherwise, additional soil removal will be required.

Step 3: Identify Inputs to the Decision: A sampling program will be conducted, and sample analysis results for benzene will be used to make the cleanup attainment determination.

Step 4: Define the Boundaries: The DQO decision unit is the top 6 inches of soil within the boundary of the SWMU. Based on prior sample analysis results and field observations, two strata are identified: fine-grained soils in 20 percent of the unit ("Stratum 1"), and coarse-grained soils comprising the other 80 percent of the unit ("Stratum 2"). Based on the relative mass of the two strata, a weighting factor W_h is assigned to each $h\text{th}$ stratum such that $W_1 = 0.2$ and $W_2 = 0.8$.

Step 5: Develop a Decision Rule: The parameter of interest is established as the mean, and the Action Level for benzene is set at 1.5 mg/kg. If the mean concentration of benzene within the DQO decision unit is less than or equal to 1.5 mg/kg, then the unit will be considered "clean." Otherwise, another layer of soil will be removed.

Step 6: Specify Limits on Decision Errors: In the interest of being protective of the environment, the null hypothesis is established as "the mean concentration of benzene within the decision unit boundary exceeds 1.5 mg/kg," or Ho: mean (benzene) > 1.5 mg/kg. The boundaries of the gray region are set at the Action Level (1.5 mg/kg) and at a value less than the Action Level at 1.0 mg/kg. The Type I error rate (α) is set at 0.10 and the Type II error rate (β) is set at 0.25. Sample analysis results from $n = 8$ initial non-composite samples provided an estimate of the overall standard deviation of $s = 1.83$, and the standard deviations (s_h) within each $h\text{th}$ stratum of $s_1 = 2.5$ and $s_2 = 1.3$ (and $s_1^2 = 6.25$ and $s_2^2 = 1.69$).

What is the appropriate number of samples to collect and analyze for a stratified random sampling design?

Solution: Using Equation 12 for the degrees of freedom under proportional allocation:

$$df_1 = \left((0.2 \times 6.25) + (0.8 \times 1.69)\right)^2 \Bigg/ \left[\frac{(0.2 \times 6.25)^2}{8(0.2) - 1} + \frac{(0.8 \times 1.69)^2}{8(0.8) - 1} \right] = 2.3 \approx 2$$

Then, looking up the *t*-quantiles (from Table G-1, Appendix G) with 2 degree of freedom and taking $\Delta = 0.5$ (i.e., 1.5 ppm - 1.0 ppm), the total sample size (using Equation 12) works out to

$$n_1 = \frac{[1.886 + 0.816]^2}{(0.5)^2} \left((0.2 \times 6.25) + (0.8 \times 1.69)\right) = 76$$

Since the equations must be solved iteratively, recompute the formulas using $n = 76$. The same calculations give $df_2 = 48$ and $n_2 = 41$. After two more iterations, the sample size stabilizes at $n = 42$. Using the proportional allocation with $n = 42$ one should take 42(0.2) = 8.4 (round up to 9) measurements from the first stratum and 42(0.8) = 33.6 (round up to 34) measurements from the second stratum. Since four samples already were collected from each stratum, at least five additional random samples should be obtained from the first stratum and at least thirty additional random samples should be collected from the second stratum.

In the example in Box 10, stratified random sampling provides a more efficient and cost-effective design compared to simple random sampling of the same unit. If simple random sampling were used, a total of 52 samples would be required. With stratified random sampling, only 42 samples are required, thereby reducing sampling and analytical costs.

5.4.3 Number of Samples to Estimate the Mean: Systematic Sampling

Despite the attractiveness and ease of implementation of systematic sampling plans, whether via a fixed square, rectangular, or triangular grid, or through the use of systematic random sampling, methods for estimating the standard error of the mean are beyond the scope of this guidance (for example, see Cochran 1977) and often involve more advanced geostatistical techniques (for example, see Myers 1997). An alternate approach is to treat the set of systematic samples as though they were obtained using simple random sampling. Such an approach should provide reasonable results as long as there are no strong cyclical patterns, periodicities, or significant spatial correlations between adjacent sample locations. If such features are present or suspected to be present, consultation with a professional statistician is recommended.

By regarding the systematic sample as a simple random sample, one can simply use the algorithm and formulas for simple random sampling described in Section 5.4.1 (Equation 8) to estimate the necessary sample size. As with all the sampling designs described in this section, you should have a preliminary estimate of the sample variance before using the sample size equation.

5.4.4 Number of Samples to Estimate the Mean: Composite Sampling

In comparison to noncomposite sampling, composite sampling may have the effect of minimizing between-sample variation, thereby reducing somewhat the total number of composite samples that must be submitted for analysis.

The appropriate number of composite samples to be collected from a waste or media can be estimated by Equation 8 for simple random and systematic composite sampling. Equation 11 can be used when composite sampling will be implemented with a stratified random sampling design (using proportional allocation). Any preliminary or pilot study conducted to estimate the appropriate number of composite samples should be generated using the same compositing scheme planned for the confirmatory study. If the preliminary or pilot study data were generated using random "grab" samples rather than composites, then the sample variance (s^2) in the sample size equations should be replaced with s^2/g where g is the number of individual or grab samples used to form each composite (Edland and Van Belle 1994, page 45).

Additional guidance on the optimal number of samples required for composite sampling and the number of subsample aliquots required to achieve maximum precision for a fixed cost can be found in Edland and van Belle (1994, page 36 and page 44), Exner, et al. (1985, page 512), and Gilbert (1987, page 78).

5.5 Determining the Appropriate Number of Samples to Estimate A Percentile or Proportion

This section provides guidance for determining the appropriate number of samples (n) needed to estimate an upper percentile or proportion with a prespecified level of confidence. The approaches can be used when the objective is to determine whether the upper percentile is less than a concentration standard or whether a given proportion of the population or decision unit is less than a specified value.

Two methods for determining the appropriate number of samples are given below: (1) Section 5.5.1 provides a method based on the assumption that the population is large and the samples are drawn at random from the population, and (2) Section 5.5.2 provides a method with similar assumptions but only requires specification of the level of confidence required and the number of exceedances allowed (usually zero). For both methods, it is assumed that the measurements can be expressed as a binary variable – that is, that the sample analysis results can be interpreted as either in compliance with the applicable standard ("pass") or not in compliance with the applicable standard ("fail").

5.5.1 Number of Samples To Test a Proportion: Simple Random or Systematic Sampling

This section provides a method for determining the appropriate number of samples when the objective is to test whether a proportion or percentile of a population complies with an applicable standard. A population proportion is the ratio of the number of elements of a population that has some specific characteristic to the total number of elements. A population percentile represents the percentage of elements of a population having values less than some value. The number of samples needed to test a proportion can be calculated using

$$n = \left[\frac{z_{1-\beta}\sqrt{GR(1-GR)} + z_{1-\alpha}\sqrt{AL(1-AL)}}{\Delta} \right]^2 \qquad \textbf{Equation 15}$$

where

α = false rejection error rate
β = false acceptance error rate
z_p = the pth percentile of the standard normal distribution (from the last row of Table G-1 in Appendix G)
AL = the Action Level (e.g., the proportion of all possible samples of a given support that must comply with the standard)
GR = other bound of the gray region,
Δ = width of the gray region ($GR - AL$), and
n = the number of samples.

An example calculation of n using the approach described here is presented in Box 11.

Box 11. Example Calculation of the Appropriate Number of Samples Needed To Test a Proportion – Simple Random or Systematic Sampling

A facility is conducting a cleanup of soil contaminated with pentachlorophenol (PCP). Based on the results of a field test method, soil exceeding the risk-based cleanup level of 10 mg/kg total PCP will be excavated, classified as a solid or hazardous waste, and placed into roll-off boxes for subsequent disposal, or treatment (if needed) and disposal. The outputs of the first six steps of the DQO Process are summarized below.

Step 1: State the Problem: The project team needs to decide whether the soil being placed in each roll-off box is a RCRA hazardous or nonhazardous waste.

Step 2: Identify the Decision: If the excavated soil is hazardous, it will be treated to comply with the applicable LDR treatment standard and disposed as hazardous waste. If it is nonhazardous, then it will be disposed as solid waste in a permitted industrial waste landfill (as long as it is not mixed with a listed hazardous waste).

Step 3: Identify Inputs to the Decision: The team requires sample analysis results for TCLP PCP to determine compliance with the RCRA TC regulatory threshold of 100 mg/L.

Step 4: Define the Boundaries: The DQO "decision unit" for each hazardous waste determination is defined as a roll-off box of contaminated soil. The "support" of each sample is in part defined by SW-846 Method 1311 (TCLP) as a minimum mass of 100-grams with a maximum particle size of 9.5 mm. Samples will be obtained as the soil is excavated and placed in the roll-off box (i.e., at the point of generation).

Step 5: Develop a Decision Rule: The project team wants to ensure with reasonable confidence that little or no portions of the soil in the roll-off box are hazardous waste. The parameter of interest is then defined as the 90th percentile. If the 90th percentile concentration of PCP is less than 100 mg/L TCLP, then the waste will be classified as nonhazardous. Otherwise, it will be considered hazardous.

Step 6: Specify Limits on Decision Errors: The team establishes the null hypothesis (H_o) as the "true proportion (P) of the waste that complies with the standard is less than 0.90," or H_o: P < 0.90. The false rejection error rate (α) is set at 0.10. The false acceptance error rate (β) is set at 0.30. The Action Level (AL) is 0.90, and the other boundary of the gray region (GR) is set at 0.99.

How many samples are required?

Solution: Using Equation 15 and the outputs of the first six steps of the DQO Process, the number of samples (n) is determined as:

$$= \left[\frac{0.524\sqrt{0.99(1-0.99)} + 1.282\sqrt{0.90(1-0.90)}}{0.99-0.90} \right]^2 = 23.5 \approx 24$$

where the values for $z_{1-\alpha}$ and $z_{1-\beta}$ are obtained from the last row of Table G-1 in Appendix G.

5.5.2 Number of Samples When Using a Simple Exceedance Rule

If a simple exceedance rule is used (see Section 3.4.2.2), then it is possible to estimate the number of samples required to achieve a prespecified level of confidence that a given fraction of the waste or site has a constituent concentration less than the standard or does not exhibit a characteristic or property of concern. The approach is based on the minimum sample size required to determine a nonparametric (distribution-free) one-sided confidence bound on a percentile (Hahn and Meeker 1991 and USEPA 1989a).

If the exceedance rule specifies no exceedance of the standard in any sample, then the number of samples that must achieve the standard can be obtained from Table G-3a in Appendix G. The table is based on the expression:

$$n = \log(\alpha)/\log(p) \qquad \textbf{Equation 16}$$

where alpha (α) is the probability of a Type I error and p is the proportion of the waste or site that must comply with the standard. Alternatively, the equation can be rearranged so that statistical performance ($1-\alpha$) can determined for a fixed number of samples:

$$(1-\alpha) = 1 - p^n \qquad \textbf{Equation 17}$$

Notice that the method does not require specification of the other bound of the gray region, nor does it require specification of a Type II (false acceptance) error rate (β).

If the decision rule allows *one* exceedance of the standard in a set of samples, then the number of samples required can be obtained from Table G-3b in Appendix G.

An example application of the above equations is presented in Box 12. See also Appendix F, Section F.3.2.

Box 12. Example Calculation of Number of Samples Needed When a Simple Exceedance Rule Is Used – Simple Random or Systematic Sampling

What is the minimum number of samples required (with no exceedance of the standard in any of the samples) to determine with at least 90-percent confidence ($1-\alpha = 0.90$) that at least 90 percent of all possible samples from the waste (as defined by the DQO decision unit) are less than the applicable standard?

From Table G-3a, we find that for $1 - a = 0.90$ and $p = 0.90$ that 22 samples are required. Alternately, using Equation 16, we find

$$n = \frac{\log(\alpha)}{\log(p)} = \frac{\log(0.10)}{\log(0.90)} = \frac{-1}{-0.0457} = 21.8 \approx 22$$

If only 11 samples were analyzed (with no exceedance of the standard in any of the samples), what level of confidence can we have that at least 90 percent of all possible samples are less than the standard? Using Equation 17, we find

$$(1-\alpha) = 1 - p^n = 1 - 0.90^{11} = 1 - 0.3138 = 0.6862$$

Rounding *down*, we can say with at least 68 percent confidence that at least 90 percent of all possible samples would be less than the applicable standard.

5.6 Selecting the Most Resource-Effective Design

For additional guidance on selecting the most resource-efficient design, see ASTM standard D 6311-98, *Standard Guide for Generation of Environmental Data Related to Waste Management Activities: Selection and Optimization of Sampling Design*.

If more than one sampling design option is under consideration, evaluate the various designs based on their cost and the ability to achieve the data quality and regulatory objectives. Choose the design that provides the best balance between the expected cost and the ability to meet the objectives. To improve the balance between meeting your cost objectives and achieving the DQOs, it might be necessary to modify either the budget or the DQOs. As can be seen from the sample size equations in Section 5.4 and 5.5, there is an interrelationship between the appropriate number of samples and the desired level of confidence, expected variability (both population and measurement variability), and the width of the gray region. To reduce costs (i.e., decrease the number of samples required), several options are available:

- Decrease the confidence level for the test
- Increase the width of the "gray region" (not recommended if the parameter of interest is near the Action Level)
- Divide the population into smaller less heterogeneous decision units, or use a stratified sampling design in which the population is broken down into parts that are internally less heterogeneous
- Employ composite sampling (if non-volatile constituents are of interest and if allowed by the regulations).

Note that seemingly minor modifications to the sampling design using one or more of the above strategies may result in major increases or decreases in the number of samples needed.

When estimating costs, be sure to include the costs for labor, travel and lodging (if necessary), expendable items (such as personal protective gear, sample containers, preservatives, etc.), preparation of a health and safety plan, sample and equipment shipping, sample analysis, assessment, and reporting. Some sampling plans (such as composite sampling) may require fewer analyses and associated analytical costs, but might require more time to implement and not achieve the project objectives. EPA's *Data Quality Objectives Decision Error Feasibility Trials Software (DEFT)* (USEPA 2001a) is one tool available that makes the process of selecting the most resource effective design easier.

5.7 Preparing a QAPP or WAP

In this activity, the outputs of the DQO Process and the sampling design are combined in a planning document such as a QAPP or WAP. The Agency has developed detailed guidance on how to prepare a QAPP (see USEPA 1998a) or WAP (see USEPA 1994a). The minimum requirements for a WAP are specified at 40 CFR §264.13. The following discussion is focused on the elements of a QAPP; however, the information can be used to help develop a WAP.

Additional EPA Guidance on Preparing a QAPP or WAP

- Chapter One, SW-846
- *EPA Requirements for Quality Assurance Project Plans, EPA QA/R*-5 (replaces QAMS-005/80) (USEPA 2001b)
- *EPA Guidance for Quality Assurance Project Plans, EPA QA/G*-5 (EPA/600/R-98/018) (USEPA 1998a)
- *Guidance for Choosing a Sampling Design for Environmental Data Collection, EPA QA/G-5S - Peer Review Draft* (EPA QA/G-5S) (USEPA 2000c)
- *Waste Analysis at Facilities That Generate, Treat, Store, And Dispose Of Hazardous Wastes, a Guidance Manual* (USEPA 1994a)

The QAPP is a critical planning document for any environmental data collection operation because it documents project activities including how QA and QC activities will be implemented during the life cycle of a project. The QAPP is the "blueprint" for identifying how the quality system of the organization performing the work is reflected in a particular project and in associated technical goals. QA is a system of management activities designed to ensure that data produced by the operation will be of the type and quality needed and expected by the data user. QA, acknowledged to be a management function emphasizing systems and policies, aids the collection of data of needed and expected quality appropriate to support management decisions in a resource-efficient manner.

The activities addressed in the QAPP cover the entire project life cycle, integrating elements of the planning, implementation, and assessment phases. If the DQOs are documented (e.g., in a memo or report format), include the DQO document as an attachment to the QAPP to help document the technical basis for the project and to document any agreements made between stakeholders.

As recommended in EPA QA/G-5 (USEPA 1998a), a QAPP is composed of four sections of project-related information called "groups," which are subdivided into specific detailed "elements." The elements and groups are summarized in the following subsections.

5.7.1 Project Management

The QAPP (or WAP) is prepared after completion of the DQO Process. Much of the following guidance related to project management can be excerpted from the outputs of the DQO Process.

The following group of QAPP elements covers the general areas of project management, project history and objectives, and roles and responsibilities of the participants. The following elements ensure that the project's goals are clearly stated, that all participants understand the goals and the approach to be used, and that project planning is documented:

- Title and approval sheet
- Table of contents and document control format
- Distribution list
- Project/task organization and schedule (from DQO Step 1)
- Problem definition/background (from DQO Step 1)
- Project/task description (from DQO Step 1)
- Quality objectives and criteria for measurement data (DQO Step 3)

- Special training requirements/certification
- Documentation and records.

For some projects, it will be necessary to include the names and qualifications of the person(s) who will obtain the samples (e.g., as required under 40 CFR §261.38(c)(7) in connection with testing for the comparable fuels exclusion).

5.7.2 Measurement/Data Acquisition

This group of QAPP elements covers all aspects of measurement system design and implementation, ensuring that appropriate methods for sampling, analysis, data handling, and QC are employed and thoroughly documented. Apart from the sample design step (DQO Step 7), the following information should be included in the QAPP or incorporated by reference:

- Sampling process design/experimental design (DQO Steps 5 and 7)
- Sampling methods and SOPs
- Sample handling and chain-of-custody requirements
- Analytical methods and SOPs (DQO Step 3)
- QC requirements;
- Instrument/equipment testing, inspection, and maintenance requirements
- Instrument calibration and frequency
- Inspection/acceptance requirements for supplies and consumables
- Data acquisition requirements (non-direct measurements)
- Data management.

For some projects, under various circumstances it may be appropriate to include hard copies of the SOPs in the QAPP, rather than incorporate the information by reference. For example, under the performance-based measurement system (PBMS) approach, alternative sampling and analytical methods can be used. Such methods can be reviewed and used more readily if actual copies of the SOPs are included in the QAPP. Hard copies of SOPs also are critically important when field analytical techniques are used. Field personnel must have detailed instructions available to ensure that the methods are followed. If it is discovered that deviation from an SOP is required due to site-specific circumstances, the deviations can be documented more easily if hard copies of the SOPs are available in the field with QAPP.

5.7.3 Assessment/Oversight

The purpose of assessment is to ensure that the QAPP is implemented as prescribed. The elements below address the activities for assessing the effectiveness of the implementation of the project and the associated QA/QC activities:

- Assessments and response actions
- Reports to management.

5.7.4 Data Validation and Usability

Implementation of these elements ensures that the data conform to the specified criteria, thus enabling reconciliation with the project's objectives. The following elements cover QA activities that occur after the data collection phase of the project has been completed:

- Data review, verification, and validation requirements
- Verification and validation methods
- Reconciliation with DQOs.

5.7.5 Data Assessment

Historically, the focus of most QAPPs has been on analytical methods, sampling, data handling, and quality control. Little attention has been paid to data assessment and interpretation. We recommend that the QAPP address the data assessment steps that will be followed after data verification and validation. While it may not be possible to specify the statistical test to be used in advance of data generation, the statistical objective (identified in the DQO Process) should be stated along with general procedures that will be used to test distributional assumptions and select statistical tests. EPA's *Guidance for Data Quality Assessment* (USEPA 2000d) suggests the following five-step methodology (see also Section 8 for a similar methodology):

1. Review the DQOs
2. Conduct a preliminary data review
3. Select the statistical test
4. Verify the assumptions of the test
5. Draw conclusions from the Data.

The degree to which each QAPP element should be addressed will be dependent on the specific project and can range from "not applicable" to extensive documentation. The final decision on the specific need for these elements for project-specific QAPPs will be made by the regulatory agency. Documents prepared prior to the QAPP (e.g., SOPs, test plans, and sampling plans) can be appended or, in some cases, incorporated by reference.

6 CONTROLLING VARIABILITY AND BIAS IN SAMPLING

The DQO Process allows you to identify the problem to be solved, set specific goals and objectives, establish probability levels for making incorrect decisions, and develop a resource-efficient data collection and analysis plan. While most of the sampling designs suggested in this guidance incorporate some form of randomness so that unbiased estimates can be obtained from the data, there are other equally important considerations (Myers 1997). Sampling and analysis activities must also include use of correct devices and procedures to minimize or control random variability and biases (collectively known as "error") that can be introduced in field sampling, sample transport, subsampling, sample preparation, and analysis. Sampling error can lead to incorrect conclusions irrespective of the quality of the analytical measurements and the appropriateness of the statistical methods used to evaluate the data.

This section is organized into three subsections which respond to these questions:

1. What are the sources of error in sampling (Section 6.1)?
2. What is sampling theory (Section 6.2)?
3. How can you reduce or otherwise control sampling error in the field and laboratory (Section 6.3)?

6.1 Sources of Random Variability and Bias in Sampling

In conducting sampling, we are interested in obtaining an estimate of a population parameter (such as the mean, median, or a percentile); but an estimate of a parameter made from measurements of samples always will include some random variability (or variances) and bias (or a systematic shift away from the true value) due primarily to (1) the inherent variability of the waste or media (the "between-sampling-unit variability") and (2) imprecision in the methods used to collect and analyze the samples (the "within-sampling-unit variability") (USEPA 2001e).

Errors caused by the sample collection process can be much greater than the preparation, analytical, and data handling errors (van Ee, et al. 1990, Crockett, et al 1996) and can dominate the overall uncertainty associated with a characterization study (Jenkins, et al. 1996 and 1997). In fact, analytical errors are usually well-characterized, well-understood, and well-controlled by laboratory QA/QC, whereas sampling and sample handling errors are not usually well-characterized, well-understood, or well-controlled (Shefsky 1997). Because sampling error contributes to overall error, it is important for field and laboratory personnel to understand the sources of sampling errors and to take measures to control them in field sampling.

The two components of error -- random variability and bias -- are independent. This concept is demonstrated in the "target" diagram (see Figure 7 in Section 2), in which random variability (expressed as the variance, σ^2) refers to the "degree of clustering" and bias ($\mu - \bar{x}$) relates to the "amount of offset from the center of the target" (Myers 1997).

Random variability and bias occur at each stage of sampling. Variability occurs due to the **heterogeneity** of the material sampled and random variations in the sampling and sample handling procedures. In addition, bias can be introduced at each stage by the sampling device (or the manner in which it is used), sample handling and transport, subsampling, and analysis.

While it is common practice to calculate the variability of sample analysis results "after the fact," it is more difficult to identify the sources and potential impacts of systematic sampling bias. As discussed in more detail below, it usually is best to understand the potential sources of error "up front" and take measures to minimize them when planning and implementing the sampling and analysis program.

Even though random variability and bias are independent, they are related quantitatively (see Figure 23). Errors expressed as the variance can be added together to estimate overall or "total study error." Biases can be added together to estimate overall bias (though sampling bias is difficult to measure in practice). Conceptually, the sum of all the variances can be added to the sum of all biases (which is then squared) and expressed as the **mean square error** ($MSE(\bar{x})$) which provides a quantitative way of measuring the degree of *representativeness* of the samples. In practice, it is not necessary to try to calculate mean square error, however, we suggest you understand the sources and impacts of variability and bias so you can take steps to control them in sampling and improve the representativeness of the samples. (See Sections 5.2.4 and 5.2.5 of EPA's *Guidance for Data Quality Assessment, EPA QA/G-9 - QA00 Update* (USEPA 2000d) for a more detailed discussion of how to address measurement variability and bias in the sampling design).

Random Variability

$$\sigma^2 = \sigma_b^2 + \sigma_s^2 + \sigma_a^2$$

where

σ_b^2 = Between-sampling-unit variability (population variability)

σ_s^2 = Sampling and subsampling variability

σ_a^2 = Analytical variability

Systematic Error (Bias)

$bias =$ Sum of all biases

including

- Sampling bias (e.g., improper selection and use of sampling devices; loss or gain of constituents during sampling, transport, storage, subsampling, and sample preparation)
- Analytical bias
- Statistical bias
- Mistakes, blunders, sabotage

$$\text{MSE}(\bar{x}) = \sigma^2 + (bias)^2$$

Figure 23. Components of error and the additivity of variances and biases in sampling and analysis

The relatively new science of sampling theory and practice (Myers 1997) provides a technically based approach for addressing sampling errors (see Section 6.2). Sampling theory recognizes that sampling errors arise from or are related to the size and distribution of particles in the waste, the weight of the sample, the shape and orientation of the sampling device, the manner in which the sample is collected, sample handling, and the manner in which subsampling is performed within the laboratory. Sampling theory applies to particulate solids, liquids, and mixtures of solids and liquids. Understanding sampling theory does not allow us to completely eliminate sampling and analytical errors, but sampling theory does allow us to identify the sources and magnitudes of sampling errors so we can take steps to minimize those that are the largest. In doing so, samples will be more precise and unbiased (i.e., more "representative"), thus reducing the number of samples required (lowering costs) and improving our ability to achieve the decision error rate specified in the DQOs.

6.2 Overview of Sampling Theory

A number of environmental scientists have recognized a set of sampling theories developed by Dr. Pierre Gy (Gy 1982 and 1998) and others (Ingamells and Switzer 1973; Ingamells 1974; Ingamells and Pitard 1986; Pitard 1989; and Visman 1969) as one set of tools for improving sampling. These researchers have studied the sources of sampling error (particularly in the sampling of particulate matter) and developed techniques for quantifying the amount of error that can be introduced by the physical sampling process. The theories were originally developed in support of mineral exploration and mining and more recently were adopted by EPA for soil sampling (van Ee, et al. 1990; Mason 1992). Under some conditions, however, the theories can be applied to waste sampling as a means for improving the efficiency of the sampling and analysis process (Ramsey, et al. 1989).

As discussed in the context of this guidance, Gy's theories focus on minimizing error during the physical collection of a sample of solid and liquid media and should not be confused with the statistical sampling designs such as simple random, stratified random, etc. discussed in Section 5. Both sampling theory and sampling design, however, are critical elements in sampling: Gy's theories facilitate collection of "correct" individual samples, while statistical sampling designs allow us to conduct statistical analyses and make conclusions about the larger mass of waste or environmental media (i.e., the decision unit).

The following three subsections describe key aspects of sampling theory including heterogeneity, sampling errors, and the concept of sample support. The descriptions are mostly qualitative and intended to provided the reader with an appreciation for the types and complexities of sampling error. Detailed descriptions of the development and application of sampling theory can be found in *Sampling for Analytical Purposes* (Gy 1998), *Geostatistical Error Management* (Myers 1997), *Pierre Gy's Sampling Theory and Sampling Practice* (Pitard 1993), and in EPA's guidance document *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (Mason 1992).

6.2.1 Heterogeneity

One of the underlying principles of sampling theory is that the medium to be sampled is not uniform in its composition or in the distribution of constituents in the medium, rather, it is **heterogeneous**. Heterogeneity causes the sampling errors.

Appropriate treatment of heterogeneity in sampling depends on the scale of observation. Large-scale variations in a waste stream or site affect where and when we take samples. Small-scale variations in a waste or media affect the size, shape, and orientation of individual field samples and laboratory subsamples. Gy's theory identifies three major types of heterogeneity: (1) short-range (or small-scale) heterogeneity, (2) long-range (or large-scale) heterogeneity, and (3) periodic heterogeneity:

> **Short-range heterogeneity** refers to properties of the waste at the sample level or in the immediate vicinity of a sample location. Two other types of heterogeneity are found within short-range heterogeneity: one reflected by differences in the *composition* between individual particles, the other having to do with the *distribution* of those particles in the waste. Composition heterogeneity (also known as *constitution heterogeneity*) is constant and cannot be altered except by particle size reduction (e.g., grinding or crushing the material). The distribution heterogeneity plays an important role in sampling because particles can separate into groups. Distribution heterogeneity can be increased (e.g., by gravitational segregation of particles or liquids) and can be reduced by homogenization (mixing) or by taking many small increments to form a sample.
>
> **Large-scale heterogeneity** reflects local trends and plays an important role in deciding whether to divide the population into smaller internally homogenous decision units or to use a stratified sampling design. See Appendix C for a detailed description of large-scale heterogeneity.
>
> **Periodic heterogeneity**, another larger-scale phenomena, refers to cyclic phenomena found in flowing streams or discharges. Understanding periodic heterogeneity can aid in dividing a waste into separate waste streams or in establishing a stratified sampling design.

Forming a conceptual model of the heterogeneity of a waste will help you to determine how to address it in sampling.

6.2.2 Types of Sampling Error

Gy's theory (see also Mason 1992, Pitard 1993, and Gy 1998) identifies a number of different types of error that can occur in sampling as a result of heterogeneity in the waste and failure to correctly define the appropriate shape and volume of material for inclusion in the sample. Understanding the types and sources of the errors is an important step toward *avoiding* them. In qualitative terms, these errors include the following:

- *Fundamental error*, which is caused by differences in the composition of individual particles in the waste

- Errors due to *segregation* and *grouping* of particles and the constituent associated with the particles

- Errors due to various types of trends including small-scale trends, large-scale trends, or cycles

- Errors due to defining (or *delimiting*) the sample space and *extracting* the sample from the defined area

- Errors due to *preparation* of the sample, including shipping and handling. [Note that the term "preparation," as used here, describes all the activities that take

> place after the primary sample is obtained in the field and includes sample containerization, preservation, handling, mixing, grinding, subsampling, and other preparative steps taken prior to analysis (such as the “sample preparation methods” as described in Chapters Three, Four, and Five of SW-846).]

Errors that can occur during sampling are described below.

6.2.2.1 Fundamental Error

The composition of a sample never perfectly matches the overall composition of the larger mass from which is was obtained because the mass of an individual sample is always less than the mass of the population and the population is never completely homogeneous. These conditions result in a sampling error known as **fundamental error**. The error is referred to as “fundamental” because it is an incompressible minimum sampling error that depends on the composition, shape, fragment size distribution, and chemical properties of the material, and it is not affected by homogenization or mixing. It arises when the constituent of interest is concentrated in constituent “nuggets” in a less concentrated matrix, especially when the constituent is present at a trace concentration level (e.g., less than 1 percent). This type of sampling error occurs even when the nuggets are mixed as well as possible in the matrix (so long as they are not dissolved). The fundamental error is the only error that remains when the sampling operation is “perfect”; that is, when all parts of the sample are obtained in a probabilistic manner and each part is independent.

As a conceptual example of fundamental error, consider a container filled with many white marbles and a few black marbles that have been mixed together well (Figure 24). If a small sample comprising only a few marbles is picked at random, there is a high probability they would all be white (Sample “A” in Figure 24) and a small chance that one or more would be black. As the sample size becomes larger, the distribution in the sample will reflect more and more closely the parent population (Sample “B” in Figure 24). The situation is similar in a waste that contains rare highly concentrated “nuggets” of a constituent of concern. If a small sample is taken, it is possible, and even likely, that no nuggets of the constituent would be selected as part of the sample. This would lead to a major *underestimate* of the true parameter of interest. It also is possible with a small sample that a gross *overestimate* of the parameter of interest will occur if a nugget is included in the sample because the nugget would comprise a relatively large proportion of the analytical sample compared to the true population. To minimize fundamental error, the point is not to simply “fish” for a black marble (the contaminant), but to sample for all of the fragments and constituents such that the sample is a representation of the lot from which it is derived.

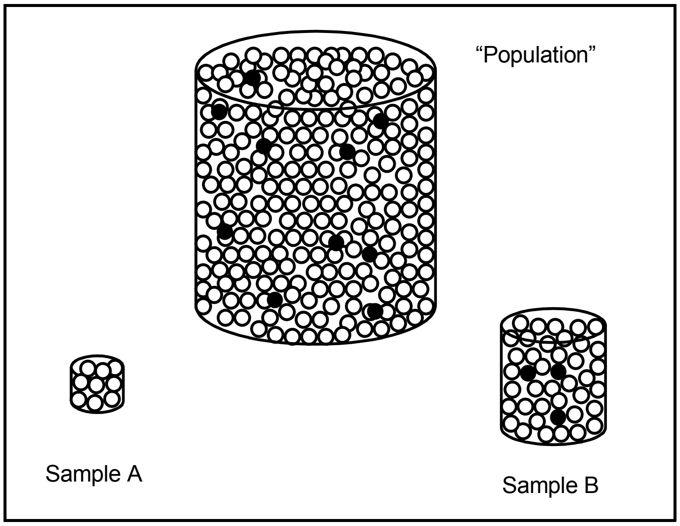

Figure 24. Effects of sample size on fundamental error. Small samples such as “A” cause the constituent of interest to be under-represented in most samples and over-represented in a small proportion of samples. Larger samples such as “B” more closely reflect the parent population.

The fundamental error is never zero (unless the population is completely homogeneous or the entire population is submitted for analysis) and it never "cancels out." It can be controlled by taking larger physical samples; however, larger samples can be difficult to handle in the field and within the laboratory, and they may pose practical constraints due to increased space needed for storage. Furthermore, small samples (e.g., less than 1 gram) generally are required for analytical purposes. To preserve the character of a large sample in the small analytical sample, subsampling and particle size reduction strategies should be employed (see also Section 7.3).

6.2.2.2 Grouping and Segregation Error

Grouping and segregation results from the short-range heterogeneity within and around the area from which a sample is collected (i.e., the sampling location) and within the sample container. This small-scale heterogeneity is caused by the tendency for some particles to associate into groups of like particles due to gravitational separation, chemical partitioning, differing moisture content, magnetism, or electrostatic charge. Grouping and segregation of particles can lead to sampling bias.

Figure 25 depicts grouping of particles (at "A") and segregation of particles (at "B") within a sample location. The grouping of particles at location "A" could result from an affinity between like particles (for example, due to electrostatic forces). Analytical samples formed from just one group of particles would yield biased results.

The segregation of particles at location "B" could result from gravitation separation (e.g., during sample shipment). If the contaminant of interest was associated with only one class of particle (for example, only the black diamond shapes), then a sample collected from the top would result in a different concentration than a sample collected from the bottom, thus biasing the sample.

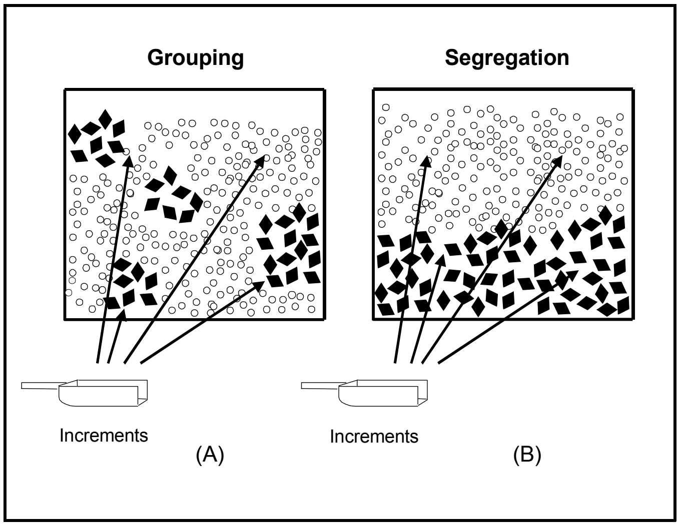

Figure 25. How grouping and segregation of particles can affect sampling results. Grouping and segregation error can be minimized by taking many small increments.

Grouping and segregation error can be minimized by properly homogenizing and splitting the sample. As an alternative, an individual sample can be formed by taking a number of increments (small portions of media) in the immediate vicinity of the sampling location and combining them into the final collected sample.[1] Pitard (1993) suggests collecting between 10 and 25 increments as a means to control grouping and segregation error. These increments are then combined to form an individual sample to be submitted to the laboratory for analysis.

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[1] This approach should not be confused with composite sampling, in which individual samples from different times or locations are pooled and mixed into a single sample.

The approach of taking multiple increments to form a sample is not recommended when volatile constituents are of interest and may have practical limitations when sampling highly heterogeneous wastes or debris containing very large fragments.

6.2.2.3 Increment Delimitation Error

Increment delimitation error occurs when the shape of the sampling device excludes or discriminates against certain portions of the material to be sampled. For example, a sampling device that only samples the top portion of a liquid effluent as it is leaves a discharge pipe (leaving a portion of the flow unsampled) causes increment delimitation error. This type of error is eliminated by choosing a sampling device capable of obtaining all of the flow for a fraction of the time (see also Sections 6.3.2 and 6.3.3).

6.2.2.4 Increment Extraction Error

Increment extraction error occurs when portions of the sample are lost or extraneous materials are included in the sample. For example, if the coring device is too small to accommodate a large fragment of waste, particles that should be in the sample might get pushed aside, causing sampling bias. Extraction error can be controlled through selection of devices designed to accommodate the physical characteristics of the waste.

6.2.2.5 Preparation Error

This error results from the incorrect preservation, handling, mixing, grinding, and subsampling that can result in loss, contamination, or altering of the sample such that it no longer is an accurate representation of the material being sampled. Proper choice and implementation of preparation methods controls this error.

6.2.3 The Concept of "Sample Support"

The weight, shape (length, width and height dimensions), and orientation of a sample describe the "sample support." The term "support" has been used in sampling and statistical literature in various ways, such as to describe the mass or volume of an "exposure unit" or "exposure area" in the Superfund program -- similar to the "decision unit" described in the DQO Process.

Conceptually, there is a continuum of support from the decision unit level (e.g., an exposure area of a waste site or a drum of solid waste) to the sample and subsample level down to the molecular level. Because it is not possible to submit the entire decision unit for analysis, samples must be submitted instead. For heterogeneous media, *the sample support will have a substantial effect on the reported measurement values*.

Measures can be taken to ensure adequate size, shape, and orientation of a sample:

- The appropriate *size* of a sample (either volume or mass) can be determined based on the relationship that exists between the particle size distribution and expected sampling error -- known as the fundamental error (see Section 6.2.2.1). In the DQO Process, you can define the amount of fundamental error that is acceptable (specified in terms of the standard deviation of the fundamental error) and estimate the volume required for field samples. The sampling tool should have dimensions three or more times larger than that of the diameter of the largest particles. Proper sizing of the sampling tool will help ensure that the particle size distribution of the sampled material is represented in the sample (see discussion at Section 6.3.1).

- The appropriate *shape and orientation* of the sample are determined by the sampling mode. For a one-dimensional waste (e.g., liquid flowing from a discharge pipe or solids on a conveyor belt), the correct or "ideal" sample is an undisturbed cross section delimited by two parallel planes (Pitard 1993, Gy 1998) (see discussion at Section 6.3.2.1). For three-dimensional waste forms (such as solids in a roll-off bin, piles, thick slabs, soil in drums, liquids in a tank, etc.), the sampling problem is best treated as a series of overlapping two-dimensional problems. The correct or ideal sample is an undisturbed core (Pitard 1993) that captures the entire thickness of the waste (see discussion at Section 6.3.2.2).

6.3 Practical Guidance for Reducing Sampling Error

This section describes steps that can be taken to control sampling error. While the details of sampling theory may appear complex and difficult to explain, in practice most sampling errors can be minimized by observing a few simple rules that, when used, can greatly improve the reliability of sampling results with little or no additional costs (Gy 1998):

- Determine the optimal mass of each field sample. For particulate solids, determine the appropriate sample weight based on the particle size distribution and characteristics, and consider any practical constraints (see Section 6.3.1). Also, determine additional amounts of the sampled material needed for split samples, for field and laboratory quality control purposes, or for archiving.

- Select the appropriate shape and orientation of the sample based on the sampling design model identified in DQO Step 7 (see Section 6.3.2).

- Select sampling devices and procedures that will minimize grouping and segregation errors and increment delimitation and increment extraction errors (see Sections 6.3.3 and 7.1).

Implement the sampling plan by obtaining the number of samples at the sampling locations and times specified in the sampling design selected in DQO Step 7, and take measures to minimize preparation errors during sample handling, subsampling, analysis, documentation, and reporting. When collecting samples for analysis for volatile organic constituents, special considerations are warranted to minimize bias due to loss of constituents (see Section 6.3.4).

Table 7 provides a summary of strategies that can be employed to minimize the various types of sampling error.

Table 7. Strategies for Minimizing Sampling Error

Type of Sampling Error	*Strategy To Minimize or Reduce Error*
Fundamental Error	• To reduce variability caused by fundamental error, increase the volume of the sample. • To reduce the volume of the sample and maintain low fundamental error, perform particle-size reduction followed by subsampling. • When volatile constituents are of interest, do not grind or mix the sample. Rather, take samples using a method that minimizes disturbances of the sample material (see also Section 6.3.4).
Grouping and Segregation Error	• To minimize grouping error, take many increments. • To minimize segregation error, homogenize the sample (but beware of techniques that promote segregation)
Increment Delimitation/Extraction Errors	• Select sampling devices that delimit and extract the sample so that all material that should be included in the sample is captured and retained by the device (Pitard 1993, Myers 1997). • For one-dimensional wastes (e.g., flowing streams or waste on a conveyor), the correct or "ideal" sample is an undisturbed cross section delimited by two parallel planes (Pitard 1993, Gy 1998). To obtain such a sample, use a device that can obtain "all of the flow for a fraction of the time" (Gy 1998) (see also Section 6.3.2.1). • For three-dimensional wastes (e.g., solids in a roll-off bin), the waste can be considered for practical purposes a series of overlapping two-dimensional wastes. The correct or "ideal" sample is an undisturbed vertical core (Pitard 1993, Gy 1998) that captures the full depth of interest.
Preparation Error	• Take steps to prevent contamination of the sample during field handling and shipment. Sample contamination can be checked through preparation and analysis of field quality control samples such as field blanks, trip blanks, and equipment rinsate blanks. • Prevent loss of volatile constituents through proper storage and handling. • Minimize chemical transformations via proper storage and chemical/physical preservation. • Take care to avoid unintentional mistakes when labeling sample containers, completing other documentation, and handling and weighing samples.

6.3.1 Determining the Optimal Mass of a Sample

As part of the DQO Process (Step 4 - Define the Boundaries), we recommend that you determine the appropriate size (i.e., the mass or volume), shape, and orientation of the primary field sample. For heterogeneous materials, the size, shape, and orientation of each field sample will affect the analytical result. To determine the optimal mass (or weight) of samples to be collected in the field, you should consider several key factors:

- The number and type of chemical and/or physical analyses to be performed on each sample, including extra volumes required for QA/QC. (For example, SW-846 Method 1311 (TCLP) specifies the minimum sample mass to be used for the extraction.)

- Practical constraints, such as the available volume of the material and the ability to collect, transport, and store the samples

- The characteristics of the matrix (such as particulate solid, sludge, liquid, debris, oily waste, etc.)
- Health and safety concerns (e.g., acutely toxic, corrosive, reactive, or ignitable wastes should be transported and handled in safe quantities)
- Availability of equipment and personnel to perform particle-size reduction (if needed) in the field rather than within a laboratory.

Often, the weight (or mass) of a field sample is determined by "whatever will fit into the jar." While this criterion may be adequate for some wastes or media, it can introduce serious biases – *especially in the case of sampling particulate solids*.

If a sample of particulate material is to be representative, then it needs to be representative of the largest particles of interest (Pitard 1993). This is relevant if the constituent of concern is not uniformly distributed across all the particle size fractions. To obtain a sample representative of the largest particles of interest, the sample must be of sufficient weight (or mass) to control the amount of fundamental error introduced during sampling.

If the constituent(s) of concern is uniformly distributed throughout all the particle size fractions, then determination of the optimal sample mass using Gy's approach will not improve the representativeness of the sample. Homogeneous or uniform distribution of contaminants among all particle sizes, however, is not a realistic assumption, especially for contaminated soils. In contaminated soils, concentrations of metals tend to be higher in the clay- and silt-size fractions and organic contaminants tend to be associated with organic matter and fines in the soil.

The following material provides a "rule of thumb" approach for determining the particle-size sample-weight relationship sufficient to maintain fundamental error (as measured by the standard deviation of the fundamental error) within desired limits. A detailed quantitative method is presented in Appendix D. Techniques for calculating the variance of the fundamental error also are presented in Mason (1992), Pitard (1993), Myers (1997), and Gy (1998).

The variance of the fundamental error (s_{FE}^2) is directly proportional to the size of the largest particle and inversely proportional to the mass of the sample.[2] To calculate the appropriate mass of the sample, Pitard (1989) proposed a "Quick Safety Rule" for use in environmental sampling based on a standard deviation of the fundamental error of 5 percent ($s_{FE} = \pm 5\%$):

$$\mathrm{M_S} \geq 10000d^3 \qquad \textbf{Equation 18}$$

where $\mathrm{M_S}$ is the mass of the sample in grams (g) and d of the diameter of the largest particle in centimeters (cm).

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[2] In this section, we use the "relative variance" ($s^2 / \bar{x}^2$) and the "relative standard deviation" ($s / \bar{x}$). The values are dimensionless and are useful for comparing results from different experiments.

Alternatively, if we are willing to accept $s_{FE} = \pm 16\%$, we can use

$$M_S \geq 1000d^3$$ **Equation 19**

An important feature of the fundamental error is that it does not "cancel out." On the contrary, the variance of the fundamental error adds together at each stage of subsampling. As pointed out by Myers (1997), the fundamental error quickly can accumulate and exceed 50 percent, 100 percent, 200 percent, or greater unless it is controlled through particle-size reduction *at each stage of sampling and subsampling*. The variance, s_{FE}^2 , calculated at each stage of subsampling and particle-size reduction, must be added together at the end to derive the total s_{FE}^2 . A example of how the variances of the fundamental error can be added together is provided in Appendix D.

6.3.2 Obtaining the Correct Shape and Orientation of a Sample

When sampling heterogeneous materials, the shape and orientation of the sampling device can affect the composition of the resulting samples and facilitate or impede achievement of DQOs. The following two subsections provide guidance on selecting the appropriate shape and orientation of samples obtained from a moving stream of material and a stationary batch or unit of material.

6.3.2.1 Sampling of a Moving Stream of Material

In sampling a moving stream of material, such as solids, liquids, and multi-phase mixtures moving through a pipe, on a conveyor, etc., the material can be treated as a one-dimensional mass. That is, the material is assumed to be linear in time or space.

The correct or "ideal" sample is an undisturbed cross section delimited by two parallel planes (Pitard 1993, Gy 1998). The approach is depicted in Figure 26 in which all of the flow is collected for part of the time. In practice, the condition can be met by using "cross-stream" sampling devices positioned at the discharge of a conveyor, hose, duct, etc. (Pitard 1993). Alternatively, in sampling solids from a conveyor belt, a transverse cutter or flat scoop (with vertical sides) can be used to obtain a sample, preferably with the conveyor stopped (though this condition may not be practical for large industrial conveyors).

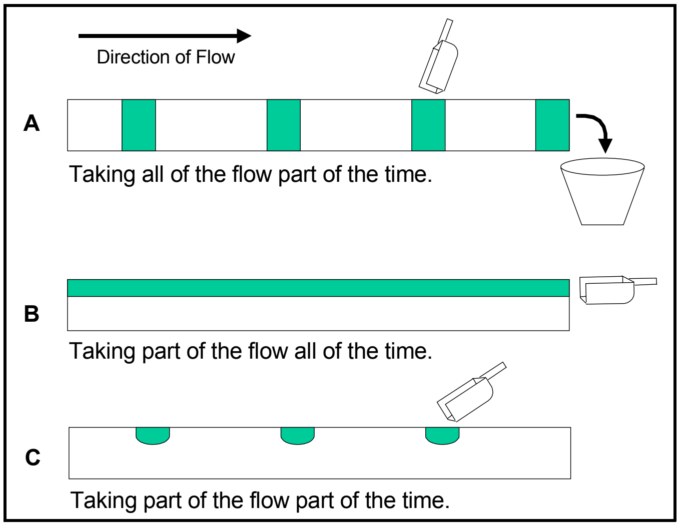

Figure 26. Three ways of obtaining a sample from a moving stream. "A" is correct. "B" and "C" will obtain biased samples unless the material is homogeneous (modified after Gy 1998).

For sampling of liquids, if the entire stream cannot be obtained for a fraction of the time (e.g., at the discharge point), then it may be necessary to introduce turbulence in the stream using baffles and to obtain a portion of the mixed stream part of the time (Pitard 1993).

6.3.2.2 *Sampling of a Stationary Batch of Material*

Sampling of a stationary batch of material, such as filter cake in a roll-off bin, soil in a drum, or liquid in a tank can be approached by viewing the three-dimensional space as a series of overlapping two-dimensional (i.e., relatively flat) masses in a horizontal plane. The correct or “ideal” sample of a is a core that obtains the full thickness of the material of interest.

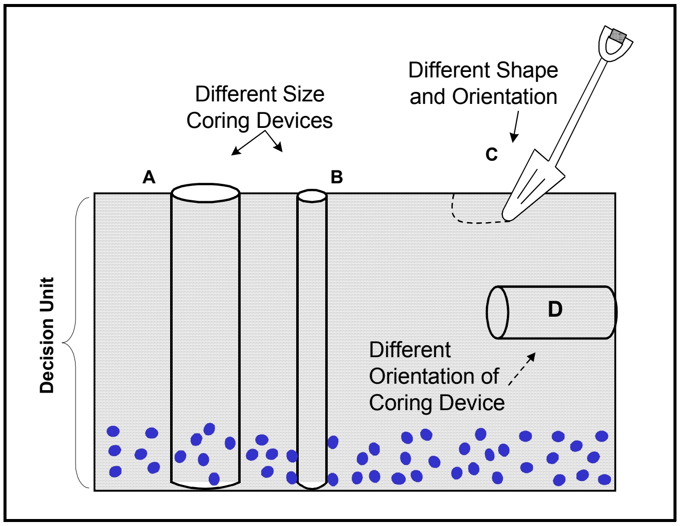

Figure 27. Sampling a three-dimensional waste by treating the sampling problem as a series of overlapping two-dimensional wastes. Only device “A” provides the correct size, shape, and orientation of the sample.

For example, Figure 27 shows a bin of granular waste with fine grain material in the upper layer and larger fragments in the bottom layer. The entire batch of material is the “decision unit.” Coring device “A” is correct: it is wide enough and long enough to include the largest fragments in the waste. Coring device “B” is too narrow. It either fails to capture the larger particles or simply pushes them out of the way (causing *increment delimitation error*). Device “C,” a trowel or small shovel, can collect an adequate volume of sample, but it preferentially selects only the finer grained material near the top of the bin. Device “D” is the correct shape, but it is not in the correct orientation. Devices “B,” “C,” and “D” yield incorrect sample support.

6.3.3 Selecting Sampling Devices That Minimize Sampling Errors

As part of the project planning process, you should establish performance goals for the sampling devices to be used and understand the possible limitations of any candidate sampling devices or equipment. The performance goals can then be used to select specific sampling devices or technologies with a clear understanding of the limitations of those devices in the field. Detailed guidance on the selection of specific sampling devices is provided in Section 7 and Appendix E of this document.

6.3.3.1 *General Performance Goals for Sampling Tools and Devices*

Selection of the appropriate sampling device and sampling method will depend on the sampling objectives, the physical characteristics of the waste or media, the chemical constituents of concern, the sampling location, and practical concerns such as technology limitations and safety issues (see also Section 7). The following general performance goals apply to the selection of sampling devices for use in those situations in where it is desirable to control or otherwise minimize biases introduced by the sampling device:

- The device should not include or exclude portions of the waste that do not belong in the sample (in other words, the device should minimize delimitation and extraction errors).

- If volatile constituents are of interest, the device should obtain samples in an undisturbed state to minimize loss of volatile constituents.

- The device should be constructed of materials that will not alter analyte concentrations due to loss or gain of analytes via sorption, desorption, degradation, or corrosion.

- The device should retain the appropriate size (volume or mass) and shape of sample, and obtain it in the orientation appropriate for the sampling condition -- preferably in one pass.

Other considerations not related to performance follow:

- "Ease of use" of the sampling device under the conditions that will be encountered in the field. This includes the ease of shipping to and from the site, ease of deployment, and ease of decontamination.

- The degree of hazard associated with the deployment of one sampling device versus another (e.g., consider use of an extension pole instead of a boat to sample from a waste lagoon).

- Cost of the sampling device and of the labor (e.g., single vs. multiple operators) for its deployment (including training) and maintenance.

6.3.3.2 Use and Limitations of Common Devices

Unfortunately, many sampling devices in common use today lack the properties required to minimize certain types of sampling error. In fact, there are few devices available that satisfy all the general performance goals stated above. Pitard (1993), however, has identified a number of devices that can help minimize delimitation and extraction error (depending on the physical form of the waste to be sampled). These devices include:

- COLIWASA (or “composite liquid waste sampler”) -- for sampling free-flowing liquids in drums or containers

- Shelby tube or similar device -- for obtaining core samples of solids

- Kemmerer depth sampler -- for obtaining discrete samples of liquids

- Flat scoop (with vertical walls) -- for subsampling solids on a flat surface.

Some devices in common use that can cause delimitation and extraction errors include the following: auger, shovel, spoon, trowel, thief, and trier. In spite of the limitations of many conventional sampling devices, it is necessary to use them under some circumstances encountered in the field because there are few alternatives. When selecting a sampling tool, choose the one that will introduce the least sampling error. In cases in which no such tool exists, document the approach used and be aware of the types of errors likely introduced and their possible impact on the sampling results. To the extent possible and practicable, minimize sampling errors by applying the concepts presented in this chapter.

6.3.4 Special Considerations for Sampling Waste and Soils for Volatile Organic Compounds

In most contaminated soils and other solid waste materials, volatile organic compound (VOCs), when present, coexist in gaseous, liquid, and solid (sorbed) phases. Of particular concern with regard to the collection, handling, and storage of samples for VOC characterization is the retention of the gaseous component. This phase exhibits molecular diffusion coefficients that allow for the immediate loss of gas-phase VOCs from a freshly exposed surface and continued losses from well within a porous matrix. Furthermore, once the gaseous phase becomes depleted, nearly instantaneous volatilization from the liquid and sorbed phases occurs in an attempt to restore the temporal equilibrium that often exists, thereby allowing the impact of this loss mechanism to continue.

Another mechanism that can influence VOC concentrations in samples is biological degradation. In general, this loss mechanism is not expected to be as large a source of determinate error as volatilization. This premise is based on the observation that losses of an order of magnitude can occur on a time scale of minutes to hours due solely to diffusion and advection, whereas losses of a similar magnitude due to biological processes usually require days to weeks. Furthermore, under aerobic conditions, which is typical of most samples that are transported and stored, biological mechanisms favor the degradation of aromatic hydrocarbons over halogenated compounds. Therefore, besides the slower rate of analyte loss, biodegradation is compound selective.

To limit the influence of volatilization and biodegradation losses, which, if not addressed can biased results by one or more orders of magnitude, it is currently recommended that sample collection and preparation, however not necessarily preservation, follow one or the other of these two protocols:

- The immediate in-field transfer of a sample into a weighed volatile organic analysis vial that either contains VOC-free water so that a vapor partitioning (purge-and-trap or headspace) analysis can be performed without reopening or that contains methanol for analyte extraction in preparation for analysis, or

- The collection and up to 2-day storage of intact samples in airtight containers before initiating one of the aforementioned sample preparation procedures.

In both cases, samples should be held at 4±2 °C while being transported from the sampling location to the laboratory.

The *Standard Guide for Sampling Waste and Solids for Volatile Organics* (ASTM D 4547-98) is recommended reading for those unfamiliar with the many challenges associated with collecting and handling samples for VOC analysis.

7 IMPLEMENTATION: SELECTING EQUIPMENT AND CONDUCTING SAMPLING

This section provides guidance on selecting appropriate sampling tools and devices (Section 7.1), conducting field sampling activities (Section 7.2), and using sample homogenization, splitting, and subsampling techniques (Section 7.3).

7.1 Selecting Sampling Tools and Devices

For additional guidance on the selection and use of sampling tools and devices, see:

- 40 CFR 261, Appendix I, Representative Sampling Methods
- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities* (ASTM D 6232)

The tools, devices, and methods used for sampling waste materials will vary with the form, consistency, and location of the waste materials to be sampled. As part of the DQO Process, you identify the location (type of unit or other source description) from which the samples will be obtained and the “dimension” of the sampling problem (such as “one-dimensional” or “two-dimensional”). In the DQO Process, you also specify the appropriate size, shape, orientation and other characteristics for each sample (called the “sample support”). In addition to the DQOs for the sample, you will identify performance goals for the sampling device. You may need a device that meets the following qualifications:

- Minimizes delimitation and extraction errors so that it does not include material that should not be in the sample, nor exclude material that should be in the sample
- Provides a largely undisturbed sample (e.g., one that minimizes the loss of volatile constituents, if those are constituents of concern)
- Is constructed of materials that are compatible with the media and the constituents of concern (e.g., the materials of construction do not cause constituent loss or gain due to sorption, desorption, degradation, or corrosion)
- Is easy to use under the conditions of the sampling location, and the degree of health or safety risks to workers is minimal
- Is easy to decontaminate
- Is cost-effective during use and maintenance.

Unfortunately, few devices will satisfy all of the above goals for a given waste or medium and sampling design. When selecting a device, try first to choose one that will introduce the least sampling error and satisfy other performance criteria established by the planning team, within practical constraints.

Figure 28 summarizes the steps you can use to select an optimal device for obtaining samples.

Using the outputs from the DQO Process, a description of the medium to be sampled, and knowledge of the site or location of sample collection, Tables 8 and 9 (beginning on pages 109 and 115 respectively) can be used to quickly identify an appropriate sampling device. For most situations, the information in the tables will be sufficient to make an equipment selection; however, if you need additional guidance, review the more detailed information provided in Appendix E or refer to the references cited.

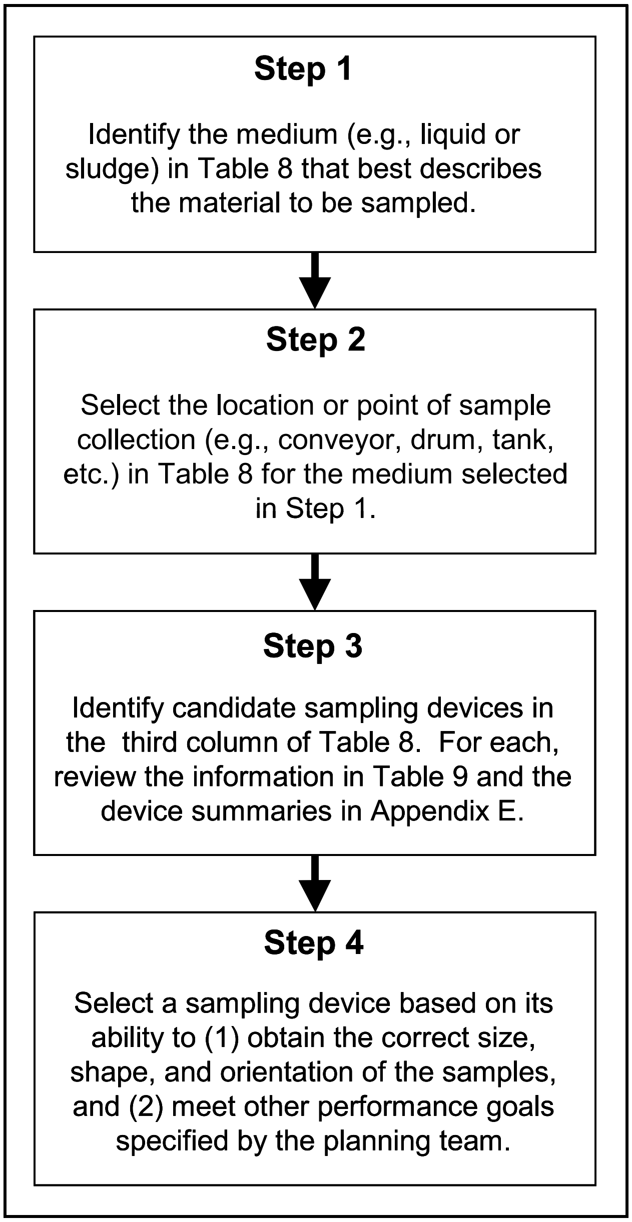

Figure 28. Steps for selecting a sampling device

If desired, you can refer to the documents (such as ASTM standards) referenced by Table 8 for supplementary guidance specific to sampling a specific medium and site, or refer to those referenced by Table 9 for supplementary guidance on a device.[1] The contents of the ASTM standards are summarized in Appendix J. (For more information on ASTM or purchasing their publications, including the standards referenced in this chapter, contact ASTM at: ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, or by telephone at 610-832-9585, via the World Wide Web at http://www.astm.org.)

In particular, we recommend that you review the guidance found in ASTM Standard D 6232, *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*. Most of the information on sampling devices found in this chapter and in Tables 8 and 9 came from that standard. As noted by the standard, it covers criteria that should be considered when selecting sampling equipment for collecting environmental and waste samples for waste management activities. It also describes many of the typical devices used during such sampling.

Because each sampling situation is unique, the guidance in this chapter may not adequately cover your specific sampling scenario. You may have to modify a part of the device or modify the device application to improve its performance or to facilitate sample collection. For

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[1] ASTM is a consensus standards development organization. Consistent with the provisions of the National Technology Transfer and Advancement Act of 1995 (NTTAA), Public Law 104-113, Section 12(d), which directs EPA to use voluntary consensus standards to the extent possible, this guidance supports the use of and provides references to ASTM standards applicable to waste sampling.

example, you might use a rope or an extension handle on a device to access a particular location within a waste management unit. In other cases, you may need auxiliary equipment that will increase the cost or complexity of sampling operation (such as a drill rig to drive a split barrel sampler or a power supply to run a pump). The physical state of the waste or design of the unit also may affect how the equipment is deployed. You should address such variations as part of your sampling plan and make sure that any modifications do not cause sampling bias.

Finally, other sampling devices not addressed in this chapter can and should be used if appropriate (e.g., if the device meets the performance goals and is more practical). New or innovative devices not discussed in this chapter also should be considered for use if they allow you to meet the sampling objectives in a more cost-effective manner. In other words, we encourage and recommend a performance-based approach for selecting sampling equipment.

7.1.1 Step 1: Identify the Waste Type or Medium to be Sampled

The first column of Table 8 (page 109) lists the media type or waste matrix commonly sampled under RCRA. These media may include liquids, sludges or slurries, various unconsolidated solids, consolidated solids and debris, soil, ground water, sediment, soil gas, and air. In general, the types of media describe the physical state of the material to be sampled. The physical characteristics of the waste or medium affect many aspects of sampling, including the volume of material required, selection of the appropriate sampling device, how the device is deployed, and the containers used for the samples. Table 10 provides an expanded description of the media listed in Table 8.

7.1.2 Step 2: Identify the Site or Point of Sample Collection

In the second column of Table 8, identify the site or point of sample collection that best describes where you plan to obtain the samples. The “site or point of sample collection” may include (1) the point at which the waste is generated (e.g., as the waste exits a pipe, moves along a conveyor, or is poured or placed into a container, tank, impoundment or other waste management unit); (2) the unit in which the waste is stored (such as a drum, collection hopper, tank, waste pile, surface impoundment, sack or bag) or transported (such as a drum, tanker truck, or roll-off box); or (3) the environmental medium to be sampled (such as surface soil, subsurface soil, ground water, surface water, soil gas, or air).

When testing a solid waste to determine if it should be characterized as a hazardous waste or to determine if the waste is restricted from land disposal, such a determination must be made at the **point of waste generation**.

7.1.2.1 Drums and Sacks or Bags

Drums and sacks or bags are portable containers used to store, handle, or transport waste materials and sometimes are used in waste disposal (e.g., drums in a landfill). “Drums” include metal drums and pails, plastic drums, or durable fiberboard paper drums or pails (USEPA 1994a). Drums and pails may contain nearly the full range of media -- liquids (single or multi-layered), sludges, slurries, or solids. Sacks or bags include less rigid portable containers and thus can contain only solids. The sampling approach (including number of samples, locations of samples, sampling device, depth of samples) for these containers will depend on the number of containers to be sampled, waste accessibility, physical and chemical characteristics of the waste, and component distribution within the containers.

Review ASTM Standards D 6063, *Guide for Sampling Drums and Similar Containers by Field Personnel*, and D 5679, *Practice for Sampling Consolidated Solids in Drums or Similar Containers*, for more information on the sampling of drums and sacks or bags. Other useful guidance on sampling drums includes "Drum Sampling" (USEPA 1994b), issued by EPA's Environmental Response Team.

7.1.2.2 Surface Impoundments

Surface impoundments include natural depressions, manmade excavations, or diked areas that contain an accumulation of liquids or wastes containing free liquids and solids. Examples of surface impoundments are ponds, lagoons, and holding, storage, settling, and aeration pits (USEPA 1994a). The appropriate sampling device for sampling a surface impoundment will depend on accessibility of the waste, the type and number of phases of the waste, the depth, and chemical and physical characteristics of the waste.

7.1.2.3 Tanks

A tank is defined at § 260.10 as a stationary device, designed to contain an accumulation of hazardous waste which is constructed primarily of non-earthen materials which provide structural support. A container is defined at § 260.10 as a portable device, in which a material is stored, transported, treated, disposed of, or otherwise handled. The distinction that a tank is not a container is important because the regulations at 261.7 set forth conditions to distinguish whether hazardous waste in a container is subject to regulation. Nevertheless, for the purpose of selecting an appropriate sampling device, the term "tank" as used in Table 8 could include other units such as tank trucks and tanker cars even though they are portable devices.

The selection of equipment for sampling the pipes and sampling ports of a tank system is covered separately under those categories. The equipment used to sample a pipe or spigot can be very different from that used to sample an open tank.

Tanks usually contain liquids (single or multi-layered), sludges, or slurries. In addition, suspended solids or sediments may have settled in the bottom of the tank. When sampling from a tank, one typically considers how to acquire a sufficient number of samples from different locations (including depths) to adequately represent the entire content of the tank.

Waste accessibility and component distribution will affect the sampling strategy and equipment selection. In addition to discharge valves near the bottom, most tanks have hatches or other openings at the top. It is usually desirable to collect samples via a hatch or opening at the top of the tank because of the potential of waste stratification in the tank (USEPA 1996b). In an open tank, the size of the tank may restrict sampling to the perimeter of the tank. Usually, the most appropriate type of sampling equipment for tanks depends on the design of the tanks and the media contained within the tank.

You can find additional guidance on sampling tanks in "Tank Sampling" (USEPA 1994c), issued by the EPA's Environmental Response Team.

7.1.2.4 Pipes, Point Source Discharges, or Sampling Ports

For the purpose of this guidance, pipes or point source discharges include moving streams of sludge or slurry discharging from a pipe opening, sluice, or other discharge point (such as the point of waste generation). Sampling ports include controlled liquid discharge points that were installed for the purpose of sampling, such as may be found on tank systems, a tank truck, or leachate collection systems at waste piles or landfills.

A dipper also is used to sample liquids from a sampling port. Typically, it is passed through the stream in one sweeping motion so that it is filled in one pass. In that instance, the size of the dipper beaker should be related to the stream flow rate. If the cross-sectional area of the stream is too large, more than one pass may be necessary to obtain a sample (USEPA 1993b). Besides the use of a dipper or other typical sampling devices, sometimes the sample container itself is used to sample a spigot or point source discharge. This eliminates the possibility of contaminating the sample with intermediate collection equipment, such as a dipper (USEPA 1996b).

See ASTM D 5013-89 *Standard Practices for Sampling Wastes from Pipes and Other Point Discharges* for more information on sampling at this location. Also see Gy (1998) and Pitard (1989, 1993).

7.1.2.5 Storage Bins, Roll-Off Boxes, or Collection Hoppers

Discharges of unconsolidated solids from a process, such as filter cakes, often fall from the process into a collection hopper or other type of open-topped storage container. Sometimes the waste materials are combined into large a storage bin, such as a roll-off box or collection hopper. A storage bin also may be used to collect consolidated solids, such as construction debris. The waste can be sampled either as it is placed in the container or after a certain period of accumulation, depending on the technical and regulatory objectives of the sampling program.

7.1.2.6 Waste Piles

Waste piles include the non-containerized accumulation of solid and nonflowing waste material on land. The size of waste piles can range from small heaps to large aggregates of wastes. Liners may underlie a waste pile, thereby preventing direct contact with the soil. As with other scenarios, waste accessibility and heterogeneity will be key factors in the sampling design and equipment selection. Besides the devices listed in this chapter, excavation equipment may be needed at first to properly sample large piles. Waste piles may present unique sample delimitation problems (Pitard 1993 and Myers 1997), and special considerations related to sampling design may be necessary (such as the need to flatten the pile).

We recommend a review of ASTM Standard D 6009, *Guide for Sampling Waste Piles* for more information. Another source of information on sampling waste piles is "Waste Pile Sampling" (USEPA 1994d), issued by EPA’s Environmental Response Team.

7.1.2.7 Conveyors

Solid process discharges are sometimes sampled from conveyors such as conveyor belts or screw conveyors. Conveyor belts are open moving platforms used to transport material between locations. Solid or semi-solid wastes on a conveyor belt can be sampled with a flat scoop or similar device (see also Section 6.3.2.1). Screw conveyors usually are enclosed systems that require access via a sampling port, or they can be sampled at a discharge point. See also ASTM D 5013 and Gy (1998, pages 43 through 56).

7.1.2.8 Structures and Debris

This guidance assumes that the sampling of structure or debris typically will include the sampling of consolidated solids such as concrete, wood, or other structure debris. Appendix C provides supplemental guidance on developing a sampling strategy for such heterogeneous wastes. See also AFCEE (1995), Koski, et al. (1991), Rupp (1990), USEPA and USDOE (1992), and ASTM Standard D 5956, *Standard Guide For Sampling Strategies for Heterogeneous Wastes*.

7.1.2.9 Surface or Subsurface Soil

Selection of equipment for sampling soil is based on the depth of sampling, the grain-size distribution, physical characteristics of the soil, and the chemical parameters of interest (such as the need to analyze the samples for volatiles). Your sampling strategy should specify the depth and interval (e.g., “0 to 6 inches below ground surface”) of interest for the soil samples.

Simple manual techniques and equipment can be used for surface or shallow depth sampling. To obtain samples of soil from greater depths, powered equipment (e.g., power augers or drill rigs) will be required; however, those are not used for actual sample collection, but are used solely to gain easier access to the required sample depth (USEPA 1996b). Once at the depth, surface sampling devices may be used.

ASTM has developed many informative standards on the sampling of soil, including D 4700, *Standard Guide for Soil Sampling from the Vadose Zone*, and D 4220, *Standard Practices for Preserving and Transporting Soil Samples*. In addition, see EPA-published guidance such as *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (Mason 1992) and *Description and Sampling of Contaminated Soils - A Field Pocket Guide* (USEPA 1991b).

7.1.3 Step 3: Consider Device-Specific Factors

After you identify the medium and site of sample collection, refer to the third column of Table 8 for the list of candidate sampling devices. We listed common devices that are appropriate for the given media and site. Next, refer to the information in Table 9 for each of the candidate devices to select the most appropriate one for your sampling effort.

Table 9 provides device-specific information to help you choose the appropriate device based on the study objective and the DQOs established for volume (size), shape, depth, and orientation of the sample, and sample type (discrete or composite, surface or at depth).

For easy reference, the devices are listed alphabetically in Table 9. Appendix E contains a summary description of key features of each device and sources for other information. Under the third column in Table 9, “Other Device-Specific Guidance,” we have identified some of those sources, especially relevant ASTM standards (see summaries of ASTM standards in Appendix J).

7.1.3.1 Sample Type

The column “Sample Type” Table 9 identifies whether the device can sample at surface only, shallow or at a deeper profile (depth), and whether the device can obtain a discrete sample or a composite sample. For example, a COLIWASA or drum thief can be used to sample a container that is 3-feet deep, but a Kemmerer sampler may be required to sample the much deeper depth of an impoundment. We also identify in this column whether the device collects a undisturbed or disturbed solid sample. Also, the actual depth capacity may depend on the design of the device. Some devices can be modified or varied to collect at different depths or locations in a material. You should refer to the device summary in Appendix E if you need specifics regarding the sampling depth available for a given device.

7.1.3.2 Sample Volume

The column for volume in Table 9 identifies the range of sample volume, in liters, that the device can obtain. It may be possible to increase or decrease this value through modification of the device. During the planning process, you should determine the correct volume of sample needed. Volume is one of the components of sample “support” (that is, the size, shape, and orientation of the sample).

7.1.3.3 Other Device-Specific Considerations

The last column of Table 9 notes other considerations for device selection. The comments focus on those factors that may cause error to be introduced or that might increase the time or cost of sampling. For some devices, the column includes comments on how easy the equipment is to use, such as whether it needs a power source or is heavy, and whether it can be decontaminated easily. The table also mentions whether the device is appropriate for samples requiring the analysis of volatile organic constituents and any other important considerations regarding analyte and device compatibility. The equipment should be constructed of materials that are compatible with the waste and not susceptible to reactions that might alter or bias the physical or chemical characteristics of the sample of the waste.

7.1.4 Step 4: Select the Sampling Device

Select the sampling device based on its ability to (1) obtain the correct size, shape, and orientation of the samples (see Sections 6.3.1 and 6.3.2) and (2) meet any other performance criteria specified by the planning team in the DQO Process (see Section 6.3.3.1). In addition, samples to be analyzed for volatile organic constituents should be obtained using a sampling technique that will minimize the loss of constituents and obtain a sample volume required for the analytical method (see Section 6.3.4).

Table 8. Device Selection Guide -- Media and Site of Sample Collection

Media (See Section 7.1.1)	***Site or Point of Sample Collection (See Section 7.1.2)***	***Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)***	***Other Related Guidance***
Liquids, no distinct layer of interest Examples: Containerized spent solvents, leachates or other liquids discharged from a pipe or spigot	Drum	COLIWASA Dipper Drum thief Liquid grab sampler Peristaltic pump Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler Valved drum sampler	ASTM D 5743 ASTM D 6063 EPA/ERT SOP 2009 (USEPA 1994b)
	Surface impoundment	Automatic sampler Bacon bomb Bailer Bladder pump Centrifugal sub-pump Dipper Displacement pump Kemmerer sampler Liquid grab sampler Peristaltic pump Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler	ASTM D 6538 USEPA (1984, 1985, and 1989c)
	Tank	Bacon bomb Bailer COLIWASA Dipper Drum thief Kemmerer sampler Liquid grab sampler Peristaltic pump Plunger type sampler Settleable solids profiler Submersible pump Swing jar sampler Syringe sampler	ASTM D 6063 ASTM D 5743 EPA/ERT SOP 2010 (USEPA 1994c)

* Copies of EPA/ERT SOPs are available on the Internet at http://www.ert.org/

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

Media (See Section 7.1.1)	*Site or Point of Sample Collection (See Section 7.1.2)*	*Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)*	*Other Related Guidance*
Liquids, no distinct layer of interest (continued)	Pipe, point source discharge	Automatic sampler Bladder pump Centrifugal submersible pump Dipper Displacement pump Liquid grab sampler Plunger type sampler Sample container Swing jar sampler	ASTM D 5013 ASTM D 5743 ASTM D 6538 Gy 1998
	Sampling port (e.g., spigot)	Beaker, bucket, sample container Swing jar sampler	Gy 1998
Liquids, multi-layered, with one or more distinct layers of interest Examples: Non-aqueous phase liquids (NAPLs) in a tank; mixtures of antifreeze in a tank.	Drum	COLIWASA Discrete level sampler Drum thief Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler Valved drum sampler	ASTM D 6063
	Surface impoundment	Automatic sampler Bacon bomb Bailer (point source bailer) Bladder pump Centrifugal submersible pump Discrete level sampler Displacement pump Peristaltic pump Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler	ASTM D 6538 USEPA (1989c)
	Tank	COLIWASA Centrifugal submersible pump Bacon bomb Bailer Discrete level sampler Peristaltic pump Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler Valved drum sampler	ASTM D 6063 ASTM D 5743 EPA/ERT SOP 2010 (USEPA 1994c)

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

Media (See Section 7.1.1)	***Site or Point of Sample Collection (See Section 7.1.2)***	***Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)***	***Other Related Guidance***
Sludges, slurries, and solid-liquid suspensions Examples: Paint sludge, electroplating sludge, and ash and water slurry.	Drum	COLIWASA Dipper Liquid grab sampler Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler	ASTM D 6063
	Tank	COLIWASA Dipper Lidded sludge/water sampler Liquid grab sampler Plunger type sampler Ponar dredge Settleable solids profiler Swing jar sampler Syringe sampler	ASTM D 6063 EPA/ERT 2010 (USEPA 1994c)
	Surface impoundment	Dipper Lidded sludge/water sampler Liquid grab sampler Peristaltic pump Plunger type sampler Ponar dredge Settleable solids profiler Swing jar sampler	USEPA (1989c)
	Pipe or conveyor	Dipper or bucket Scoop/trowel/shovel Swing jar sampler	ASTM D 5013
Granular solids – unconsolidated Examples: Filter press cake, powders, excavated (*ex situ*) soil, incinerator ash	Drum	Bucket auger Coring type sampler (w/valve) Miniature core sampler Modified syringe sampler Trier Scoop/trowel/shovel	ASTM D 5680 ASTM D 6063 EPA/ERT SOP 2009 (USEPA 1994b)
	Sack or bag	Concentric tube thief Miniature core sampler Modified syringe sampler Scoop/trowel/shovel Trier	ASTM D 5680 ASTM D 6063

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

Media (See Section 7.1.1)	***Site or Point of Sample Collection (See Section 7.1.2)***	***Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)***	***Other Related Guidance***
Granular solids – unconsolidated (continued)	Storage bin, roll-off box, or collection hopper	Bucket auger Concentric tube thief Coring type sampler (w/valve) Miniature core sampler Modified syringe sampler Scoop/trowel Trier	ASTM D 5680 ASTM D 6063
	Waste pile	Bucket auger Concentric tube thief Coring type sampler (w/valve) Miniature core sampler Modified syringe sampler Scoop/trowel/shovel Thin-walled tube Trier	ASTM D 6009 EPA/ERT SOP 2017 (USEPA 1994d)
	Pipe (e.g., vertical discharge from cyclone centrifuge or baghouse) or conveyor	Bucket, dipper, pan, or sample container Miniature core sampler Scoop/trowel/shovel Trier	ASTM D 5013 Gy (1998) Pitard (1993)
Other solids – unconsolidated Examples: Waste pellets, catalysts, or large-grained solids.	Drum	Bucket auger Scoop/trowel/shovel	ASTM D 5680 ASTM D 6063 EPA/ERT SOP 2009 (USEPA 1994b)
	Sack or bag	Bucket auger Scoop/trowel/shovel	ASTM D 5680 ASTM D 6063
	Storage bin, roll-off box, or collection hopper	Bucket auger Scoop/trowel/shovel	ASTM D 5680 ASTM D 6063
	Waste pile	Bucket auger Scoop/trowel/shovel Split barrel Thin-walled tube	ASTM D 6009 EPA/ERT SOP 2017 (USEPA 1994d)
	Conveyor	Scoop/trowel/shovel	ASTM D 5013 Gy (1998) Pitard (1993)

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

***Media* (See Section 7.1.1)**	***Site or Point of Sample Collection (See Section 7.1.2)***	***Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)***	***Other Related Guidance***
Soil and other unconsolidated geologic material Examples: *In situ* soil at a land treatment unit or *in situ* soil at a SWMU	Surface	Bucket auger Concentric tube thief Coring type sampler Miniature core sampler Modified syringe sampler Penetrating probe sampler Scoop/trowel/shovel Thin-Walled Tube Trier	ASTM D 5730 ASTM E 1727 ASTM D 4700 EISOPQA Manual (USEPA 1996b)
	Subsurface	Bucket auger Coring type sampler Miniature core sampler Mod. syringe sampler Penetrating probe sampler Shovel/scoop/shovel Split barrel Thin-walled tube	ASTM D 4700 ASTM D 5730 ASTM D 6169 ASTM D 6282 USEPA (1996b) USEPA (1993c)
Solids – consolidated Examples: Concrete, wood, architectural debris*	Storage bin (e.g., roll-off box)	Penetrating probe sampler Rotating coring device	ASTM D 5679 ASTM D 5956 ASTM D 6063 USEPA and USDOE (1992)
	Waste pile	Penetrating probe sampler Rotating coring device Split barrel	ASTM D 6009 USEPA and USDOE (1992)
	Structure	Rotating coring device (See also Appendix C, Section C.5)	AFCEE (1995) Koski, et al (1991) USEPA and USDOE (1992)

* The term “debris” has a specific definition under 40 CFR 268.2(g) (Land Disposal Restrictions regulations) and includes “solid material exceeding a 60 mm particle size that is intended for disposal and that is a manufactured object; or plant or animal matter; or natural geologic material.” § 268.2(g) also identifies materials that are not debris. In general, debris includes materials of either a large particle size or variation in the items present.

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

Selected References for Sampling of Other Media	
Air Example: BIF emissions	Chapter Ten SW-846 EISOPQA Manual (USEPA 1996b)
Sediment Example: Surface impoundment sediment	*QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations* (USEPA 1995d) *Superfund Program Representative Sampling Guidance Volume 5; Water and Sediment, Part I – Surface Water and Sediment, Interim Final Guidance* (USEPA 1995e) Region 4 EISOPQA Manual (USEPA 1996b) *Sediment Sampling* (USEPA 1994e) ASTM D 4823; ASTM D 5387
Soil Gas or Vapor Examples: Soil, soil water, or gas in the vadose zone at a waste disposal site	*Subsurface Characterization and Monitoring Techniques - A Desk Reference Guide* (USEPA 1993c) ASTM *Standard Guide for Soil Gas Monitoring in the Vadose Zone* (ASTM D 5314) *Soil Gas Sampling* (USEPA 1996c)
Ground Water Example: Ground-water monitoring wells at a landfill	*RCRA Ground-Water Monitoring Draft Technical Guidance* (USEPA 1992c) *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures* (Puls and Barcelona 1996) ASTM D4448-01 Standard Guide for Sampling Ground-Water Monitoring Wells ASTM D 5092-90 *Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers* ASTM D 6286-98 *Standard Guide for Selection of Drilling Methods for Environmental Site Characterization* ASTM D 6282 *Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations* ASTM D 6771-02 *Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations*

Table 9. Device Selection Guide – Device-Specific Factors

Sampling Device (Listed in Alphabetical Order)	***Description, Appendix E, Section No.***	***Other Device-Specific Guidance (in Addition to ASTM D 6232)***	***Sample Type***	***Volume (Liters per Pass)***	***Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)***
Automatic sampler	E.1.1	ASTM D 6538 EISOPQA Manual (USEPA 1996b)	Shallow (25 in.), discrete or composite	Unlimited	Auto samplers are available to collect samples for volatile organics analysis, provide a grab or composite sample, and may be unattended. Need power source/battery. Commonly used at waste water treatment plants. Must be knowledgeable of compatibility of waste and sampler components.
Bacon bomb	E.3.1	USEPA 1984 USEPA 1994c	Depth, discrete	0.1 to 0.5	For parameters that do not require a polytetrafluroethylene (PTFE) sampler. Recommended for sampling of lakes, ponds, large tanks, or lagoons. May be difficult to decontaminate and materials of construction may not be compatible with sample matrix.
Bailer	E.7.1	ASTM D 4448 USEPA 1992c USEPA 1994c	Depth, discrete	0.5 to 2.0	Bailers are not recommended for sampling ground water for trace constituent analysis due to sampling induced turbidity (USEPA 1992c and Puls and Barcelona 1996). Unable to collect samples from specific depths (unless a point-source bailer is used). Available in a variety of sizes as either reusable or single use devices. May be chemically incompatible with certain matrices unless constructed of resistant material.
Bladder pump	E.1.2	ASTM D 4448 USEPA 1992c USEPA 1996b	Depth, discrete	Unlimited	For purging or sampling of wells, surface impoundments, or point discharges. Contact parts are made of PTFE, PVC and stainless steel. Requires a power source, compressed gas, and a controller. Difficult to decontaminate (based on design). Suitable for samples requiring VOAs. May require a winch or reel.
Bucket auger	E.5.1	ASTM D 1452 ASTM D 4700 ASTM D 6063 Mason 1992 USEPA 1993c	Surface or depth, disturbed	0.2 to 1.0	Easy and quick for shallow subsurface samples but not recommended for VOAs. Requires considerable strength and labor and destroys soil horizons.

Table 9. Device Selection Guide – Device-Specific Factors (Continued)

Sampling Device (listed in alphabetical order)	***Description, Appendix E, Section***	***Other Device-Specific Guidance (in addition to ASTM D 6232)***	***Sample Type***	***Volume (Liters Per Pass)***	***Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)***
Centrifugal submersible pump	E.1.4	ASTM D 4448 ASTM D 4700 USEPA 1992c	Depth, discrete	Unlimited	For purging or sampling wells, surface impoundments, or point discharges. Contact parts are made of PTFE and stainless steel. Requires a power source. Adjustable flow rate and easy to decontaminate. Not compatible with liquids containing high percent solids. May require a winch or reel.
COLIWASA	E.6.1	ASTM D 5495 ASTM D 5743 ASTM D 6063 USEPA 1980	Shallow, composite	0.5 to 3.0	Reusable and single use models available. Inexpensive. Glass type devices may be difficult to decontaminate. Collects undisturbed sample. For mixed solid/liquid media will collect semi-liquid only. Not for high viscosity liquids.
Concentric tube thief	E.4.3	ASTM D 6063 USEPA 1994d	Surface, relatively undisturbed, selective	0.5 to 1.0	Recommended for powdered or granular materials or wastes in piles or in bags, drums or similar containers. Best used in dry, unconsolidated materials. Not suitable for sampling large particles due to narrow width of slot.
Coring type sampler (with or without valve)	E.4.6	ASTM D 4823 USEPA 1989c	Surface or depth, disturbed	0.2 to 1.5	Designed for wet soils and sludge. May be equipped with a plastic liner and caps. May be used for VOAs. Reusable and easy to decontaminate.
Dipper (or “pond sampler”)	E.7.2	ASTM D 5358 ASTM D 5013 USEPA 1980	Shallow, composite	0.5 to 1.0	For sampling liquids in surface impoundments. Inexpensive. Not appropriate for sampling stratified waste if discrete characterization needed.
Discrete level sampler	E.3.5		Depth, discrete	0.2 to 0.5	Easy to decontaminate. Obtains samples from a discrete interval. Limited by sample volume and liquids containing high solids. Can be used to store and transport sample.
Displacement pumps	E.1.5	ASTM D 4448	Depth, discrete	Unlimited	Can be used for purging or sampling of wells, impoundments, or point discharges. Contact parts are made of PVC, stainless steel, or PTFE to reduce risk of contamination when trace levels or organics are of interest. Requires a power source and a large gas source. May be difficult to decontaminate (piston displacement type). May require a winch or reel to deploy.

Table 9. Device Selection Guide – Device-Specific Factors (Continued)

Sampling Device (listed in alphabetical order)	*Description, Appendix E, Section*	*Other Device-Specific Guidance (in addition to ASTM D 6232)*	*Sample Type*	*Volume (Liters Per Pass)*	*Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)*
Drum thief	E.6.2	ASTM D 6063 ASTM D 5743 USEPA 1994b	Shallow, composite	0.1 to 0.5	Usually single use. If made of glass and reused, decontamination may be difficult. Limited by length of sampler, small volume of sample collected, and viscosity of fluids.
Kemmerer sampler	E.3.2		Depth, discrete	1.0 to 2.0	Recommended for lakes, ponds, large tanks or lagoons. May be difficult to decontaminate. Materials may not be compatible with sample matrix but all PTFE construction is available. Sample container exposed to media at other depths while being lowered to sample point.
Lidded sludge/water sampler	E.3.4		Discrete, composite	1.0	1-L sample jar placed into device (low risk of contamination). May sample at different depths and samples up to 40-percent solids. Equipment is heavy and limited to one bottle size.
Liquid grab sampler	E.7.3		Shallow, discrete, composite-suspended solids only	0.5 to 1.0	For sampling liquids or slurries. Can be capped and used to transport sample. Easy to use. May be lowered to specific depths. Compatibility with sample parameters is a concern.
Miniature core sampler	E.4.7	ASTM D 4547 ASTM D 6418	Discrete	0.01 to 0.05	Used to retrieve samples from surface soil, trench walls, or sub-samples from soil cores. O-rings on plunger and cap minimize loss of volatiles and allow device to be used to transport sample. Designed for single use. Cannot be used on gravel or rocky soils must avoid trapping air with samples.
Modified syringe sampler	E.4.8	ASTM D 4547	Discrete	0.01 to 0.05	Made by modifying a plastic, medical, single-use syringe. Used to collect a sample from a material surface or to sub-sample a core. The sample is transferred to a vial for transportation. Inexpensive. Must ensure device is clean and compatible with media to be sampled.

Table 9. Device Selection Guide – Device-Specific Factors (Continued)

Sampling Device (listed in alphabetical order)	***Description, Appendix E, Section***	***Other Device-Specific Guidance (in addition to ASTM D 6232)***	***Sample Type***	***Volume (Liters Per Pass)***	***Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)***
Penetrating probe sampler	E.4.1	USEPA 1993c	Discrete, undisturbed	0.2 to 2.0	Used to sample soil vapor, soil, and ground water (pushed or hydraulically driven). Versatile, make samples available for onsite analysis and reduces investigation derived waste. Limited by sample volume and composition of subsurface material.
Peristaltic pump	E.1.3	ASTM D 4448 ASTM D 6063 USEPA 1996b	Shallow, discrete or composite-suspended solids only	Unlimited	Possible to collect samples from multiple depths up to 25 feet. Decontamination of pump is not required and tubing is easy to replace. Can collect samples for purgeable organics with modified equipment, but may cause loss of VOAs.
Plunger type sampler	E.6.4	ASTM D 5743	Surface or depth, discrete	0.2 to Unlimited	Made of high-density polyethylene (HDPE) or PTFE with optional glass sampling tubes. Used to collect a vertical column of liquid. Either a reusable or single use device. Decontamination may be difficult (with glass tubes).
Ponar dredge	E.2.1	ASTM D 4387 ASTM D 4342 USEPA 1994e	Bottom surface, rocky or soft, disturbed	0.5 to 3.0	One of the most effective samplers for general use on all types of substrates (silt to granular material). May be difficult to repeatedly collect representative samples. May be heavy.
Rotating coring device	E.5.2	ASTM D 5679	Surface or depth, undisturbed	0.5 to 1.0	May obtain a core of consolidated solid. Requires power and water source and is difficult to operate. Sample integrity may be affected.
Scoop	E.7.5	ASTM D 5633 ASTM D 4700 ASTM D 6063	Surface, disturbed, selective	<0.1 to 0.6	Usually for surface soil and solid waste samples. Available in different materials and simple to obtain. May bias sample because of particle size. May exacerbate loss of VOCs.
Settleable solids profiler	E.6.5		Depth, composite-suspended solids only	1.3 to 4.0	Typically used at waste water treatment plants, waste settling ponds, and impoundments to measure and sample settleable solids. Easy to assemble, reusable and unbreakable under normal use. Not recommended for caustics or high viscosity materials.

Table 9. Device Selection Guide – Device-Specific Factors (Continued)

Sampling Device (listed in alphabetical order)	*Description, Appendix E, Section*	*Other Device-Specific Guidance (in addition to ASTM D 6232)*	*Sample Type*	*Volume (Liters Per Pass)*	*Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)*
Shovel	E.7.5	ASTM D 4700	Surface, disturbed	1.0 to 5.0	Used to collect surface material or large samples from waste piles. Easy to decontaminate and rugged. Limited to surface use and may exacerbate the loss of samples for VOAs.
Split barrel sampler	E.4.2	ASTM D 1586 ASTM D 4700 ASTM D 6063	Discrete, undisturbed	0.5 to 30.0	May be driven manually, or mechanically by a drill rig with trained personnel. May collect a sample at depth. A liner may be used in the device to minimize disturbance or for samples requiring VOAs.
Swing jar sampler	E.7.4		Shallow, composite	0.5 to 1.0	Used to sample liquids, powders, or small solids at a distance up to 12 feet. Adaptable to different container sizes. Not suitable for discrete samples. Can sample a wide variety of locations.
Syringe sampler	E.3.3	ASTM D 5743 ASTM D 6063	Shallow, discrete, disturbed	0.2 to 0.5	Recommended for highly viscous liquids, sludges and tar-like substances. Easy to decontaminate. Obtains samples at discrete depths but limited to length of device. Waste must be viscous enough to stay in sampler.
Thin-walled tube	E.4.5	ASTM D 1587 ASTM D 4823 ASTM D 4700	Surface or depth, undisturbed	0.5 to 5.0	Useful for collecting an undisturbed sample (depends on extension). May require a catcher to retain soil samples. Inexpensive, easy to decontaminate. Samples for VOAs may be biased when sample is extruded.
Trier	E.4.4	ASTM D 5451 ASTM D 6063	Surface, relatively undisturbed, selective	0.1 to 0.5	Recommended for powdered or granular materials or wastes in piles or in bags, drums, or similar containers. Best for moist or sticky materials. Will introduce sampling bias when used to sample coarse-grained materials.
Trowel	E.7.5	ASTM D 5633 ASTM D 4700 ASTM D 6063	Surface, disturbed, selective	0.1 to 0.6	Usually for surface soil and solid waste samples. Available in different materials and simple to obtain. May bias sample because of particle size, and may exacerbate loss of VOAs.
Valved drum sampler	E.6.3		Shallow, composite	0.3 to 1.6	Used to collect a vertical column of liquid. Available in various materials for repeat or single use. High viscosity liquids may be difficult to sample.

Table 10. Descriptions of Media Listed in Table 8.

Media	***Description***	***Examples***
Liquids -- no distinct layer of interest	Liquids (aqueous or nonaqueous) that are or are not stratified and samples from discrete intervals are not of interest. Sampling devices for this medium do not need to be designed to collect liquids at discrete depths.	Containerized leachates or spent solvents; leachates or other liquids released from a spigot or discharged from a pipe.
Liquids -- one or more distinct layers of interest	Liquids (aqueous or nonaqueous) that are stratified with distinct layers and collection of samples from discrete intervals is of interest. Sampling devices for this media do need to be designed to collect liquids at discrete depths.	Mixtures of antifreeze and used oil; light or dense non-aqueous phase liquids and water in a container, such as a tank.
Sludges or slurries	Materials that are a mixture of liquids and solids and that may be viscous or oily. Includes materials with suspended solids.	Waste water treatment sludges from electroplating; slurry created by combining solid waste incinerator ash and water.
Granular solids, unconsolidated	Solids which are not cemented, or do not require significant pressure to separate into particles, and are comprised of relatively small particles or components.	Excavated (*ex situ*) soil in a staging pile; filter press cake; fresh cement kiln dust; incinerator ash.*
Other solids, unconsolidated	Solids with larger particles than those covered by granular solids. The sampling device needs to collect a larger diameter or volume of sample to accommodate the larger particles.	Waste pellets or catalysts.

* For EPA-published guidance on the sampling of incinerator ash, see *Guidance for the Sampling and Analysis of Municipal Waste Combustion Ash for the Toxicity Characteristic* (USEPA 1995f).

Table 10. Descriptions of Media Listed in Table 8 (Continued).

Media	***Description***	***Examples***
Soil (in-situ) and other unconsolidated geologic material	Soil in its original undisturbed location or other geologic material that does not require significant pressure to separate into particles. *In situ* soil sampling may be conducted at subsurface or surface depths. Surface soils generally are defined as soils between the ground surface and 6 to 12 inches below the ground surface (USEPA 1996b); however, the definition of surface soils in State programs may vary considerably from EPA's.	Subsurface soil at a land treatment unit; surface soil contaminated by a chemical spill on top of the ground or soil near a leak from an excavated underground storage tank.*
Solids, consolidated	Cemented or otherwise dense solids that require significant physical pressure to break apart into smaller parts.	Concrete, wood, and architectural debris.
Air	For the purpose of RCRA sampling, air includes emissions from stationary sources or indoor air.	Emissions from boilers and industrial furnaces (BIFs).**
Sediment	Settled, unconsolidated solids beneath a flowing or standing liquid layer.	Sediment in a surface water body.
Soil gas or vapor	Gas or vapor phase in the vadose zone. The vadose zone is the hydrogeological region extending from the soil surface to the top of the principal water table.	Soil gas overlying a waste disposal site.
Ground water	"Water below the land surface in a zone of saturation" (40 CFR 260.10). Water can also be present below the land surface in the unsaturated (vadose) zone.	Ground water in monitoring wells surrounding a hazardous waste landfill.***

* Detailed guidance on soil sampling can be found in *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (Mason 1992), which provides a discussion of the advantages and disadvantages of various sample collection methods for soil.

** See Chapter Ten of SW-846 for EPA-approved methods for sampling air under RCRA.

*** Detailed guidance on ground-water sampling can be found in *RCRA Ground-Water Monitoring -- Draft Technical Guidance* (USEPA 1992c), which updates technical information in Chapter Eleven of SW-846 (Rev. 0, Sept. 1986) and the *Technical Enforcement Guidance Document* (TEGD).

7.2 Conducting Field Sampling Activities

This section provides guidance on performing field sampling activities that typically are performed during implementation of the sampling plan. Additional guidance can be found in *Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual* (USEPA 1994a), *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, U.S. EPA Region 4, May 1996* (USEPA 1996b), other USEPA guidance cited in the reference section of this chapter, and various ASTM standards summarized in Appendix J of this guidance. See also Appendix C of EPA's *Guidance for Quality Assurance Project Plans* (USEPA 1998a). The latter document includes extensive checklists, including the following:

- Sample handling, preparation, and analysis checklist
- QAPP review checklist
- Chain-of-custody checklist.

In this section, we provide guidance on the following topics:

- Sample containers (Section 7.2.1)
- Sample preservation and holding times (Section 7.2.2)
- Documentation of field activities (Section 7.2.3)
- Field quality control samples (Section 7.2.4)
- Sample identification and chain-of-custody procedures (Section 7.2.5)
- Decontamination of equipment and personnel (Section 7.2.6)
- Health and safety (Section 7.2.7)
- Sample packaging and shipping (Section 7.2.8).

7.2.1 Selecting Sample Containers

All samples should be placed in containers of a size and construction appropriate for the volume of material specified in the sampling plan and as appropriate for the requested analyses. If sufficient sample volume is not collected, the analysis of all requested parameters and complete quality control determinations may not be possible. In addition, minimum sample volumes may be required to control sampling errors (see Section 6). Chapters Two, Three, and Four of SW-846 identify the appropriate containers for RCRA-related analyses by SW-846 methods.

> Chapters Two, Three, and Four of SW-846 identify some of the appropriate containers for RCRA-related analyses by SW-846 methods.

It is important to understand that a single "sample" may need to be apportioned to more than one container to satisfy the volume and preservation requirements specified by different categories of analytical methods. Furthermore, the analytical plan may require transport of portions of a sample to more than one laboratory.

Factors to consider when choosing containers are compatibility with the waste components, cost, resistance to breakage, and volume. Containers must not distort, rupture, or leak as a result of chemical reactions with constituents of waste samples. The containers must have adequate wall thickness to withstand handling during sample collection and transport. For analysis of non-volatile constituents, containers with wide mouths are often desirable to facilitate transfer of samples from the equipment. The containers must be large enough to contain the optimum sample volume specified in the DQO Process.

You should store samples containing light-sensitive organic constituents in amber glass bottles with Teflon®-lined lids. Polyethylene containers are not appropriate for use when the samples are to be analyzed for organic constituents because the plastics could contribute organic contaminants and potentially introduce bias. If liquid samples are to be submitted for analysis of volatile compounds, you must store the samples in air-tight containers with zero head space. You can store samples intended for metals and other inorganic constituent analyses in polyethylene containers with polyethylene-lined lids. We recommend that you consult with a chemist for further direction regarding chemical compatibility of available containers and the media to be sampled. We recommend that an extra supply of containers be available at the sampling location in case you want to collect more sample material than originally planned or you need to retain splits of each sample.[2]

Always use clean sample containers of an assured quality. For container cleaning procedures and additional container information, refer to the current iteration of *Specifications and Guidance for Contaminant-Free Sample Containers* (USEPA 1992d). You may wish to purchase pre-cleaned/quality assured bottles in lieu of cleaning your own bottles (USEPA 2001g).

7.2.2 Sample Preservation and Holding Times

Samples are preserved to minimize any chemical or physical changes that might occur between the time of sample collection and analysis. Preservation can be by physical means (e.g., kept at a certain temperature) or chemical means (e.g., with the addition of chemical preservatives). If a sample is not preserved properly, the levels of constituents of concern in the sample may be altered through chemical, biological, or photo-degradation, or by leaching, sorption, or other chemical or physical reactions within the sample container.

The appropriate method for preserving a sample will depend on the physical characteristics of the sample (such as soil, waste, water, etc.), the concentration of constituents in the sample, and the analysis to be performed on the sample. Addition of chemical preservatives may be required for samples to be analyzed for certain parameters. You should not chemically preserve highly concentrated samples. Samples with low concentrations, however, should be preserved. You should consult with a chemist at the laboratory regarding the addition of chemical preservatives and the possible impact on the concentration of constituents in the sample. Also, be aware that addition of some chemical preservatives to highly concentrated waste samples may result in a dangerous reaction.

Regardless of preservation measures, the concentrations of constituents within a sample can degrade over time. Therefore, you also should adhere to sample holding times (time from sample collection to analysis), particularly if the constituents of concern are volatiles in low concentrations. Analytical data generated outside of the specified holding times are considered to be minimum values only. You may use such data to demonstrate that a waste is hazardous

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[2] For example, when inspections are conducted under Section 3007 of RCRA (42 U.S.C. § 6927), and samples are obtained, EPA must provide a split sample to the facility, upon request.

where the value of a constituent-of-concern is above the regulatory threshold, but you cannot use the data to demonstrate that a waste is not hazardous. Exceeding a holding time when the results are above a decision level does not invalidate the data.

Appropriate sample preservation techniques and sample holding times for aqueous matrices are listed in Chapters Two, Three, and Four of SW-846. You should also consult the methods to be used during analysis of the sampled waste. In addition, *Standard Guide for Sampling Waste and Soil for Volatile Organic Compounds* (ASTM D 4547-98) provides information regarding the preservation of volatile organic levels in waste and soil samples.

7.2.3 Documentation of Field Activities

This section provides guidance on documenting field activities. Records of field activities should be legible, identifiable, retrievable and protected against damage, deterioration, and loss. You should record all documentation in waterproof, non-erasable ink. If you make an error in any of these documents, make corrections by crossing a single line through the error and entering the correct information adjacent to it. The corrections should then be initialed and dated. Stick-on labels of information should not be removable without evidence of the tampering. Do not put labels over previously recorded information.

Keep a dedicated logbook for each sampling project with the name of the project leader, team members, and project name written inside the front cover. Document all aspects of sample collection and handling in the logbook. Entries should be legible, accurate, and complete. The language should be factual and objective.

You also should include information regarding sample collection equipment (use and decontamination), field analytical equipment and the measurements, calculations and calibration data, the name of the person who collected the sample, sample numbers, sample location description and diagram or map, sample description, time of collection, climatic conditions, and observations of any unusual events. Document the collection of QC samples and any deviations from procedural documents, such as the QAPP and SOPs.

When videos, slides, or photographs are taken, you should number them to correspond to logbook entries. The name of the photographer, date, time, site location, and site description should be entered sequentially into the logbook as photos are taken. A series entry may be used for rapid aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. Special lenses, films, filters, or other image enhancement techniques must be noted in the logbook. Chain-of-custody procedures for photoimages depend on the subject matter, type of film, and the processing it requires. Adequate logbook notations and receipts may be used to account for routine film processing. Once developed, the slides or photographic prints should be serially numbered corresponding to the logbook descriptions and labeled (USEPA 1992e).

7.2.4 Field Quality Control Samples

Quality control samples are collected during field studies to monitor the performance of sample collection and the risk of sampling bias or errors. Field QC samples could include the following:

Equipment blank: A rinse sample of the decontaminated sampling equipment using organic/analyte free water under field conditions to evaluate the effectiveness of equipment decontamination or to detect sample cross contamination.

Trip blank: A sample prepared prior to the sampling event and stored with the samples throughout the event. It is packaged for shipment with the samples and not opened until the shipment reaches the laboratory. The sample is used to identify any contamination that may be attributed to sample handling and shipment.

Field blank: A sample prepared in the field using organic/analyte free water to evaluate the potential for contamination by site contaminants not associated with the sample collected (e.g., airborne organic vapors)

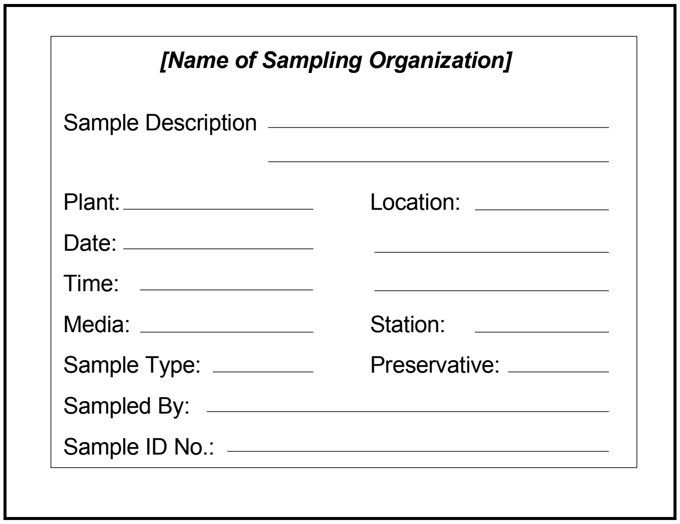
Field split sample: Two or more representative portions taken from the same sample and submitted for analysis to different laboratories. Field split samples are used to estimate interlaboratory precision.

In addition to collecting field QC samples, other QC procedures include sample storage, handling, and documentation protocols. These procedures are covered separately in the following sections. In addition, Chapter One of SW-846, entitled "Quality Control", contains guidance regarding both field and laboratory QA/QC. We also recommend reviewing the following for information on field QA/QC:

- *EPA Guidance for Quality Assurance Project Plans* (USEPA 1998a)
- *Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Quality Assurance and Quality Control Planning and Implementation* (ASTM D 5283-92).

7.2.5 Sample Identification and Chain-of-Custody Procedures

You should identify samples for laboratory analysis with sample tags or labels. An example of a sample label is given in Figure 29. Typically, information on the sample label should include the sample identification code or number, date, time of collection, preservative used, media, location, initials of the sampler, and analysis requested. While not required, you may elect to seal each sample container with a custody seal (Figure 30).



[Name of Sampling Organization]

Sample Description ____________________

Plant: __________ Location: __________

Date: __________

Time: __________

Media: __________ Station: __________

Sample Type: __________ Preservative: __________

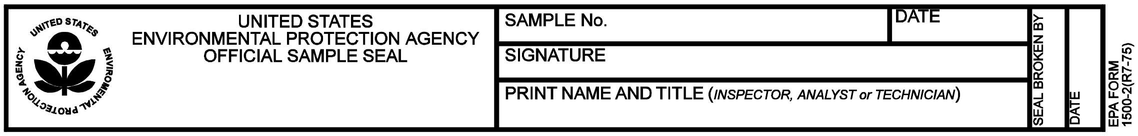
Sampled By: ____________________

Sample ID No.: ____________________

Figure 29. Sample label

You should use chain-of-custody procedures to record the custody of the samples. Chain-of-custody is the custody of samples from time of collection through shipment to analysis. A sample is in one's custody if:

- It is in the actual possession of an investigator
- It is in the view of an investigator, after being in their physical possession
- It is in the physical possession of an investigator, who secures it to prevent tampering
- It is placed in a designated secure area.



UNITED STATES
ENVIRONMENTAL PROTECTION AGENCY
OFFICIAL SAMPLE SEAL

SAMPLE No.	DATE
SIGNATURE	
PRINT NAME AND TITLE (*INSPECTOR, ANALYST or TECHNICIAN*)	

SEAL BROKEN BY

DATE

EPA FORM
1500-2(R7-75)

Figure 30. Custody seal

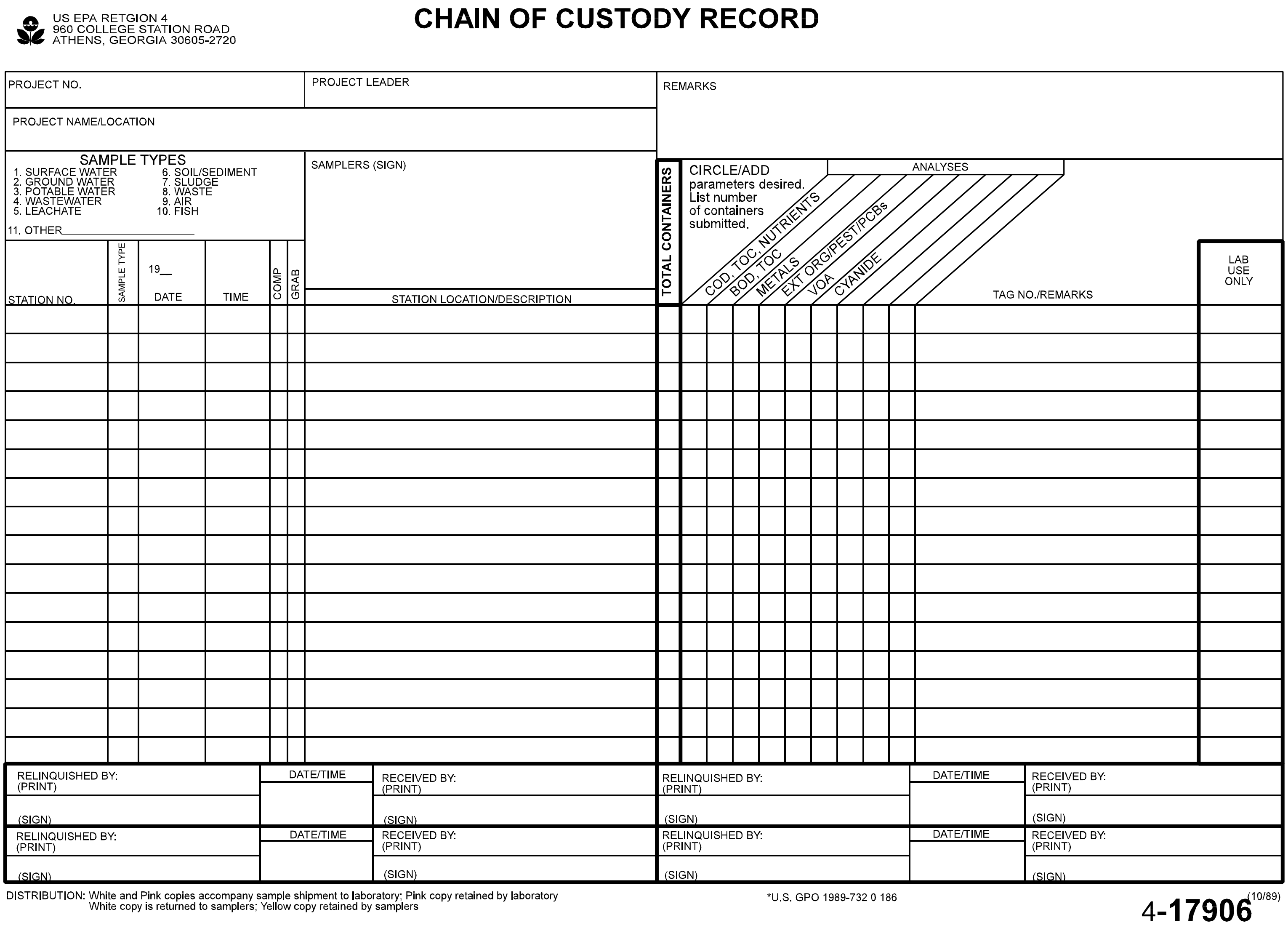
All sample sets should be accompanied by a chain-of-custody form. This record also serves as the sample logging mechanism for the laboratory sample custodian. Figure 31 illustrates the content of a chain-of-custody form. When the possession of samples is transferred, both the individual relinquishing the samples and the individual receiving the samples should sign, date, and note the time on the chain-of-custody document. If you use overnight shipping service to transport the samples, record the air bill number on the chain-of-custody form. This chain-of-custody record represents the official documentation for all transfers of the sample custody until the samples have arrived at the laboratory. The original form of the chain-of-custody record should accompany each shipment. A copy should be retained by a representative of the sampling team.

When sample custody is transferred between individuals, the samples or coolers containing the samples are sealed with a custody seal. This seal cannot be removed or broken without destruction of the seal, providing an indicator that custody has been terminated.

EPA's Superfund Program has developed software called *Field Operations and Records Management System (FORMS) II Lite™* that automates the printing of sample documentation in the field, reduces time spent completing sample collection and transfer documentation, and facilitates electronic capture of data prior to and during field sampling activities. For information on *FORMS II Lite™*, see http://www.epa.gov/superfund/programs/clp/f2lite.htm.

For additional information on chain-of-custody procedures, we recommend ASTM D 4840, *Standard Guide for Sampling Chain-of-Custody Procedures*.

Figure 31. Chain-of-custody form



US EPA RETGION 4
960 COLLEGE STATION ROAD
ATHENS, GEORGIA 30605-2720

CHAIN OF CUSTODY RECORD

PROJECT NO.

PROJECT LEADER

REMARKS

PROJECT NAME/LOCATION

SAMPLE TYPES

1. SURFACE WATER
2. GROUND WATER
3. POTABLE WATER
4. WASTEWATER
5. LEACHATE
6. SOIL/SEDIMENT
7. SLUDGE
8. WASTE
9. AIR
10. FISH
11. OTHER______________________

SAMPLERS (SIGN)

CIRCLE/ADD parameters desired. List number of containers submitted.

ANALYSES

STATION NO.	SAMPLE TYPE	19__ DATE	TIME	COMP	GRAB	STATION LOCATION/DESCRIPTION	TOTAL CONTAINERS	COD, TOC, NUTRIENTS	BOD, TOC	METALS	EXT ORG/PEST/PCBs	VOA	CYANIDE				TAG NO./REMARKS	LAB USE ONLY

RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)
(SIGN)		(SIGN)	(SIGN)		(SIGN)
RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)
(SIGN)		(SIGN)	(SIGN)		(SIGN)

DISTRIBUTION: White and Pink copies accompany sample shipment to laboratory; Pink copy retained by laboratory
White copy is returned to samplers; Yellow copy retained by samplers

*U.S. GPO 1989-732 0 186

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7.2.6 Decontamination of Equipment and Personnel

Decontamination of sampling equipment refers to the physical and chemical steps taken to remove any chemical or material contamination. Equipment decontamination helps prevent sampling bias. All equipment that comes in contact with the sampled material should be free of components that could influence (contaminate) the true physical or chemical composition of the material. Besides the equipment used to collect the samples, any containers or equipment used for sample compositing or for field subsampling should be free of contamination.

Equipment decontamination also prevents cross-contamination of samples when the equipment is used to collect more than one sample. Disposable equipment or the use of dedicated equipment provides the most effective means of avoiding cross-contamination; however, the use of such equipment is not always practical.

You should decontaminate equipment to a level that meets the minimum requirements for your data collection effort. Your decontamination steps (e.g., use of solvents versus use of only soap and water), therefore, should be selected based on the constituents present, their concentration levels in the waste or materials sampled, and their potential to introduce bias in the sample analysis results if not removed from the sampling equipment. You should describe the project-specific decontamination procedures in your planning document for the sampling effort. In addition, items used to clean the equipment, such as bottle brushes, should be free of contamination.

The following procedure is an example of one you could use to decontaminate a sampling device to be used for collecting samples for trace organic or inorganic constituent analyses (from USEPA 1996b):

1. Clean the device with tap water and soap, using a brush if necessary to remove particulate matter and surface films.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with analyte- or organic-free water.

4. Rinse thoroughly with solvent. Do not solvent-rinse PVC or plastic items.

5. Rinse thoroughly with organic/analyte free water, or allow equipment to dry completely.

6. Remove the equipment from the decontamination area. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.

The specifications for the cleaning materials are as follows (you should justify and document the use of substitutes):

- "Soap" should be a phosphate-free laboratory detergent such as Liquinox®. It must be kept in clean plastic, metal, or glass containers until used and poured directly from the container when in use.

- "Solvent" should be pesticide-grade isopropanol. It must be stored in the unopened original containers until used. It may be applied using the low pressure nitrogen system fitted with a Teflon® nozzle, or using Teflon® squeeze bottles. For equipment highly contaminated with organics (such as oily waste), a laboratory-grade hexane may be a more suitable alternative to isopropanol.

- "Tap water" may be used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute. Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose or tap.

- "Analyte free water" (deionized water) is tap water treated by passing it through a standard deionizing resin column. At a minimum, it must contain no detectable heavy metals or other inorganic compounds as defined by a standard ICP (or equivalent) scan. It may be obtained by other methods as long as it meets the analytical criteria. Analyte free water must be stored in clean glass, stainless steel, or plastic containers that can be closed prior to use. It can be applied from plastic squeeze bottles.

- "Organic/analyte free water" is tap water that has been treated with activated carbon and deionizing units. A portable system to produce such water under field conditions is available. At a minimum, the water must meet the criteria of analyte free water and not contain detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined for a given set of analyses. Organic/analyte free water obtained by other methods is acceptable, as long as it meets the analytical criteria. It must be stored in clean glass, Teflon®, or stainless steel containers. It may be applied using Teflon® squeeze bottles or with the portable system.

Clean the field equipment prior to field use. Designate a decontamination zone at the site and, if necessary, construct a decontamination pad at a location free of surface contamination. You should collect wastewater from decontamination (e.g., via a sump or pit) and remove it frequently for appropriate treatment or disposal. The pad or area should not leak contaminated water into the surrounding environment. You also should collect solvent rinses for proper disposal.

You should always handle field-cleaned equipment in a manner that prevents recontamination. For example, after decontamination but prior to use, store the equipment in a location away from the cleaning area and in an area free of contaminants. If it is not immediately reused, you should cover it with plastic or aluminum foil to prevent recontamination.

Decontamination will generate a quantity of wastes called investigation derived waste (IDW). You should address the handling and disposal of IDW in your sampling plan. You must handle this material in accordance with whether it is nonhazardous or suspected of, or known to be, hazardous. You should minimize the generation of hazardous IDW and keep it separated from nonhazardous IDW. For example, you should control the volume of spent solvents during equipment decontamination by applying the minimum amount of liquid necessary and capturing it separately from the nonhazardous washwater. For additional guidance on handling IDW, see *Management of Investigation-Derived Wastes* (USEPA 1992f).

Decontamination of personnel and their protective gear also is often necessary during hazardous waste sampling. This important type of decontamination protects personnel from chemical exposure and prevents cross-contamination when personnel change locations. The level or degree of such decontamination will depend on site-specific considerations, such as the health hazards posed by exposure to the sampled waste. You should address these decontamination procedures in your health and safety plan.

For additional information regarding decontamination, see ASTM D 5088, *Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites*. Another source of additional information is "Sampling Equipment Decontamination" (USEPA 1994f), issued by EPA's Environmental Response Team.

7.2.7 Health and Safety Considerations

Regulations published by the Occupational Safety and Health Administration (OSHA) at 29 CFR Part 1910.120 govern workers at hazardous waste sites and include requirements for training, equipment, medical monitoring, and other practices. Many sampling activities covered by this guidance may require compliance with OSHA's health and safety regulations. Specific guidance on worker health and safety is beyond the scope of this chapter; however, development and use of a project-specific health and safety plan may be required. It is the responsibility of the sampling team leader and others in charge to ensure worker safety.

Some important health and safety considerations follow:

- Field personnel should be up-to-date in their health and safety training.

- Field personnel should have a medical examination at the initiation of sampling activities and routinely thereafter, as appropriate and as required by the OSHA regulations. Unscheduled examinations should be performed in the event of an accident or suspected exposure to hazardous materials.

- Staff also should be aware of the common routes of exposure at a site and be instructed in the proper use of safety equipment and protective clothing and equipment. Safe areas should be designated for washing, drinking, and eating.

- To minimize the impact of an emergency situation, field personnel should be aware of basic first aid and have immediate access to a first aid kit.

The guidance manual *Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities* (OSHA 1985, revised 1998) was jointly developed by the National Institute for Occupational Safety and Health (NIOSH), OSHA, the United States Coast Guard (USCG), and EPA. Its intended audience is those who are responsible for occupational safety and health programs at hazardous waste sites.

7.2.8 Sample Packaging and Shipping

During transport of waste samples, you should follow all State and Federal regulations governing environmental sample packaging and shipment and ship according to U.S. Department of Transportation (DOT) and International Air Transportation Association (IATA) regulations. Minimum guidelines for sample packaging and shipping procedures follow in the next subsections; however, the rules and regulations for sample packaging and shipping are complex, and for some samples and shipping situations the procedures outlined below may need to be exceeded.

7.2.8.1 Sample Packaging

You should package and label samples in an area free of contamination. You also should ship or transport samples to a laboratory within a time frame that meets recommended sample holding times for the respective analyses. Additional guidelines follow:

- Aqueous samples for inorganic analysis and volatile organic analysis may require chemical preservation. The specific preservation requirements will depend on the analytical method to be used.

- Make sure all lids/caps are tight and will not leak.

- Make sure sample labels are intact and covered with a piece of clear tape for protection.

- Enclose the sample container in a clear plastic bag and seal the bag. Make sure the sample labels are visible. If bubble wrap or other wrapping material will be placed around the labeled containers, write the sample number and fraction (e.g., "BLH01-VOCs") so that it is visible on the outside of the wrap, then place the wrapped container in a clear plastic bag and seal the bag.

- Make sure that all samples that need to be kept cold ($4 \pm 2\ ^{\circ}C$) have been thoroughly cooled before placing in packing material so that the packing material serves to insulate the cold. Change the ice prior to shipment as needed. Ideally, pack the cooled samples into shipping containers that have already been chilled. (Of course, these precautions are not necessary if none of the samples in the shipping container need to be kept cold.)

- Any soil/sediment samples suspected to be of medium/high concentration or containing dioxin must be enclosed in a metal can with a clipped or sealable lid (e.g., paint cans) to achieve double containment of those samples. Place suitable absorbent packing material around the sample container in the can. Make sure the sample is securely stored in a can and the lid is sealed. Label the outer metal container with the sample number and fraction of the sample inside.

- Use *clean* waterproof metal or hard plastic ice chests or coolers that are in good repair for shipping samples.

- Remove the inapplicable previous shipping labels. Make sure any drain plugs are shut. Seal plugs shut on the inside and outside with a suitable tape such as duct tape. Line the cooler with plastic (e.g., large heavy-duty garbage bag) before inserting samples.

- Ship samples at 4 ± 2 °C, place double-bagged ice on top of samples. Ice must be sealed in double plastic bags to prevent melting ice from soaking the packing material. Loose ice should not be poured into the cooler.

- Conduct an inventory of sample numbers, fractions, and containers when placing samples into the coolers. Check the inventory against the corresponding chain-of-custody form before sealing the cooler to make sure that all samples and containers are present.

- Pack the lined shipping containers with noncombustible absorbent packing material, such as vermiculite or rock wool. Place the packing material on the bottom of the shipping container (inside the plastic liner) and around sample bottles or metal cans to avoid breakage during shipment. Never use earth, ice, paper, or styrofoam to pack samples. Earth is a contaminant, melted ice may cause complications and allow the sample containers to bang together when the shipping container is moved, and styrofoam presents a disposal problem (it also may easily blow out of the shipping container at the site).

- For samples that need to be shipped at 4 ± 2ºC, place double-bagged ice on top of samples and fill remaining space with packing material. If sample bottles have been protected with packaging material such as bubble wrap, then some double-bagged ice or ice packs also may be placed between samples.

- Use tape to securely fasten the top of the plastic used to line the shipping container. It is a good idea to then place a completed custody seal around the top of the bag that contains the sample in case the outer seals placed across the cooler lid are inadvertently damaged during shipment.

- Enclose all sample documentation (i.e., chain-of-custody forms and cooler return shipping documents) in a waterproof plastic bag, and tape the bag to the underside of the cooler lid. This documentation should address all samples in the cooler, but not address samples in any other cooler.

- If more than one cooler is being used, place separate sample documentation in each cooler. Instructions for returning the cooler should be documented inside the cooler lid. Write a return name and address for the sample cooler on the inside of the cooler lid in permanent ink to ensure return of the cooler.

- Tape the cooler shut using strapping tape over the hinges. Place completed custody seals across the top and sides of the cooler lid so that lid cannot be opened without breaking the seal.

- Place clear tape over the seal to prevent inadvertent damage to the seal during shipment. Do not place clear tape over the seals in a manner that would allow the seals to be lifted off with the tape and then reaffixed without breaking the seal.

For additional detailed guidance on sample documentation, packaging, and shipping, we recommend the *Contract Laboratory Program (CLP) Guidance for Field Samplers - Draft Final* (USEPA 2001g).

7.2.8.2 Sample Shipping

In general, samples of drinking water, most ground waters and ambient surface waters, soil, sediment, treated waste waters, and other low concentration samples can be shipped as environmental samples; however, shipment of high concentration waste samples may require shipment as dangerous goods (not as “hazardous waste”). Note that RCRA regulations specifically exempt samples of hazardous waste from RCRA waste identification, manifest, permitting, and notification requirements (see 40 CFR §261.4(d)). The shipment of samples to and from a laboratory, however, must comply with U.S. DOT, U.S. Postal Service, or any other applicable shipping requirements. If a sample is a hazardous waste, once received at the laboratory, it must be managed as a hazardous waste.

For information on shipping dangerous goods visit the International Air Transport Association (IATA) Dangerous Goods Information Online at http://www.iata.org/cargo/dg/index.htm or call 1-800-716-6326.

In recent years, commercial overnight shipping services have adopted the regulations of the IATA for shipment of dangerous goods by air. The IATA Dangerous Goods Regulations contain all provisions mandated by the International Civil Aviation Organization and all rules universally agreed to by airlines to correctly package and safely transport dangerous goods by air. Contact IATA for a copy of the IATA Dangerous Goods Regulations and for assistance in locating suppliers of specialized packaging for dangerous goods.

When shipping samples, perform the following activities:

- Clearly label the cooler and fill out appropriate shipping papers.
- Place return address labels clearly on the outside of the cooler.
- If more than one cooler is being shipped, mark each cooler as "1 of 2," "2 of 2," etc.
- Ship samples through a commercial carrier. Use appropriate packaging, mark and label packages, and fill out all required government and commercial carrier shipping papers according to DOT and IATA commercial carrier regulations.
- Ship all samples by overnight delivery in accordance with DOT and IATA regulations.

7.3 Using Sample Homogenization, Splitting, and Subsampling Techniques

7.3.1 Homogenization Techniques

The objective of homogenization (mixing) is to minimize grouping and segregation of particles so they are randomly distributed within the sample. While homogenization can reduce grouping and segregation of particles, it will not eliminate it and will not make the material "homogeneous." If homogenization is successful, subsamples of the homogenized material will show less variability than if the material was not homogenized. Homogenization, combined with a composite sampling strategy, can be an efficient method for improving the accuracy and precision in sampling of particulate material (Jenkins, et al. 1996). Homogenization can be applied to solids, liquids, slurries, and sludges.

Pitard (1993) recognizes two processes for homogenization:

> **Stationary processes** - in which the material is not mixed but is redistributed so that any correlation between the characteristics of individual fragments or particles is lost or minimized. An example of this process is the collection of many small increments to form an individual sample (ideally we would pick many individual particles at random to form the sample, but this is not possible).
>
> **Dynamic processes** - in which the material is mechanically mixed to remove or minimize correlation between the characteristics of the fragment or particle and its position within the sample. Examples of this process include mechanical mixing within a container and use of magnetic stirrers in a beaker.

Note that the benefits of homogenization may be temporary because gravity-induced segregation can occur during shipment, storage, and handling of samples. For this reason, consider carrying out homogenization (mixing) immediately prior to analysis.

Some homogenization techniques work better than others. The strengths and limitations of homogenization equipment and procedures (cone and quartering, riffle splitters, rotary splitters, multiple cone splitters, and V-blenders) have been reviewed in the literature by Pitard (1993), Schumacher, et al. (1991), ASTM (Standard D 6051-96), and others. The preferred techniques for use within the laboratory follow:

- Riffling (see also Section 7.3.2)
- Fractional shoveling (see also Section 7.3.2)
- Mechanical mixing
- Cone and quartering
- Magnetic stirrers (e.g., to homogenize the contents of an open beaker)
- V-blenders.

Fractional shoveling and mechanical mixing also can be used in the field. Note that some techniques for homogenization, such as riffling and fractional shoveling, also are used for splitting and subsampling. Note that Pitard (1993) discourages the use of "sheet mixing" (also called "mixing square") and vibratory spatulas because they tend to segregate particles of different density and size.

7.3.2 Sample Splitting

Splitting is employed when a field sample is significantly larger than the required analytical sample. The goal of splitting is to reduce the mass of the retained sample and obtain an aliquot of the field sample that reflects the average properties of the entire field sample. It is often necessary to repeat the splitting process a number of times to achieve a sufficient reduction in mass for analytical purposes.

Splitting can be used to generate a reduced mass aliquot that can be analyzed in its entirety or a much reduced and homogenized mass from which an analytical or subsample can be collected. ASTM's *Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities* (ASTM D 6323-98), lists and discusses a variety of splitting equipment (such as sectorial splitters and riffle splitters) and splitting procedures (such as cone and quartering and the alternate scoop method). Gerlach, et al. (2002) also evaluated sample splitting methods (riffle splitting, paper cone riffle splitting, fractional shoveling, coning and quartering, and grab sampling) and found that riffle splitting methods performed the best.

A simple alternative to riffle splitting a sample of solid media is a technique called "fractional shoveling." To perform fractional shoveling, deal out small increments from the larger sample in sequence into separate piles, randomly select one of the piles and retain it as the subsample (or retain more than one if a portion of the sample is to be "split" with another party and/or retained for archive purposes), and reject the others (see Figure 32).

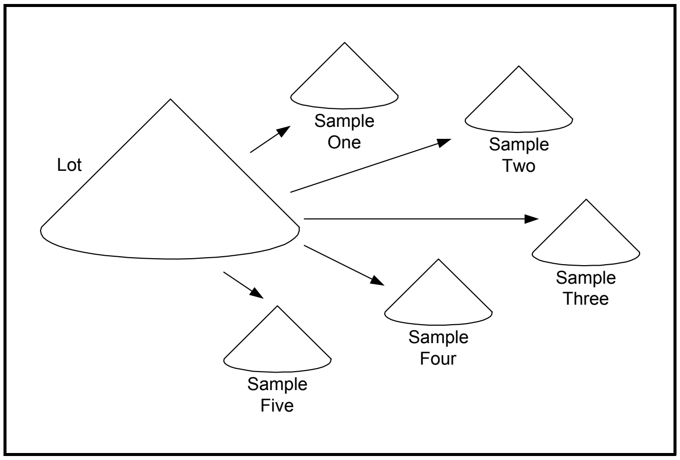

Figure 32. Fractional shoveling as a sample splitting method (after Pitard 1993)

7.3.3 Subsampling

The size of the sample submitted to the laboratory (either an individual sample or a composite) by field personnel typically far exceeds that required for analysis. Consequently, subsampling is needed. A subsample is defined as "a portion of material taken from a larger quantity for the purpose of estimating properties or the composition of the whole sample" (ASTM D 4547-98). Taking a subsample may be as simple as collecting the required mass from a larger mass, or it may involve one or more preparatory steps such as grinding, homogenization, and/or splitting of the larger mass prior to removal of the subsample.

Specific procedures for maintaining sample integrity (e.g., minimizing fundamental error) during splitting and subsampling operations typically are not addressed in quality assurance, sampling, or analytical plans, and error may be introduced unknowingly in subsampling and sample preparation. Many environmental laboratories do not have adequate SOPs for subsampling; therefore, it is important for the data users to provide the laboratory personnel clear instruction if any special subsampling or sample handling procedures are needed (such as instructions on mixing of the sample prior to analysis, removing particles greater than a certain size, analyzing phases separately, etc.). If proper subsampling procedures are not specified in planning documents, SOPs, or documents shipped with the samples, it may be difficult to assess the usability of the results.

The following sections provide general guidance on obtaining subsamples of liquids, mixtures of liquids and solids, and soils and solid media. For additional guidance and detailed procedures, see *Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities* (ASTM D 6051-96) and *Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities* (ASTM D 6323-98).

7.3.3.1 Subsampling Liquids

In the case of subsampling a liquid, special precautions may be warranted if the liquid contains suspended solids and/or the liquid comprises multiple liquid phases. In practice, samples may contain solids and/or separate phases that are subject to gravitational action (Gy 1998). Even a liquid that appears clear (absent of solids and without iridescence) may not be "homogeneous."

Subsampling of liquids (containing solids and/or in multiple phases) can be addressed by using one or the other of two possible approaches:

- Mixing the sample such that all phases are homogenized, and then taking a subsample (using a pipette, for example)

- Allowing all of the phases to separate followed by subsampling and analysis of each phase separately.

Of course, the characteristics of the waste and the type of test must be considered. For example, mixing of multi-phasic wastes to be analyzed for volatiles should be avoided due to the potential loss of constituents. Some multi-phasic liquid wastes can form an emulsion when mixed. Others, in spite of mixing, will quickly separate back into distinct phases.

7.3.3.2 Subsampling Mixtures of Liquids and Solids

If the sample is a mixture of liquids and solids, subsampling usually requires that the phases be separated. The separate phases are then separately subsampled. Subsampling of the liquid phase can be accomplished as described above, while subsampling of the solid phase should be done according to sampling theory, as summarized below.

7.3.3.3 Subsampling Soils and Solid Media

To correctly subsample soil or solid media, use sampling tools and techniques that minimize delimitation and extraction error. If the particles in the sample are too coarse to maintain fundamental error within desired limits, it may be necessary to perform a series of steps of particle size reduction followed by subsampling (see Appendix D). If the field sample mass is equal to or less than the specified analytical size, the field sample can be analyzed in its entirety. If the mass of the field sample is greater than the specified analytical sample size, subsampling will be required.

One possible alternative to particle-size reduction prior to subsampling is to simply remove the coarse particles (e.g., via a sieve or visually) from the sample. This selective removal technique is *not* recommended in situations in which the larger particles contribute to the overall concentration of the constituent of concern in the waste. In other words, do not remove the large particles if the constituents of concern tend to be concentrated in the large particles relative to the smaller particles.

If the largest particle size of the field sample exceeds the allowable size for maintaining the fundamental error specified by the DQO *and* the analyte of interest is volatile, it may be necessary to analyze the sample as is and accept a large fundamental error. Guidance on handling VOCs in samples can be found in Section 6.3.4 and in ASTM Standard D 4547-98.

The *Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities* (ASTM D 6323-98) lists a variety of equipment for performing particle-size reduction (e.g., cutting mills, jar mills, disc mills, dish and puck mills, mortar grinders and jaw crushers) and tabulates their uses and limitations.

The techniques discussed below are most relevant to subsampling of solid particulate matter for analysis of nonvolatile constituents. Mason (1992, page 5-7) provides a field procedure that can be used to reduce the volume of a field soil sample for submission to the laboratory.

The issues regarding the subsampling of particulate-containing materials are identical to those considered when collecting the original field samples and are as follows:

- The tool used to collect the analytical sample must be correct and not discriminate against any portion of the sample (in other words, the tool should not introduce increment delimitation and increment extraction errors).
- The mass of the subsample must be enough to accommodate the largest of the particles contained within the parent sample (to reduce fundamental error).
- The sample mass and the manner in which it is collected must accommodate the short-term heterogeneity within the field sample (to reduce grouping and segregation error).

The sampling tool must be constructed such that its smallest dimension is at least three times greater than the largest particle size contained within the material being subsampled. The construction of the sampling tool must be such that it does not discriminate against certain areas of the material being sampled. For example, Pitard (1993) argues that all scoops for subsampling should be rectangular or square in design with flat bottoms as opposed to having curved surfaces (Figure 33).

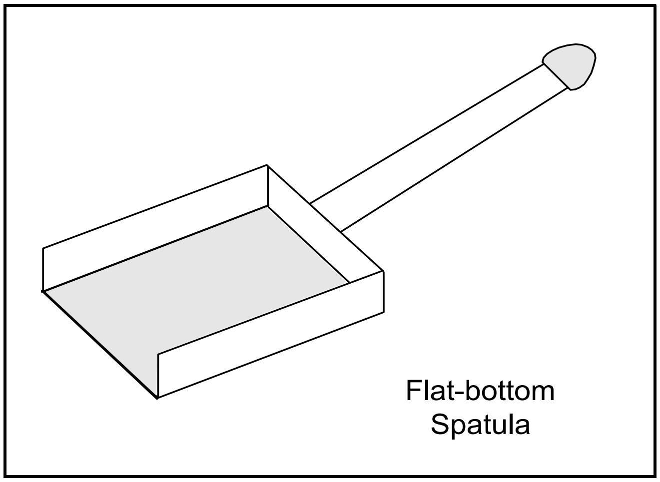

Figure 33. Example of correctly designed device for subsampling. Flat bottom and vertical side walls minimize increment delimitation error.

Pitard (1993) and ASTM D 6323-98 suggest subsampling from relatively flat elongated piles using a transversal subsampling technique that employs a sampling scoop or spatula and a flat working surface (Figure 34(a)). The objective is to convert the sampling problem to a one-dimensional approach. Specifically, Pitard (1993) recommends the following procedure:

- Empty the sample from the sample container onto a smooth and clean surface or appropriate material.
- Do not try to homogenize the sample, as this may promote segregation of particles.
- Reduce the sample by using the fractional shoveling technique (Figure 32) until a sample 5 to 10 times larger than the analytical sample is obtained.
- Shape the remaining material into an elongated pile with uniform width and thickness (Figure 34(a)).
- Take increments all across the pile through the entire thickness.
- Reshape the pile perpendicular to its long axis, and continue to take increments across the pile until the appropriate sample weight is reached.

Fractional shoveling and alternate scoop techniques alone (Figure 32) also can be used to generate subsamples.

When using these techniques, several stages or iterations of subsampling followed by particle size reduction may be needed to minimize fundamental error (also see Appendix D). At each stage, the number of increments should be at least 10 and preferably 25 to control grouping and segregation (short-term heterogeneity) within the sample. In the final stage, however, where very small analytical samples are required, the number of increments required will be much less.

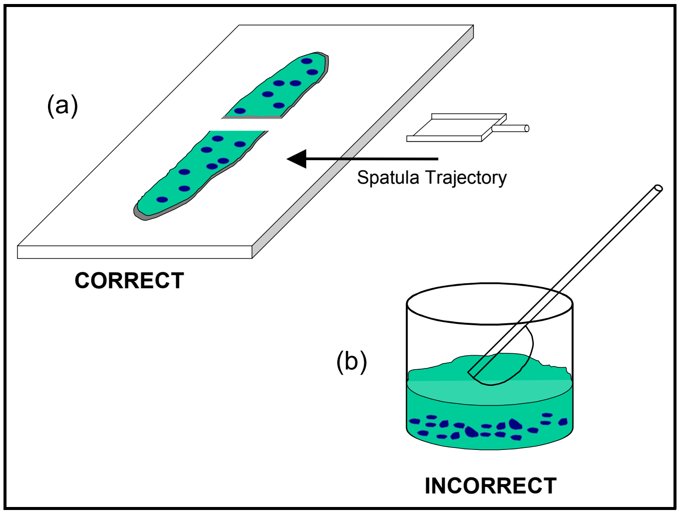

Figure 34. Correct (a) and incorrect (b) laboratory techniques for obtaining subsamples of granular solid media ((a) modified after Pitard 1993).

The subsampling procedures described above offer a more correct and defensible alternative to an approach to subsampling in which the analyst simply opens the sample jar or vial and removes a small increment from the top for preparation and analysis (Figure 34(b)).

8 ASSESSMENT: ANALYZING AND INTERPRETING DATA

This section presents guidance for the assessment of sampling and analytical results. In performing data assessment, evaluate the data set to determine whether the data are sufficient to make the decisions identified in the DQO Process. The data assessment process includes (1) sampling assessment and analytical assessment, and (2) data quality assessment (DQA) (Figure 35) and follows a series of logical steps to determine if the data were collected as planned and to reach conclusions about a waste relative to RCRA requirements.

At the end of the process, EPA recommends reconciliation with the DQOs to ensure that they were achieved and to decide whether additional data collection activities are needed.

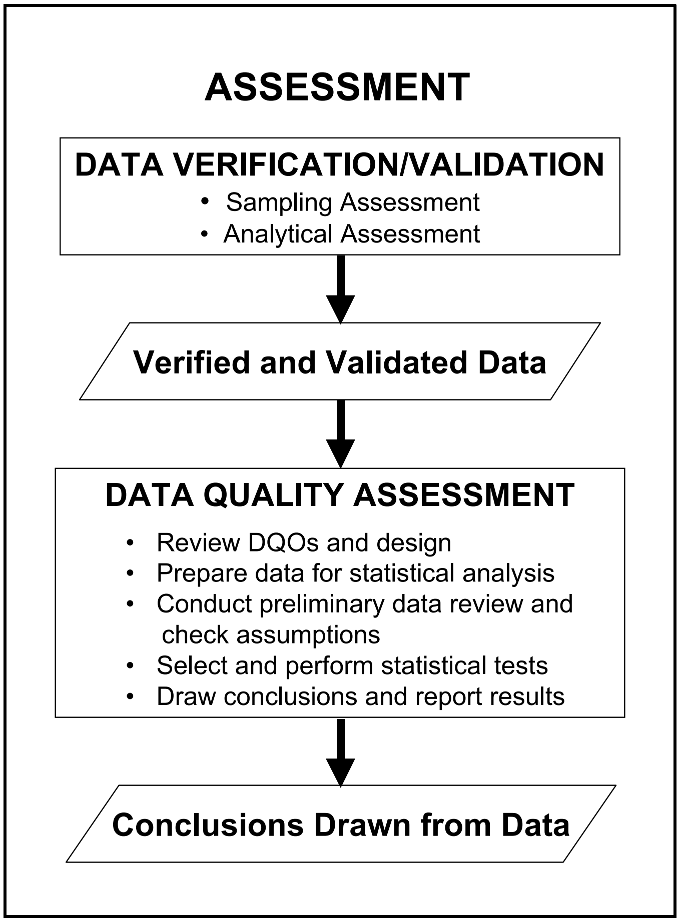

Figure 35. Elements of the quality assurance assessment process (modified after USEPA 1998a)

8.1 Data Verification and Validation

Data verification and validation are performed to ensure that the sampling and analysis protocols specified in the QAPP or WAP were followed and that the measurement systems performed in accordance with the criteria specified in the QAPP or WAP. The process is divided into two parts:

- sampling assessment (Section 8.1.1), and
- analytical assessment (Section 8.1.2).

Guidance on analytical assessment is provided in Chapter One of SW-846 and in the individual analytical methods. Additional guidance can be found in *Guidance on Environmental Data Verification and Data Validation EPA QA/G-8,* published by EPA's Office of Environmental Information (USEPA 2001c). For projects generating data for input into risk assessments, see EPA's *Guidance for Data Usability in Risk Assessment, Final* (USEPA 1992g).

8.1.1 Sampling Assessment

Sampling assessment is the process of reviewing field sampling and sample handling methods to check conformance with the requirements specified in the QAPP. Sampling assessment activities include a review of the sampling design, sampling methods, documentation, sampling handling and custody procedures, and preparation and use of quality control samples.

The following types of information are useful in assessing the sampling activity:

- Copies of the sampling plan, QAPP, and SOPs.

- Copies of logbooks, chain-of-custody records, bench sheets, well logs, sampling sequence logs, field instrument calibration records and performance records, and/or other records (including electronic records such as calculations) that describe and/or record all sampling operations, observations, and results associated with samples (including all QC samples) while in the custody of the sampling team. Records/results from the original sampling and any resampling, regardless of reason, should be retained. Also, retain copies of the shipping manifest and excess sample disposition (disposal) records describing the ultimate fate of any sample material remaining after submission to the laboratory.

- Copies of all records/comments associated with the sample team review of the original data, senior staff review, and QA/QC review of the sampling activity. Copies of any communication (telephone logs, faxes, E-mail, other records) between the sampling team and the customer dealing with the samples and any required resampling or reporting should be provided.

The following subsections outline the types of sampling information that should be assessed.

8.1.1.1 Sampling Design

Review the documentation of field activities to check if the number and type of samples called for in the sampling plan were, in fact, obtained and collected from the correct locations. Perform activities such as those described below:

- Sampling Design: Document any deviations from the sampling plan made during the field sampling effort and state what impact those modifications might have on the sampling results.

- Sample Locations/Times: Confirm that the locations of the samples in time or space match those specified in the plan.

- Number of Samples: Check for completeness in the sampling in terms of the number of samples obtained compared to the number targeted. Note the cause of the deficiencies such as structures covering planned locations, limited access due to unanticipated events, samples lost in shipment or in the laboratory, etc.

- Discrete versus Composite Samples: If composite sampling was employed, confirm that each component sample was of equal mass or volume. If not, determine if sufficient information is presented to allow adjustments to any calculations made on the data. Both field and laboratory records should be reviewed because compositing can occur at either location.

8.1.1.2 Sampling Methods

Details of how a sample was obtained from its original time/space location are important for properly interpreting the measurement results. Review the selection of sampling and ancillary equipment and procedures (including equipment decontamination) for compliance with the QAPP and sampling theory. Acceptable departures (for example, alternate equipment) from the QAPP and the action to be taken if the requirements cannot be satisfied should be specified for each critical aspect. Note potentially unacceptable departures from the QAPP and assess their potential impact on the quality and usefulness of the data. Comments from field surveillance on deviations from written sampling plans also should be noted.

Sampling records should be reviewed to determine if the sample collection and field processing were appropriate for the analytes being measured. For example, sampling for volatiles analysis poses special problems due to the likely loss of volatiles during sample collection. Also, determination of the appropriate "sample support" should be reviewed, whether it was obtained correctly in the field, whether any large particles or fragments were excluded from the sample, and whether any potential biases were introduced.

Laboratory subsampling and sample preparation protocols should be examined for the same types of potential bias as the field procedures. When found, they should be discussed in the assessment report.

8.1.1.3 Sample Handling and Custody Procedures

Details of how a sample is physically treated and handled between its original site or location and the actual measurement site are extremely important. Sample handling activities should be reviewed to confirm compliance with the QAPP or WAP for the following areas:

- Sample containers
- Preservation (physical and chemical)
- Chain-of-custody procedures and documentation
- Sample shipping and transport
- Conditions for storage (before analysis)
- Holding times.

8.1.1.4 Documentation

Field records generally consist of bound field notebooks with prenumbered pages, sample collection forms, sample labels or tags, sample location maps, equipment maintenance and calibration forms, chain-of-custody forms, sample analysis request forms, and field change request forms. Documentation also may include maps used to document the location of sample collection points or photographs or video to record sampling activities.

Review field records to verify they include the appropriate information to support technical interpretations, judgments, and discussions concerning project activities. Records should be legible, identifiable, and retrievable and protected against damage, deterioration, or loss. Especially note any documentation of deviations from SOPs and the QAPP.

8.1.1.5 Control Samples

Assess whether the control samples were collected or prepared as specified in the QAPP or WAP. Control samples include blanks (e.g., trip, equipment, and laboratory), duplicates, spikes, analytical standards, and reference materials that are used in different phases of the data collection process from sampling through transportation, storage, and analysis. There are many types of control samples, and the appropriate type and number of control samples to be used will depend on the data quality specifications.

See Section 7.2.4 for guidance on the type of control samples for RCRA waste-testing programs. Additional guidance on the preparation and use of QC samples can be found in the following publications:

- *Test Methods for Evaluating Solid Waste, SW-846* (USEPA 1986a), Chapter One
- *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5* (USEPA 1998a), Appendix D
- *Contract Laboratory Program (CLP) Guidance for Field Samplers - Draft Final* (USEPA 2001g), Section 3.1.1.

8.1.2 Analytical Assessment

Analytical assessment includes an evaluation of analytical and method performance and supporting documentation relative to the DQOs. Proper data review is necessary to minimize decision errors caused by out-of-control laboratory processes or calculation or transcription errors. The level and depth of analytical assessment is determined during the planning process and is dependent on the types of analyses performed and the intended use of the data.

Analytical records needed to perform the assessment of laboratory activities may include the following:

- Contract Statement of Work requirements
- SOPs
- QAPP or WAP
- Equipment maintenance documentation
- Quality assurance information on precision, bias, method quantitation limits, spike recovery, surrogate and internal standard recovery, laboratory control standard recovery, checks on reagent purity, and checks on glassware cleanliness

- Calibration records
- Traceability of standards/reagents (which provide checks on equipment cleanliness and laboratory handling procedures)
- Sample management records
- Raw data
- Correspondence
- Logbooks and documentation of deviation from procedures.

If data gaps are identified, then the assessor should prepare a list of missing information for correspondence and discussion with the appropriate laboratory representative. At that time, the laboratory should be requested to supply the information or to attest that it does not exist in any form.

8.1.2.1 Analytical Data Verification

The term **data verification** is confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. Data verification is the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements. The goal of data verification is to ensure that the data are what they purport to be, that is, that the reported results reflect what was actually done, and to document that the data fulfill specific requirements. When deficiencies in the data are identified, then those deficiencies should be documented for the data user’s review and, where possible, resolved by corrective action (USEPA 2001c).

Data verification may be performed by personnel involved with the collection of samples or data, generation of analytical data, and/or by an external data verifier. The verification process normally starts with a list of requirements that apply to an analytical data package. It compares the laboratory data package to the requirements and produces a report that identifies those requirements that were met and not met. Requirements that were not met can be referred to as exceptions and may result in flagged data. Examples of the types of exceptions that are found and reported are listed below:

- Failure to analyze samples within the required holding times
- Required steps not carried out by the laboratory (i.e., failure to maintain sample custody, lack of proper signatures, etc.)
- Procedures not conducted at the required frequency (i.e., too few blanks, duplicates, etc.)
- Contamination found in storage, extraction, or analysis of blanks
- Procedures that did not meet pre-set acceptance criteria (poor laboratory control, poor sample matrix spike recovery, unacceptable duplicate precision, etc).

The verification report should detail all exceptions found with the data packages. If the laboratory was able to provide the missing information or a suitable narrative explanation of the exceptions, they should be made part of the report and included in the data package for use by the people who determine the technical defensibility of the data.

8.1.2.2 Analytical Data Validation (Evaluation)

The term **data validation** (also known as “evaluation”) is the confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Data validation is an analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set. Data validation criteria are based upon the measurement quality objectives developed in the QAPP or similar planning document, or presented in the sampling or analytical method. Data validation includes a determination, where possible, of the reasons for any failure to meet method, procedural, or contractual requirements, and an evaluation of the impact of such failure on the overall data set (USEPA 2001c)

Data validation includes inspection of the verified data and both field and analytical laboratory data verification documentation; a review of the verified data to determine the analytical quality of the data set; and the production of a data validation report and, where applicable, qualified data. A focused data validation may also be required as a later step. The goals of data validation are to evaluate the quality of the data, to ensure that all project requirements are met, to determine the impact on data quality of those requirements that were not met, and to document the results of the data validation and, if performed, the focused data validation. The main focus of data validation is determining data quality in terms of accomplishment of measurement quality objectives.

As in the data verification process, all planning documents and procedures not only must exist, but they should also be readily available to the data validators. A data validator’s job cannot be completed properly without the knowledge of the specific project requirements. In many cases, the field and analytical laboratory documents and records are validated by different personnel. Because the data validation process requires knowledge of the type of information to be validated, a person familiar with field activities usually is assigned to the validation of the field documents and records. Similarly, a person with knowledge of analytical laboratory analysis, such as a chemist (depending on the nature of the project), usually is assigned to the validation of the analytical laboratory documents and records. The project requirements should assist in defining the appropriate personnel to perform the data validation (USEPA 2001c).

The personnel performing data validation should also be familiar with the project-specific data quality indicators (DQIs) and associated measurement quality objectives. One of the goals of the data validation process is to evaluate the quality of the data. In order to do so, certain data quality attributes are defined and measured. DQIs (such as precision, bias, comparability, sensitivity, representativeness, and completeness) are typically used as expressions of the quality of the data (USEPA 2001c).

The outputs that may result from data validation include validated data, a data validation report, and a focused validation report. For detailed guidance on data validation, see Chapter One of SW-846 and *Guidance on Environmental Data Verification and Data Validation EPA QA/G-8*

(USEPA 2001c).

8.2 Data Quality Assessment

Data quality assessment (DQA) is the scientific and statistical evaluation of data to determine if the data are of the right type, quality, and quantity to support their intended purpose (USEPA 2000d). The focus of the DQA process is on the use of statistical methods for environmental decision making – though not every environmental decisions necessarily must be made based on the outcome of a statistical test (see also Section 3). If the sampling design established in the planning process requires estimation of a parameter or testing of a hypothesis, then the DQA process can be used to evaluate the sample analysis results.

The DQA process described in this section includes five steps: (1) reviewing the DQOs and study design, (2) preparing the data for statistical analysis, (3) conducting a preliminary review of the data and checking statistical assumptions, (4) selecting and performing statistical test, and (5) drawing conclusions from the data (Figure 36).

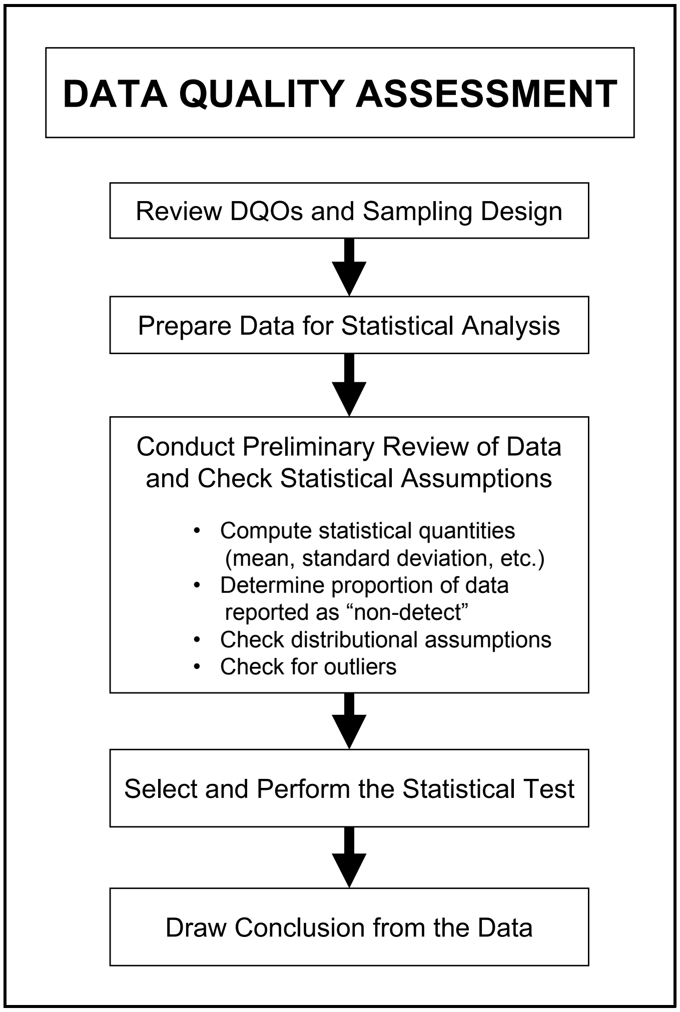

Figure 36. The DQA Process (modified from USEPA 2000d)

Detailed guidance on the statistical analysis of data can be found in Appendix F. Additional guidance can be found in *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d). A list of software tools to help you implement the DQA is provided in Appendix H.

8.2.1 Review the DQOs and the Sampling Design

Review the DQO outputs to ensure that they are still applicable. Refer back to Sections 4 and 5 of this document for more information on the DQO Process or see USEPA 2000a or 2000b. A clear understanding of the original project objectives, as determined during the systematic planning process, is critical to selecting the appropriate statistical tests (if needed) and interpreting the results relative to the applicable RCRA regulatory requirements.

8.2.2 Prepare Data for Statistical Analysis

After data validation and verification and before the data are available in a form for further analysis, several intermediate steps usually are required. For most situations, EPA recommends you prepare the data in computer-readable format. Steps in preparing data for statistical analysis are outlined below (modified from Ott 1988):

Steps in Preparing Data for Statistical Analysis

1. Receive the verified and validated data source.
2. Create a data base from the verified and validated data source.
3. Check and edit the data base.
4. Create data files from the data base.

1. *Receive the verified and validated source from the QA reports.* Data are supplied to the user in a variety of formats and readiness for use, depending on the size and complexity of the study and the types of analyses requested. Most laboratories supply a QA evaluation package that includes the verification/validation review, a narrative, tabulated summary forms (including the results of analyses of field samples, laboratory standards, and QC samples), copies of logbook pages, and copies of chain-of-custody records. From this information, you can create a data base for statistical analysis.

2. *Create a data base from the verified and validated data source.* For most studies in which statistical analyses are scheduled, a computer-readable data base is the most efficient method for managing the data. The steps required to create the data base and the format used will depend on the software systems used to perform the analysis. For example, the data base may be as simple as a string of concentration values for a single constituent input into a spreadsheet or word processor (such as required for use of EPA's DataQUEST software (USEPA 1997b)), or it may be more complex, requiring multiple and related data inputs, such as sample number, location coordinates, depth, date and time of collection, constituent name and concentration, units of measurements, test method, quantitation limit achieved, QC information, etc.

   If the data base is created via manual data entry, the verified and validated data should be checked for legibility. Any questions pertaining to illegible information should be resolved before the data are entered. Any special coding considerations, such as indicating values reported as "nondetect" should be specified in a coding guide or in the QAPP. For very large projects, it may be appropriate to prepare a separate detailed data management plan in advance.

3. *Check and edit the data base.* After creation of the data set, the data base should be checked against the data source to verify accurate data entry and to correct any errors discovered. Even if the data base is received from the laboratory in electronic format, it should be checked for obvious errors, such as unit errors, decimal errors, missing values, and quantitation limits.

4. *Create data files from the data base.* From the original data files, work files are created for use within the statistical software package. This step could entail separating data by constituent and by DQO decision unit and separating any QA/QC data from the record data. When creating the final data files for use in the statistical software, be sure to use a file naming and storage convention that facilitates easy retrieval for future use, reference, or reporting.

8.2.3 Conduct Preliminary Review of the Data and Check Statistical Assumptions

Many statistical tests and procedures require that certain assumptions be met for their use. Failure to satisfy these assumptions can result in biased estimates of the parameter of interest; therefore, it is important to conduct preliminary analyses of the data to learn about the characteristics. EPA recommends that you compute statistical quantities, determine the proportion of the data reported as “nondetect” for each constituent of concern, check whether the data exhibit a normal distribution, then determine if there are any “outliers” that deserve a closer look. The outputs of these activities are used to help select and perform the appropriate statistical tests.

8.2.3.1 Statistical Quantities

To help “visualize” and summarize the data, calculate basic statistical quantities such as the:

- Mean
- Maximum
- Percentiles
- Variance
- Standard deviation
- Coefficient of variation.

Calculate the quantities for each constituent of concern. Example calculations of the mean, variance, standard deviation, and standard error of the mean are given in Section 3. Detailed guidance on the calculation of statistical quantities is provided in Chapter Two of EPA’s *QA/G-9* guidance document (USEPA 2000d). The useful quantities easily can be computed using EPA’s DataQUEST software (USEPA 1997b, see also Appendix H) or any similar statistical software package.

When calculating statistical quantities, determine which data points were reported as below a limit of detection or quantitation - known as “nondetects” (NDs). See also Section 8.2.4.2 (“Treatment of Nondetects”).

8.2.3.2 Checking Data for Normality

Check the data sets for normality by using graphical methods, such as histograms, box and whisker plots, and normal probability plots (see also Section 3.1.3), or by using numerical tests, such as the Shapiro-Wilk test for normality (see Appendix F). Table 11 provides a summary of recommended methods. Detailed guidance on the use of graphical and statistical methods can be found in USEPA 1989b, 1992b, 1997b, and 2000d.

Table 11. Recommended Graphical and Statistical Methods for Checking Distributional Assumptions

Test	*Use*	*Reference*
Graphical Methods		
Histograms and frequency plots	Provides visual display of probability or frequency distribution	See USEPA 2000d. Construct via EPA's DataQUEST software (USEPA 1997b) or use a commercial software package.
Normal probability plot	Provides visual display of deviation from expected normality	See USEPA 2000d. Construct via EPA's DataQUEST software (USEPA 1997b) or use a commercial software package.
Box and Whisker Plot	Provides visual display of potential "outliers" or extreme values	See USEPA 2000d. Construct via EPA's DataQUEST software (USEPA 1997b) or use a commercial software package.
Numerical Tests for Normality		
Shapiro-Wilk Test	Use for sample sizes of ≤ 50	See procedure in Appendix F, Section F.1.2. This test also can be performed using EPA's DataQUEST software (USEPA 1997b).
Filliben's Statistic	Use for sample sizes of > 50	See USEPA 2000d. This test can be performed using EPA's DataQUEST software (USEPA 1997b).

Graphical methods allow you to visualize the central tendency of the data, the variability in the data, the location of extreme data values, and any obvious trends in the data. For example, a symmetrical "mound" shape of a histogram is an indicator of an approximately normal distribution. If a normal probability plot is constructed on the data (see Figure 5 in Section 3.1.3), a straight line plot usually is an indicator of normality. (Note that interpretation of a probability plot depends on the method used to construct it. For example, in EPA's DataQUEST software, normally distributed data will form an "S"-shaped curve rather than a straight line on a normal probability plot.)

The Shapiro-Wilk test is recommended as a superior method for testing normality of the data. The specific method for implementing the Shapiro-Wilk Test is provided in Appendix F. The method also is described in Gilbert (1987), EPA's guidance on the statistical analysis of ground-water monitoring data (USEPA 1992b), and can be performed with EPA's DataQUEST software or other commercially available statistical software.

8.2.3.3 How To Assess "Outliers"

A measurement that is very different from other values in the data set is sometimes referred to as an "outlier." EPA cautions that the term "outlier" be used advisedly, since a common reaction to the presence of "outlying" values has been to "cleanse the data," thereby removing any "outliers" prior to further analysis. In fact, such discrepant values can occur for many reasons, including (1) a catastrophic event such as a spill or process upset that impacts measurements at the sampling point, (2) inconsistent sampling or analytical chemistry methodology that may result in laboratory contamination or other anomalies, (3) errors in the transcription of data values or decimal points, and (4) *true* but extreme hazardous constituent measurements.

While any one of these events can cause an apparent “outlier,” it should be clear that the appropriate response to an outlier will be very different depending on the origin. Because high values due to contaminated media or waste are precisely what one may be trying to identify, it would not be appropriate to eliminate such data in the guise of “screening for outliers.” Furthermore, depending on the form of the underlying population, unusually high concentrations may be real but infrequent such as might be found in lognormally distributed data. Again, it would not be appropriate to remove such data without adequate justification.

A *statistical outlier* is defined as a value originating from a different underlying population than the rest of the data set. If the value is not consistent with the distributional behavior of the remaining data and is “too far out in one of the tails” of the assumed underlying population, it may test out as a statistical outlier. Defined as it is strictly in statistical terms, however, an outlier test may identify values as discrepant when no physical reason can be given for the aberrant behavior. One should be especially cautious about indiscriminate testing for statistical outliers for this reason.

If an outlier is suspected, an initial and helpful step is to construct a probability plot of the data set (see also Section 3.1.3 and USEPA 2000d). A probability plot is designed to judge whether the sample data are consistent with an underlying normal population model. If the rest of the data follow normality, but the outlier comes from a distinctly different population with higher (or lower) concentrations, this behavior will tend to show up on a probability plot as a lone value “out of line” with the remaining observations. If the data are lognormal instead, but the outlier is again from a distinct population, a probability plot on the logged observations should be constructed. Neither of these plots is a formal test; still, they provide invaluable visual evidence as to whether the suspected outlier should really be considered as such.

Methods for conducting outlier tests are described in Chapter 4 of EPA’s QA/G-9 guidance document (USEPA 2000d), and statistical tests are available in the DataQUEST software (for example, Rosner’s Test and Walsh’s Test) (USEPA 1997b).

8.2.4 Select and Perform Statistical Tests

This section provides guidance on how you can select the appropriate statistical test to make a decision about the waste or media that is the subject of the study. It is important to select the appropriate statistical test because decisions and conclusions derived from incorrectly used statistics can be expensive (Singh, et al. 1997).

Prior to selecting the statistical test, consider the following factors:

- The objectives of the study (identified in DQO Step 2)
- Whether assumptions of the test are fulfilled
- The nature of the underlying distribution

- The decision rule and null hypothesis (identified in DQO Step 5)
- The relative performance of the candidate tests (for example, parametric tests generally are more efficient than their nonparametric counterparts)
- The proportion of the data that are reported as nondetects (NDs).

The decision-tree presented in Figure 37 provides a starting point for selecting the appropriate statistical test. The statistical methods are offered as guidance and should not be used as a "cook book" approach to data analysis. The methods presented here usually will be adequate for the tests conducted under the specified conditions (see also Appendix F). An experienced statistician should be consulted whenever there are questions.

Based on the study objective (DQO Step 2), determine which category of statistical tests to use. Note the statistical methods recommended in the flow charts in Figure 38 and Figure 39 are for use when the objective is to compare the parameter of interest to a fixed standard. Other methods will be required if the objective is different (e.g., when comparing two populations, detecting trends, and evaluating spatial patterns or relationships of sampling points).

8.2.4.1 Data Transformations in Statistical Tests

Users of this guidance may encounter data sets that show significant evidence of non-normality. Due to the assumption of underlying normality in most parametric tests, a common statistical strategy when encountering this predicament is to search for a mathematical transformation that will lead to normally-distributed data on the transformed scale. Unfortunately, because of the complexities associated with interpreting statistical results from data that have been transformed to another scale and the common occurrence of lognormal patterns in environmental data, EPA generally recommends that the choice of scale be limited to either the original measurements (for normal data) or a log-transformed scale (for lognormal data). If neither of these scales results in approximate normality, it is typically easiest and wisest to switch to a nonparametric (or “distribution-free”) version of the same test.

If a transformation to the log scale is needed, and a confidence limit on the mean is desired, special techniques are required. If a data set exhibits a normal distribution on the log-transformed scale, it is a common mistake to assume that a standard normal-based confidence interval formula can be applied to the transformed data with the confidence interval endpoints retransformed to the original scale to obtain the confidence interval on the mean. Invariably, such an interval will be biased to the low side. In fact, the procedure just described actually produces a confidence interval around the *median* of a lognormal population, rather than the higher *mean*. To correctly account for this “transformation bias”, special procedures are required (Land 1971 and 1975, Gilbert 1987). See Section F.2.3 in Appendix F for detailed guidance on calculating confidence limits for the mean of a lognormal population.

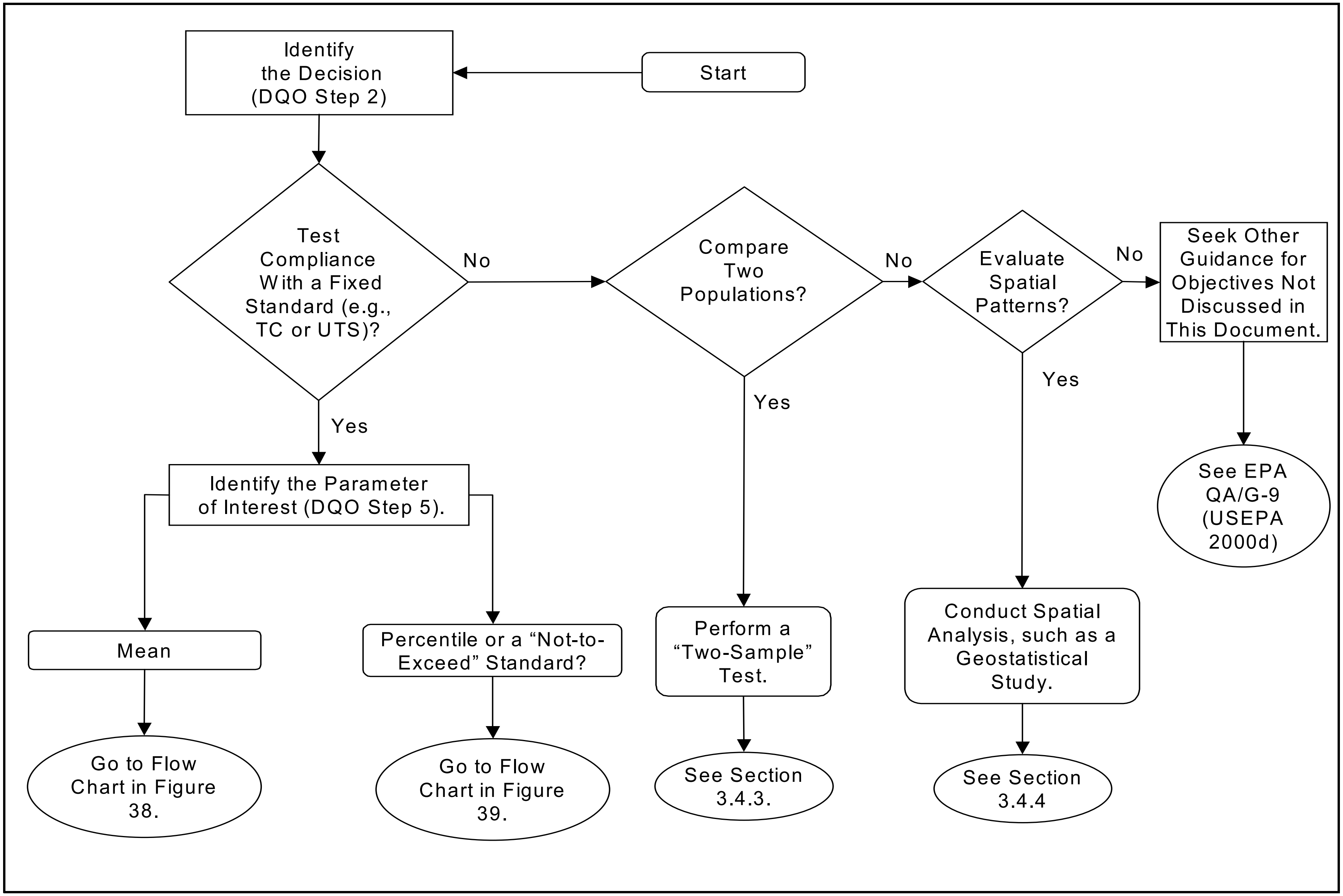

Figure 37. Flow chart for selecting a statistical method

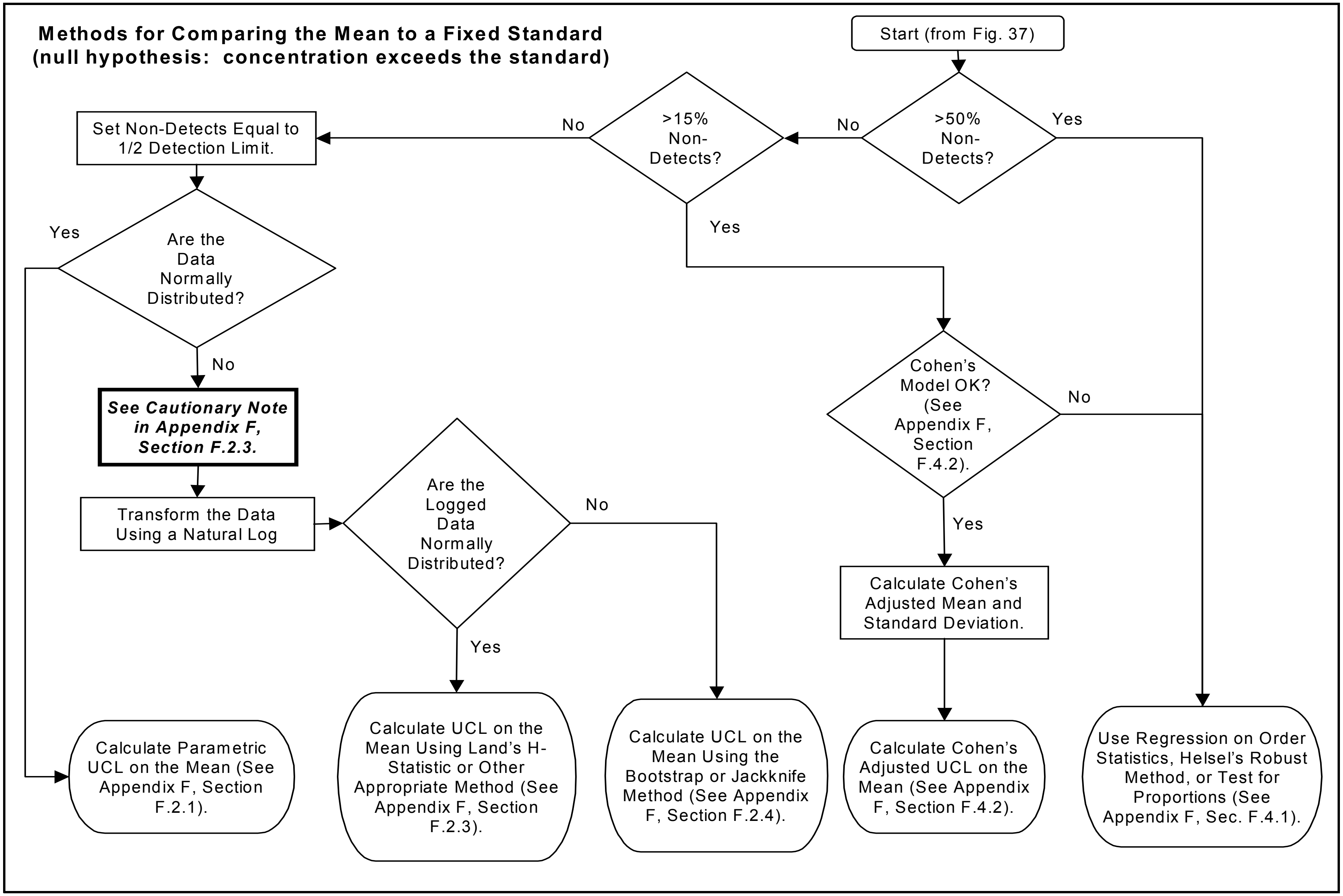

Figure 38. Flowchart of statistical methods for comparing the mean to a fixed standard (null hypothesis is "concentration exceeds the standard")

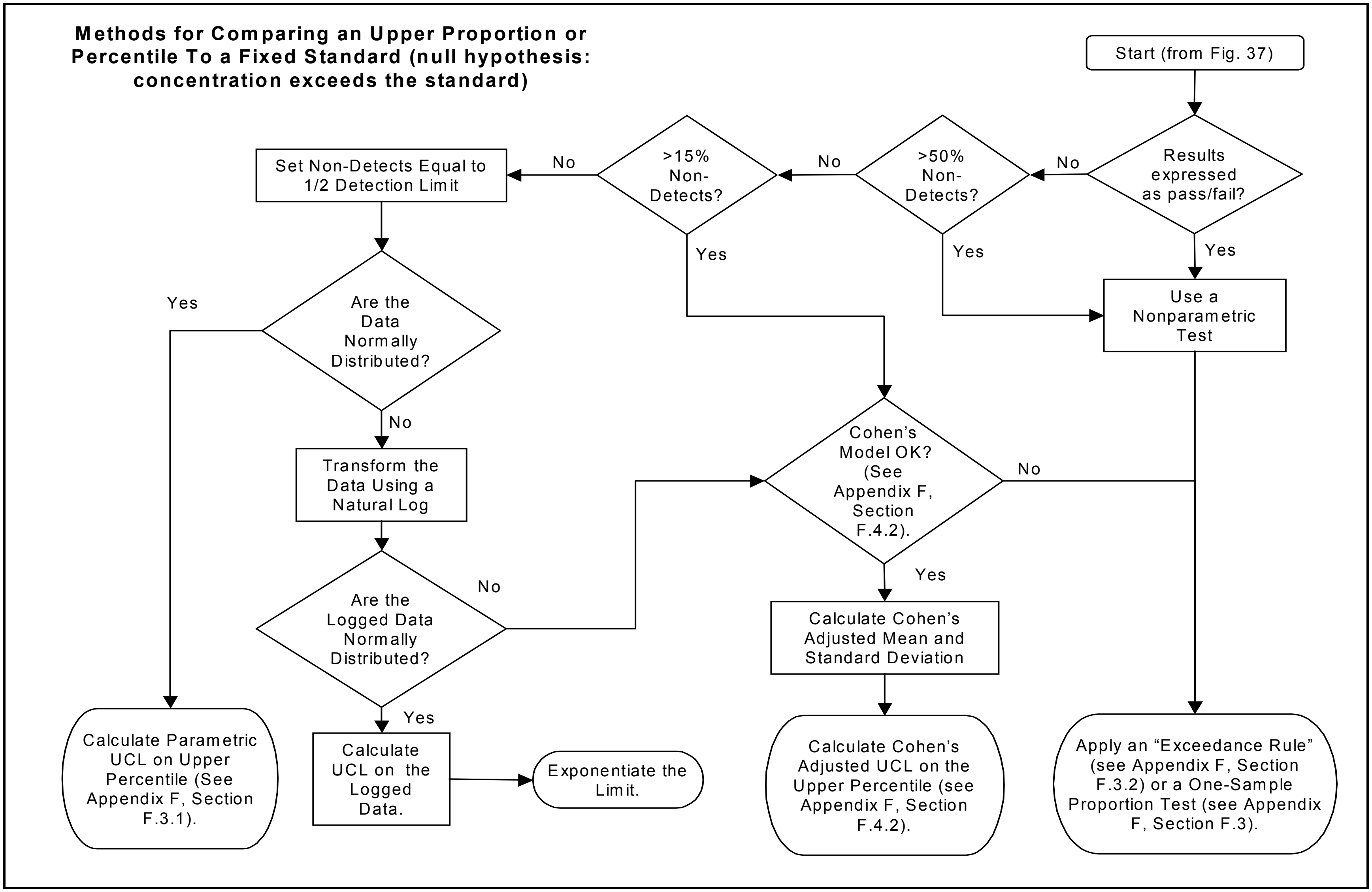

Figure 39. Flowchart of statistical methods for comparing an upper proportion or percentile to a fixed standard (null hypothesis is "concentration exceeds the standard")

If the number of samples is small, it may not be possible to tell whether the distribution is normal, lognormal, or any other specific function. You are urged not to read too much into small data sets and not to attempt overly sophisticated evaluations of data distributions based on limited information. If the distribution of data appears to be highly skewed, it is best to take operational measures (such as more samples or samples of a larger physical size) to better characterize the waste.

8.2.4.2 Treatment of Nondetects

If no more than approximately 15 percent of the samples for a given constituent are nondetect (i.e., reported as below a detection or quantitation limit), the results of parametric statistical tests will not be substantially affected if nondetects are replaced by half their detection limits (known as a substitution method) (USEPA 1992b). When a larger percentage of the sample analysis results are nondetect, however, the treatment of nondetects is more crucial to the outcome of statistical procedures. Indeed, simple substitution methods (such as replacing the detection limit with one-half the detection limit) tend to perform poorly in statistical tests when the nondetect percentage is substantial (Gilliom and Helsel 1986, Helsel 1990).

Guidance on selecting an approach for handling nondetects in statistical intervals is given in Appendix F, Section F.4. Guidance also is given in Section 4.7 of EPA's *Guidance for Data Quality Assessment Practical Methods for Data Analysis EPA QA/G-9* (USEPA 2000d).

8.2.5 Draw Conclusions and Report Results

The final step in the DQA Process is to draw conclusions from the data, determine if further sampling is required, and report the results. This step brings the planning, implementation, and assessment process "full circle" in that you attempt to resolve the problem and make the decision identified in Steps 1 and 2 of the DQO Process.

In the DQO Process, you establish a "null hypothesis" and attempt to gather evidence via sampling that will allow you to reject that hypothesis; otherwise, the null hypothesis must be accepted. If the decision making process involves use of a statistical method (such as the calculation of a statistical confidence limit or use of a statistical hypothesis test), then the outcome of the statistical test should be reported along with the uncertainty associated with the result. If other decision making criteria are used (such as use of a simple exceedance rule or a "weight of evidence" approach), then the outcome of that decision making process should be reported.

Detailed guidance on the use and interpretation of statistical methods for decision making can be found in **Appendix F**. Additional guidance can found in EPA's *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d).

Most of the statistical methods suggested in this document involve the construction of one-sided confidence limits (or bounds). The upper confidence limit, whether calculated on a mean, median, or percentile, provides a value below which one can claim with specified confidence that the true value of the parameter lies. Figure 40 demonstrates how you can use a confidence limit to test a hypothesis: In the situation depicted at "A," the upper confidence limit calculated from the sample data is less than the applicable standard and provides the evidence needed to reject the null hypothesis. The decision can be made that the waste concentration is below the standard with sufficient confidence and without further analysis.

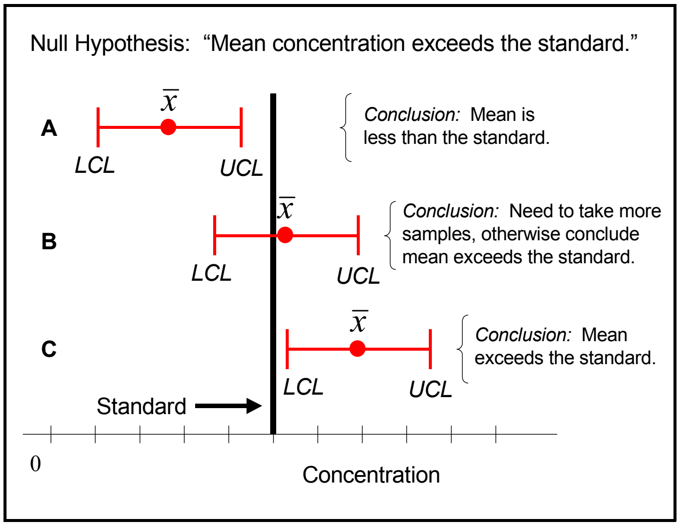

Figure 40. Using confidence limits on the mean to compare waste concentrations to a fixed standard.

In situation "B," we cannot reject the null hypothesis; however, because the interval "straddles" the standard, it is *possible* that the true mean lies below the standard and a Type II (false acceptance) error has been made (i.e., to conclude the concentration is above the standard, when in fact it is not). One possible remedy to this situation is to obtain more data to "tighten" the confidence interval.

In situation "C," the Type II (false acceptance) decision error rate is satisfied and we must conclude that the mean concentration exceeds the standard.

One simple method for checking the performance of the statistical test is use the information obtained from the samples to retrospectively estimate the number of samples required. For example, the sample variance can be input into the sample size equation used (see Section 5.4 and 5.5, DQO Process Step 7). (An example of this approach is presented in Appendix I.) If this theoretical sample size is less than or equal to the number of samples actually taken, then the test is sufficiently powerful. If the required number of samples is greater than the number actually collected, then additional samples would be required to satisfy the data user's performance criteria for the statistical test. See EPA's *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d) for additional guidance on this topic.

Finally, if a simple exceedance rule is used to measure compliance with a standard, then interpretation of the results is more straightforward. For example, if zero exceedances are allowed, and one or more samples exceeds the standard, then there is evidence of noncompliance with that standard (see Appendix F, Section F.3.2).

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