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Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities[1]

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1. Scope

1.1 Compositing and subsampling are key links in the chain of sampling and analytical events that must be performed in compliance with project objectives and instructions to ensure that the resulting data are representative. This guide discusses the advantages and appropriate use of composite sampling, field procedures and techniques to mix the composite sample, and procedures to collect an unbiased and precise subsample(s) from a larger sample. It discusses the advantages and limitations of using composite samples in designing sampling plans for characterization of wastes (mainly solid) and potentially contaminated media. This guide assumes that an appropriate sampling device is selected to collect an unbiased sample.

1.2 The guide does not address: where samples should be collected (depends on the objectives) (see Guide D6044), selection of sampling equipment, bias introduced by selection of inappropriate sampling equipment, sample collection procedures or collection of a representative specimen from a sample, or statistical interpretation of resultant data and devices designed to dynamically sample process waste streams. It also does not provide sufficient information to statistically design an optimized sampling plan, or determine the number of samples to collect or calculate the optimum number of samples to composite to achieve specified data quality objectives (see Practice D5792). Standard procedures for planning waste sampling activities are addressed in Guide D4687.

1.3 The sample mixing and subsampling procedures described in this guide are considered inappropriate for samples to be analyzed for volatile organic compounds. Volatile organics are typically lost through volatilization during sample collection, handling, shipping, and laboratory sample preparation unless specialized procedures are used. The enhanced mixing described in this guide is expected to cause significant losses of volatile constituents. Specialized procedures should be used for compositing samples for determination of volatiles such as combining directly into methanol (see Guide D4547).

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C702/C702M Practice for Reducing Samples of Aggregate to Testing Size

D1129 Terminology Relating to Water

D4439 Terminology for Geosynthetics

D4547 Guide for Sampling Waste and Soils for Volatile Organic Compounds

D4687 Guide for General Planning of Waste Sampling

D5088 Practice for Decontamination of Field Equipment Used at Waste Sites

D5792 Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives

D6044 Guide for Representative Sampling for Management of Waste and Contaminated Media

E856 Definitions of Terms and Abbreviations Relating to Physical and Chemical Characteristics of Refuse Derived Fuel (Withdrawn 2011)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *composite sample, n*—a combination of two or more samples. **D1129**

[1] This guide is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.01 on Planning for Sampling.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

3.1.2 *sample, n*—a portion of material taken from a larger quantity for the purpose of estimating properties or composition of the larger quantity. **E856**

3.1.3 *specimen, n*—a specific portion of a material or laboratory sample upon which a test is performed or which is taken for that purpose. **D4439**

3.1.4 *subsample, n*—a portion of a sample taken for the purpose of estimating properties or composition of the whole sample.

3.1.4.1 *Discussion*—a subsample, by definition, is also a sample.

4. Summary of Guide

4.1 This guide describes how the collection of composite samples, as opposed to individual samples, may be used to: more precisely estimate the mean concentration of a waste analyte in contaminated media, reduce costs, efficiently determine the absence or possible presence of a hot spot (a highly contaminated local area), and, when coupled with retesting schemes, efficiently locate hot spots. Specific procedures for mixing a sample(s) and collecting subsamples for transport to a laboratory are provided.

5. Significance and Use

5.1 This guide provides guidance to persons managing or responsible for designing sampling and analytical plans for determining whether sample compositing may assist in more efficiently meeting study objectives. Samples must be composited properly, or useful information on contamination distribution and sample variance may be lost.

5.2 The procedures described for mixing samples and obtaining a representative subsample are broadly applicable to waste sampling where it is desired to transport a reduced amount of material to the laboratory. The mixing and subsampling sections provide guidance to persons preparing sampling and analytical plans and field personnel.

5.3 While this guide generally focuses on solid materials, the attributes and limitations of composite sampling apply equally to static liquid samples.

6. Attributes of Composite Sampling for Waste Characterization

6.1 In general, the individual samples to be composited should be of the same mass; however, proportional sampling may be appropriate in some cases depending upon the objective. For example, if the objective is to determine the average drum concentration of a contaminant, compositing equal volumes of waste from each drum would be appropriate. If the objective is to determine average contaminant concentration of the waste contained in a group of drums, the volume of each sample to be composited should be proportional to the amount of waste in each drum. Another example of proportional sampling is estimating the contaminant concentration of soil overlying an impermeable zone. Soil cores should be collected from the surface to the impermeable layer, regardless of core length.

6.2 The principal advantages of sample compositing include: reduction in the variance of an estimated average concentration (**1**),[4] increasing the efficiency of locating/identifying hot spots (**2**), and reduction of sampling and analytical costs (**3**). These main advantages are discussed in the following paragraphs. However, a principle assumption needed to justify compositing is that analytical costs are high relative to sampling costs. In general, appropriate use of sample compositing can:

6.2.1 Reduce inter-sample variance, that is, improve the precision of the mean estimation while reducing the probability of making an incorrect decision;

6.2.2 Reduce costs for estimating a total or mean value, especially where analytical costs greatly exceed sampling costs (also may be effective when analytical capacity is a limitation);

6.2.3 Efficiently determine the absence or possible presence of hot spots or hot containers and, when combined with retesting schemes, identify hot spots, as long as the probability of hitting a hot spot is low;

6.2.4 Be especially useful for situations, where the nature of contaminant distribution tends to be contiguous and nonrandom and the majority of analyses are "non-detects" for the contaminant(s) of interest; and

6.2.5 Provide a degree of anonymity where population, rather than individual statistics, are needed.

6.3 *Improvement in Sampling Precision*—Samples are always taken to make inferences to a larger volume of material, and a set of composite samples from a heterogeneous population provides a more precise estimate of the mean than a comparable number of discrete samples. This occurs because compositing is a "physical process of averaging." Averages of samples have greater precision than the individual samples. Likewise, a set of composite samples is always more precise than an equal number of individual samples. Decisions based on a set of composite samples will, for practical purposes, always provide greater statistical confidence than for a comparable set of individual samples.

6.3.1 If an estimated precision of a mean is desired, then more than one composite sample is needed; a standard deviation cannot be calculated from one composite sample. However, the precision of a single composite sample may be estimated when there are data to show the relationship between the precision of the individual samples that comprise the composite sample and that of the composite sample. The precision (standard deviation) of the composite sample is approximately the precision of the individual samples divided by the square root of the number of individual samples in the composite.

6.4 *Example 1*—An example of how a single composite sample can be used for decision-making purposes is given here. Assume a regulatory limit of 1 mg/kg and a standard deviation of 0.5 mg/kg for the individual samples. If the concentration of a site is estimated to be around 0.6 mg/kg, how many individual samples should be composited to have relatively high confidence that the true concentration does not

[4] The boldface numbers in parentheses refer to a list of references at the end of this guide.

exceed the regulatory limit when only one composite sample is used? Assuming the composite is well mixed, then the precision of a composite is a function of the number of samples as follows:

Number of Individual Samples in Composite	Precision (standard deviation $\div\sqrt{n}$) of One Composite Sample
2	0.35
3	0.29
4	0.25
5	0.22
6	0.20

Thus, if six samples are included in a composite, the composite concentration of 0.6 mg/kg is two standard deviations below the regulatory limit. Therefore, if the composite concentration is actually observed to be in the neighborhood of 0.6 mg/kg, we can be reasonably confident (approximately 95 %) that the concentration of the site is below the regulatory limit, using only one composite sample.

6.5 *Example 2*—Another example is when the standard deviation of the individual samples in the previous example is relatively small, say 0.1 mg/kg. Then the standard deviation of a composite of six individual samples is 0.04 mg/kg (0.1 mg/kg divided by the square root of 6 = 0.04 mg/kg), a very small number relative to the regulatory limit of 1 mg/kg. In this case, simple comparison of the composite concentration to the regulatory limit is often quite adequate for decision-making purposes.

6.5.1 The effectiveness of compositing depends on the relative magnitude of sampling and analytical error. When sampling uncertainty is high relative to analytical error (as is usually assumed to be the case), compositing is very effective in improving precision. If analytical errors are high relative to field errors, sample compositing is much less effective.

6.5.2 Because compositing is a physical averaging process, composite samples tend to be more normally distributed than the individual samples. The normalizing effect is frequently an advantage since calculation of means, standard deviations, and confidence intervals generally assume the data are normally distributed. Although environmental residue data are commonly non-normally distributed, compositing often leads to approximate normality and avoids the need to transform the data.

6.5.3 The spatial design of the compositing scheme can be important. Depending upon the locations from which the individual samples are collected and composited, composites can be used to determine spatial variability or improve the precision of the parameter being estimated. Figs. 1 and 2 represent a site divided into four cells. Composite all samples with the same number together. The sampling approach in Fig. 1 is similar to sample random sampling, except they are now composite samples. Each composite sample in this case is a representative sample of the entire site, eliminates cell-to-cell variability, and leads to increased precision in estimating the mean concentration of the site. If there is a need to estimate the cell-to-cell variability, then the approach in Fig. 2 is suitable. In addition, if the precision of estimating the mean concentration of the cell is needed, multiple composite samples should be collected from that cell.

1	2	4	3
4	3	2	1
4	2	1	4
3	1	2	3

FIG. 1 Example of Composing Across a Site

1	1	2	2
1	1	2	2
3	3	4	4
3	3	4	4

FIG. 2 Example of Within-Cell Compositing

6.6 *Effect on Cost Reduction*—Because the composite samples yield a more precise mean estimate than the same number of individual samples, there is the potential for substantial cost saving. Given the higher precision associated with composite samples, the number of composite samples required to achieve a specified precision is smaller than that required for individual samples. This cost saving opportunity is especially pronounced when the cost of sample analysis is high relative to the cost of sampling, compositing, and analyzing.

6.7 *Hot Container/Hot Spot Identification and Retesting Schemes*—Samples can be combined to determine whether an individual sample exceeds a specified limit as long as the action limit is relatively high compared with the actual detection limit and the average sample concentration. Depending on the difficulty and probability of having to resample, it may be desirable to retain a split of the discrete samples for possible analysis depending on the analytical results from the composite sample.

6.8 *Example 3*—One hundred drums are to be examined to determine whether the concentration of PCBs exceeds 50 mg/kg. Assume the detection limit is 5 mg/kg and most drums have non-detectable levels. Compositing samples from ten drums for analysis would permit determining that none of the drums in the composite exceeds 50 mg/kg as long as the concentration of the composite is <5 mg/kg. If the detected concentration is >5 mg/kg, one or more drums may exceed 50 mg/kg and additional analyses of the individual drums are required to identify any hot drum(s). The maximum number of samples that can theoretically be composited and still detect a hot sample is the limit of concern divided by the actual detection limit (for example, 50 mg/kg ÷ 5 mg/kg = 10).

6.9 *Example 4*—Assume background levels of dioxin are non-detectable, and the analytical detection limit is 1 µg/kg and the action level is 50 µg/kg. The site is systematically gridded (the most efficient sampling design for detecting randomly distributed hot spots) using an appropriate design, and cores to a depth of 10 cm are collected. Composite samples are collected since analytical costs for dioxin are high. In theory,

groups of up to 50 samples could be composited and if the resultant concentration were <1 µg/kg, all samples represented in the composite should be below 50 µg/kg. If the contaminant concentration is >1 µg/kg, one or more spots may exist that exceed 50 µg/kg in the area covered by the composite sample, although the precise location and areal extent would not be known without further sampling and analyses. Compositing fewer samples would probably be more practical, however.

6.9.1 The relative efficiency of compositing individual samples to detect a hot spot depends on the probability of a "hot" discrete sample being used to form a composite sample. According to Garner et al. (**1**), if the probability can be estimated as low, say 1 %, the optimum number of samples to composite is about ten, which would result in a cost saving of about 80 % (assuming there is no detection limit problem). When the probability of collecting a sample from a hot spot rises to 10 %, the optimal number of samples to composite is 4, which results in a 40 % cost savings. By the time the probability of sampling a hot spot rises to 40 %, there is no cost benefit to compositing. Other resampling and testing schemes are possible and may lead to somewhat different cost saving potentials.

7. Limitations of Composite Sampling

7.1 The principal limitations of sample compositing involve the loss of the discrete information contained in a single sample and the potential for dilution of the contaminants in a sample with uncontaminated material; however, in that case, the dilution factor can be used to estimate the maximum number of samples that can be composited. The following situations may not lend themselves to cost-effective sample compositing:

7.1.1 When the integrity of individual sample values change because of compositing, for example, chemical interaction occurs between constituents in the samples being combined or volatiles are lost during mixing;

7.1.2 Where the composite sample cannot be properly mixed and subsampled or the whole composite sample cannot be analyzed;

7.1.3 When the goal is to detect hot spots and a large proportion of the samples are expected to test positive for an attribute, compositing and retesting schemes may not be cost effective;

7.1.4 When analytical costs are low relative to sampling costs (for example, in situ field portable X-ray fluorescence takes only 30 s with no sample preparation so analytical costs/sample are very low); and

7.1.5 When regulations specify that a grab sample must be collected (usually a composite sample covering a limited area is still preferred from a technical standpoint).

8. Sample Mixing Procedures

8.1 Prior to sample mixing, project-specific instructions should be followed regarding sample collection, which may include removal of extraneous sample materials such as twigs, grass, rocks, etc. If samples are sieved or large materials are removed, it may be necessary to record the mass of materials removed for later estimation of contaminant concentration in the original sample. According to particulate sampling theory (**4, 5**) the following sample masses are adequate to represent the corresponding maximum size particles in the sample with a relative standard deviation of 15 %.

Sample Mass, g	Maximum Particle Size, cm
5	0.170
50	0.37
100	0.46
500	0.79
1000	1.0
5000	1.7

8.1.1 Frequently it is necessary to mix an individual or composite sample and obtain a representative subsample(s) for transport to the analytical laboratory. This occurs when multiple containers of the identical material are desired (for example, separate sample jars for metals, semivolatile organics, etc. are desired) or when the original sample (or composite sample) size is greater than accepted by the laboratory. Even when the original sample volume is acceptable, it may be desirable to thoroughly mix the sample prior to transport to an analytical laboratory. However, some samples that have been well mixed in the field may segregate during shipment to the laboratory.

8.1.2 A laboratory typically collects a 0.5 to 30 g specimen (100 g for some extraction tests) from the sample for analysis. Specimens are frequently collected from the surface material in the container or after minimal mixing. Such procedures are inadequate to obtain a small representative specimen from a 100 to 300 g sample. Special mixing and subsampling procedures are necessary to obtain a representative subsample unless the sample is already homogenous. Field mixing should be considered essential unless it is known that the sample in the container is homogeneous or it is known that the laboratory will homogenize the sample and collect a representative specimen. To help ensure that an unbiased and precise specimen is collected, the analytical laboratory should be provided instructions (preferably with the sample shipment) on homogenizing and obtaining a specimen for analysis. Few laboratories follow good sample homogenizing and specimen collection practices. To meet both sampling and analytical objectives, field and analytical personnel and the end-user of the data must be aware of the laboratories' standard practices for handling, mixing, and obtaining a specimen or specify such practices with the sample shipment.

8.1.3 To avoid subsampling, it may be possible to collect a small sample (or composite samples) directly into the sample container that is delivered to the laboratory. (**Caution:** Small sample sizes may result in bias by excluding large particles.) While no field mixing and subsampling are needed as long as the laboratory homogenizes the sample, it may be advisable to mix such samples anyway (see 8.1.2).

8.1.4 Soil, sediment, sludge, and waste samples collected for purgeable/volatile organic compounds analyses should *not* be mixed and subsampled using procedures described in this guide, but other specialized procedures such as combining samples directly into methanol (see Guide D4547) may be appropriate.

8.1.5 A significant problem with analyzing very small samples is that the smaller the volume of sample actually extracted or analyzed, the less representative that sample may be unless thoroughly mixed/homogenized and subsampled.