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# Standard Guide for General Planning of Waste Sampling<sup>1</sup>

This standard is issued under the fixed designation D4687; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

#### INTRODUCTION

The analysis and testing of solid waste requires collection of adequately sized, representative samples. Wastes are found in various locations and physical states. Therefore, each sampling routine shall be tailored to fit the waste and situation. Wastes often occur as nonhomogeneous mixtures in stratified layers or as poorly mixed conglomerations. For example, wastes are commonly stored or disposed of in surface impoundments with stratified or layered sludges covered by ponded wastewater. In these situations, the collector may be faced with sampling the wastewater, the sludge, and some depth of soil beneath the sludges. Collecting representative samples in these situations requires a carefully assessed, well-planned, and well-executed sampling routine.

# 1. Scope

- 1.1 This guide provides information for formulating and planning the many aspects of waste sampling (see 1.2) that are common to most waste sampling situations.
- 1.2 The aspects of sampling that this guide addresses are as follows:

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- 1.3 This guide does not provide comprehensive sampling procedures for these aspects, nor does it serve as a guide to any specific application. It is the responsibility of the user to ensure that the procedures used are proper and adequate.
- 1.4 *Units*—The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.
- 1.5 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 deter-

1.6 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:<sup>2</sup>

E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

2.2 Other Document:

EPA-SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods<sup>3</sup>

### 3. Significance and Use

3.1 The procedures covered in this guide are general and provide the user with information helpful for writing sampling plans, safety plans, labeling and shipping procedures, chain-of-custody procedures, general sampling procedures, general cleaning procedures, and general preservation procedures.

mine the applicability of regulatory limitations prior to use. For more specific precautionary statements see 3.2, 3.3, and Section 4.

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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from Superintendent of Documents, U.S. Printing Office, Washington, DC 20402.

- 3.2 For purposes of this guide, it is assumed that the user has knowledge of the waste being sampled and the possible safety hazards.
- 3.3 This guide is not to be used when sampling sites or wastes when safety hazards are unknown. In such cases, the user shall use other more appropriate procedures.

#### 4. Safety Plans

4.1 Proper safety precautions shall always be observed when sampling wastes. Persons collecting samples shall be aware that the waste can be a strong sensitizer and corrosive, flammable, explosive, toxic, and capable of releasing extremely poisonous gases. The background information obtained about the waste should be helpful in deciding the extent of safety precautions to be observed and in choosing protective equipment to be used. The information obtained should be checked for hazardous properties against such references as "Sax's Dangerous Properties of Industrial Materials," the "Hawley's Condensed Chemical Dictionary," and the "Toxic and Hazardous Industrial Chemicals Safety Manual for Handling and Disposal with Toxicity and Hazard Data."

Note 1—The following safety precautions are not comprehensive. Rather, they provide additional guidance on health and safety to complement professional judgment and experience.

- 4.2 Personnel should wear protective equipment when response activities involve known or suspected atmospheric contamination, vapors, gases, or airborne particulates may be generated, or direct contact with skin-affecting substances may occur. Respirators can protect lungs, gastrointestinal tract, and eyes against air toxicants. Chemical-resistant clothing can protect the skin from contact with skin-destructive and absorbable chemicals. Good personal hygiene limits or prevents ingestion of material.
- 4.2.1 Equipment to protect the body against contact with known or anticipated chemical hazards has been divided into four categories according to the degree of protection afforded:
- 4.2.1.1 *Level A*—Should be worn when the highest level of respiratory, skin, and eye protection is needed.
- 4.2.1.2 *Level B*—Should be selected when the highest level of respiratory protection is needed, but a lesser level of skin protection. Level B protection is the minimum level recommended on initial site entries until the hazards have been further defined by on-site studies and appropriate personnel protection utilized.
- 4.2.1.3 *Level C*—Should be selected when the type(s) of airborne substance(s) is (are) known, the concentrations(s) is measured, and the criteria for using air-purifying respirators are met.
- 4.2.1.4 *Level D*—Should not be worn on any site with respiratory or skin hazards. It is primarily a work uniform providing minimal protection.

- 4.2.2 The level of protection selected should be based primarily on the following:
- 4.2.2.1 Type(s) and measured concentration(s) of the chemical substance(s) in the ambient atmosphere and its toxicity, and
- 4.2.2.2 Potential or measured exposure to substances in air, splashes of liquids, or other direct contact with material as a result of work being performed.
- 4.2.2.3 In situations where the type(s) of chemical(s), concentration(s), and possibilities of contact are not known, the appropriate level of protection shall be selected based on professional experience and judgment until the hazards can be better characterized.
  - 4.2.3 Level A Protection—Personal Protective Equipment:
- (a) Pressure-demand, self-contained breathing apparatus, approved by the Mine Safety and Health Administration (MSHA) and National Institute of Occupational Safety and Health (NIOSH),
  - (b) Fully encapsulating chemical-resistant suit,
  - (c) Coveralls,
  - (d) Long cotton underwear,<sup>7</sup>
  - (e) Gloves (outer), chemical-resistant,
  - (f) Gloves (inner), chemical-resistant,
- (g) Boots, chemical-resistant, steel toe and shank. (Depending on suit construction, worn over or under suit boot),
  - (h) Hard hat<sup>7</sup> (under suit),
- (i) Disposable protective suit, gloves, and boots<sup>7</sup> (worn over fully encapsulating suit), and
  - (j) Two-way radio communications (intrinsically safe).
- 4.2.3.1 The fully encapsulating suit provides the highest degree of protection to skin, eyes, and respiratory system if the suit material is resistant to the chemical(s) of concern during the time the suit is worn or at the measured or anticipated concentrations, or both. While Level A provides maximum protection, the suit material may be rapidly permeated and penetrated by certain chemicals from extremely high air concentrations, splashes, or immersion of boots or gloves in concentrated liquids or sludges. These limitations should be recognized when specifying the type of chemical-resistant garment. Whenever possible, the suit material should be matched with the substance it is used to protect against.
- 4.2.3.2 Many toxic substances are difficult to detect or measure in the field. When such substances (especially those readily absorbed by or destructive to the skin) are known or suspected to be present and personnel contact is unavoidable, Level A protection should be worn until more accurate information can be obtained.
  - 4.2.4 Level B Protection—Personal Protective Equipment:
- 4.2.4.1 Pressure-demand, self-contained breathing apparatus (MSHA/NIOSH approved),
- 4.2.4.2 Chemical-resistant clothing (overalls and long-sleeved jacket; coveralls; hooded, one- or two-piece chemical-splash suit; disposable chemical-resistant coveralls),
  - 4.2.4.3 Coveralls,<sup>7</sup>
  - 4.2.4.4 Gloves (outer), chemical-resistant,
  - 4.2.4.5 Gloves (inner), chemical-resistant,
  - 4.2.4.6 Boots, chemical-resistant, steel toe and shank,

<sup>&</sup>lt;sup>4</sup> Lewis, R. J. Sr., *Sax's Dangerous Properties of Industrial Materials*, 5 Volume Set, 12<sup>th</sup> ed., John Wiley and Sons, 2012.

<sup>&</sup>lt;sup>5</sup> Lewis, R. J. Sr., *Hawley's Condensed Chemical Dictionary*, 15<sup>th</sup> ed., John Wiley and Sons, 2007.

<sup>&</sup>lt;sup>6</sup> Gijutsu, Kagai and Kenkyujo, Shiryo (Tokyo, Japan), *Toxic and Hazardous Industrial Chemicals Safety Manual for Handling and Disposal with Toxicity and Hazard Data*, Tokyo International Technical Information Institute, 1975.

<sup>&</sup>lt;sup>7</sup> Equipment is optional.

- 4.2.4.7 Boots (outer), chemical-resistant (disposable, worn over permanent boots),<sup>7</sup>
  - 4.2.4.8 Hard hat (face shield),<sup>7</sup> and
  - 4.2.4.9 Two-way radio communications (intrinsically safe).
- 4.2.4.10 Level B equipment provides a high level of protection to the respiratory tract, but a somewhat lower level of protection to skin. The chemical-resistant clothing required in Level B is available in a wide variety of styles, materials, construction detail, permeability, and so forth. These factors all affect the degree of protection afforded. Therefore, a specialist should select the most effective chemical-resistant clothing (and fully encapsulating suit) based on the known or anticipated hazards or job function, or both.
- 4.2.4.11 For initial site entry and reconnaissance at an open site, approaching whenever possible from the upwind direction, Level B protection (with good quality, hooded, chemical-resistant clothing) should protect response personnel, providing the conditions described in selecting Level A are known or judged to be absent.
  - 4.2.5 Level C Protection—Personal Protective Equipment:
- 4.2.5.1 Full-face, air purifying, canister-equipped respirator (MSHA/NIOSH approved),
- 4.2.5.2 Chemical-resistant clothing (coveralls; hooded, twopiece chemical splash suit; chemical-resistant hood and apron; disposable chemical-resistant coveralls),
  - 4.2.5.3 Coveralls,<sup>7</sup>
  - 4.2.5.4 Gloves (outer), chemical-resistant,
  - 4.2.5.5 Gloves (inner), chemical-resistant,
  - 4.2.5.6 Boots, chemical resistant, steel toe and shank,
- 4.2.5.7 Boots (outer), chemical-resistant (disposable, worn over permanent boots),<sup>7</sup>
  - 4.2.5.8 Hard hat (face shield),<sup>7</sup>
  - 4.2.5.9 Escape mask,7 and
- 4.2.5.10 Two-way radio communications (intrinsically safe).
- 4.2.5.11 Level C protection is distinguished from Level B by the equipment used to protect the respiratory system, assuming the same type of chemical-resistant clothing is used. The main selection criterion for Level C is that conditions permit wearing air-purifying devices.
- 4.2.5.12 Total unidentified vapor/gas concentrations of 5 ppm above background require Level B protection. Only a qualified individual should select Level C (air-purifying respirators) protection for continual use in an unidentified vapor/gas concentration of background to 5 ppm above background.
  - 4.2.6 Level D Protection—Personal Protective Equipment:
  - 4.2.6.1 Coveralls,
  - 4.2.6.2 Gloves,<sup>7</sup>
- 4.2.6.3 Boots/shoes, leather or chemical-resistant, steel toe and shank,
- 4.2.6.4 Boots, chemical-resistant (disposable worn over permanent boots),<sup>7</sup>
  - 4.2.6.5 Safety glasses or chemical splash goggles,<sup>7</sup>
  - 4.2.6.6 Hard hat (face shield), and
  - 4.2.6.7 Escape mask.
- 4.2.6.8 Level D protection is primarily a work uniform. It should be worn in areas where: (1) only boots can be contaminated, or (2) there are no inhalable toxic substances.

- 4.3 Personnel should not eat, drink, or smoke during or after sampling until after decontamination steps are taken. Sampling personnel should be trained in safety aspects of hazardous waste sampling.
- 4.4 Testing air emission for determining the vapor/gas concentrations can be accomplished through the use of a portable organic vapor analyzer. The probe should be held 1 to 2 in. (2.5 to 5 cm) above the sampling point. Follow manufacturer's operating instructions for proper calibration, use, and care.

## 5. Sampling Plans

- 5.1 A sampling plan is a scheme or design to locate sampling points so that suitable representative samples descriptive of the waste body can be obtained. Development of sampling plans requires the following:
- 5.1.1 Review of background information about the waste and site.
  - 5.1.2 Knowledge of the waste location and situation.
  - 5.1.3 Decisions as to the types of samples needed.
  - 5.1.4 Decisions as to the sampling design required.
- 5.2 Background data on the waste is extremely helpful in preassessment of the waste's composition, hazards, and extent. (See Notes 2 and 3.)
- Note 2—If, after researching the available background information the user cannot obtain from the material enough information about the waste to determine the probable composition and probable hazards, then the user should use other procedures. Such situations are beyond the scope of this guide.
- Note 3—The background information is needed to determine necessary safety equipment, safety procedures, sampling equipment and sampling design, and procedures to be used.
- 5.2.1 Possible sources of information on the site and waste include the following:
- 5.2.1.1 File searches of state and local records including waste manifests, waste approvals, and land permit applications.
- 5.2.1.2 File searches of generator records (if the generator can be identified) including chemical analyses, safety data sheets, design drawings, and manufacturing process information
- 5.2.1.3 File searches of treatment, storage, disposal, and transport facilities. Records involved with handling the waste.
- 5.2.1.4 Researching published data concerning the site such as scientific journal articles, EPA publications, and newspaper stories. Newspapers are the most likely source, but the information is seldom very technical.
- 5.2.1.5 Interviews of key people such as past and present employees of the site or generator, state and local officials, residents of the area, and so forth.
- 5.2.1.6 Aerial photographs provide a historical record of the site development. Many federal agencies conduct aerial surveys that are available to the public. Some of these agencies include the following:
  - (1) U.S. Department of Agriculture,
  - (2) Soil Conservation Service (USDA-SCS),
  - (3) U.S. Geological Survey,
  - (4) U.S. Forest Service,
  - (5) National Air and Space Administration (NASA),

- (6) National Oceanic and Atmospheric Administration (NOAA),
  - (7) National Weather Service,
  - (8) Corps of Engineers, and
  - (9) Agricultural Stabilization and Conservation Service.
- 5.2.1.7 Published maps can also provide a historical record of the site development such as topographic, soil, and county maps.
- 5.3 Waste location and site conditions greatly influence a sampling plan. The most common waste locations may include lagoons, landfills, pipes, point discharges, piles, drums, bins, tanks, and trucks. The site conditions include the physical condition of the waste; that is, whether it is a solid (granular, consolidated, or cohesive), liquid (slurry or flowable sludge), or gas, and it describes under what conditions it was disposed, that is, does it exist as a multiphased waste in a lagoon, tank or drum; is it stratified solids in a lagoon; is it a poorly mixed concoction of municipal garbage and hazardous sludges; or a landfill containing barrels of unknown waste.
- 5.3.1 Based on these considerations, the collector will have to decide what shall be sampled. Each situation is different and requires the best judgment of the user in writing such a plan.
- 5.4 The types of samples that may be collected are most commonly either composite or single samples. The sample collector shall decide considering the complexity of the waste location, the situation, and the financial resources, and what types of samples will best provide representative samples for reliable measurements.
- 5.4.1 A composite sample, sometimes referred to as a batch sample, is a well-mixed collection of subsamples of the same waste taken from different points. A composite sample is used most commonly in determining an average measure of a parameter. Generally, composite samples are taken when differences in the waste exist because of stratification of the simultaneous deposition of different wastes such as in a landfill.
- 5.4.2 A single sample is a well-mixed sample taken from a single point. It is used to measure a particular parameter or parameter set at a given point or within a unique homogeneous layer or throughout the strata at one or several locations.
- 5.5 Sampling plans or schemes should be carefully thought out well in advance of sampling. The most common sampling schemes involve the selection of sampling points using a judgment, a coordinate system, or a grid system.
- 5.5.1 Judgment Samples—This system is commonly used when, because of resource restraints, multiple samples cannot be collected. They are collected by deciding through visual observation or knowledge of the site where a representative sample may be collected. This type of design can be very effective if the collector is familiar and knowledgeable about the site and the goal of sampling is merely to establish whether a waste meets some set criteria.
- 5.5.2 Coordinate Sampling System—This system uses a one or two-coordinate system and involves collecting samples at random points from the origin of the coordinates. Random numbers can be generated using random number tables available in most statistic texts. The origin of the coordinate system

is normally placed at some corner of the site and marked off in steps, feet, yards, and so forth, for sampling landfills, waste piles, and lagoons. For storage areas containing barrels, the numbers of barrels from the origin are often used as intervals along the coordinate. For sampling from a flowing stream the origin may be taken as time-zero (start), and samples are collected at random time intervals over the period of interest.

5.5.3 *Grid System*—This system also involves taking samples at regular intervals, grid points, along an imaginary grid system laid out over the site. The number of sampling points will vary with the size of the grid. Such sampling schemes are used when a statistically sound sampling program is required. They should be used only when the waste body is known to be homogenous or the strata have been defined. If the waste is stratified, a separate grid system may be required for each stratum.

5.6 The proper number of samples required in a statistically sound sampling program can be estimated. This can be done using Eq 1 and by estimating the sample composition and variance either from a pilot sampling effort or knowledgeable judgment. The number of samples required, n, to achieve the desired precision in waste composition is estimated using fundamental statistical concepts, as follows (financial constraints not considered):<sup>3</sup>

$$n = (t_{0.80}^2 S^2)/d^2 \tag{1}$$

where:

n = appropriate number of samples to be collected,

 $t^2_{0.80}$  = square of the tabulated value of Student's t for a two-sided confidence interval and a coverage probability of 0.80 for the unknown mean, with the degrees of freedom defined for the  $S^2$  used to estimate the population variance,  $\sigma^2$ ,

 $S^2$  = preliminary estimate of  $\sigma^2$  obtained from previous samplings, a pilot sampling effort or other information such as the likely range of the population values,

= deviation to be exceeded only in two cases out of ten in repeated sampling for the quantity  $|\bar{X} - T|$ , the difference in absolute value between the sample average, and a threshold value such as a regulatory limit.

 $\bar{X}$  = preliminary estimate of sample average, and T = threshold value, often the regulatory limit.

- 5.6.1 The variables in Eq 1 are appropriate only for a given waste type. Therefore, the appropriate number of samples, n, required to achieve the desired precision is also applicable only to that same waste type. If two or more waste types are present in the impoundment, either as strata or other segregated wastes, then a value for n should be calculated for each waste.
- 5.6.2 Although the use of Student's t distribution is based on an underlying normal distribution for the measurements, the robustness of the t statistic for many applications may be relied upon here. If ancillary information seems to indicate that normality may not be a good assumption, then the Lilliefors<sup>8</sup> goodness of fit test should be performed to determine if the

<sup>&</sup>lt;sup>8</sup> Coover, W. J., Practical Nonparametric Statistics, 3<sup>rd</sup> Edition, John Wiley & Sons, Inc., New York, 1999, 592 pp.

assumption of a normal distribution is reasonable. This test involves examining the data from a sampling and analysis program to test the hypothesis that the data are distributed normally. If the Lilliefors test shows the contention of normality is acceptable, it does not mean that the parent population is normal. But it does mean that the Student's *t* distribution does not appear to be an unreasonable approximation to the true unknown distribution. If the Lilliefors test shows that a normal distribution does not adequately fit the data, then further pilot sampling will be required to determine the spatial distributions in the impoundment.

5.6.3 The following hypothetical example illustrates the use of Eq 1:

5.6.3.1 A preliminary study of barium levels in sludge collected from a lagoon generated values of 86, 90, 98, and 104 ppm for barium in four sludge samples. Based on these values and a knowledge of the processes producing the waste, the sludge is judged to be homogeneous (not stratified) within the lagoon. Therefore, preliminary estimates of  $\bar{X}$  and  $s^2$  are calculated as follows:

$$\bar{X} = \frac{\sum_{i=1}^{n} X_{i}}{n} = \frac{86 + 90 + 98 + 104}{4} = 94.50, \text{ and}$$

$$s^{2} = \frac{\sum_{i=1}^{n} X_{i}^{2} - \left(\sum_{i=1}^{n} X_{i}\right)^{2} / n}{n - 1} \text{ Teh Standards}$$

$$= \frac{35916.00 - 35721.00}{3} = 65.00 / \text{standards.it}$$

5.6.3.2 The deviation not to be exceeded for measured barium in the sludge samples, d, is chosen as 5.50 ppm, that is, the difference between the sample average,  $\bar{X}$  or 94.5, and the threshold limit, T or 100.0 for barium (assuming 100.0 is the regulatory threshold for barium) is 5.50 ppm.

5.6.3.3 The value of  $t_{0.80}$  is obtained from tabulated values of Student's t, as shown in Table 1. Although an assumption of a t distribution would seem to be restrictive, it can be shown that even non-normal populations possessing bell-shaped distributions can be closely approximated by a t distribution. From the preliminary study n = 4, and the degrees of freedom, n - 1, is 3. Therefore,

$$t_{0.80} = 1.638$$

The appropriate number of sludge samples to be collected from the lagoon is,

$$n = t_{0.80}^2 s^2 / d^2 = \frac{(1.638^2)(65.00)}{5.50^2} = 5.77,$$

or six. That number of samples (plus extra for protection against poor preliminary estimates of  $\bar{X}$  and  $s^2$ ) is collected from the lagoon.

## 6. Quality Assurance Considerations

- 6.1 Quality assurance for solid waste sampling should include adherence to the sampling plan and safety plan and in some cases, the use of quality control samples.
- 6.2 The sampling and safety plans should be well formulated before any actual sampling is attempted. The plans shall

TABLE 1 Tabulated Values of Student's t for Evaluating Solid Wastes

Degrees of Freedom, $(n-1)^A$	Tabulated t Value <sup>B</sup>
1	3.078
2	1.886
3	1.638
4	1.533
5	1.476
6	1.440
7	1.415
8	1.397
9	1.383
10	1.372
11	1.363
12	1.356
13	1.350
14	1.345
15	1.341
16	1.337
17	1.333
18	1.330
19	1.328
20	1.325
21	1.323
22	1.321
23	1.319
24	1.318
25	1.316
26	1.315
27	1.314
28	1.313
29	1.311
	1.310
40	1.303
60	1.296
rds.itd20.ai)	1.289
1 45.11(11.41)	1.282

<sup>A</sup> Degrees of freedom, *df*, are equal to the number of samples, *n*, collected from a solid waste less one.

<sup>B</sup> Tabulated *t* values are for a two-tailed confidence interval and a probability of 0.80 (the same values are applicable to a one-tailed confidence interval and a probability of 0.90).

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be consistent with the objectives of the sampling. The sampling plan shall include the selected points of sampling and the intended number, volumes, and types of samples to be taken. The safety plan should address the proper clothing and protective equipment, all known hazards associated with the sampling activities, and the measures to be taken to avoid these hazards.

6.3 Four types of quality control samples relate to the quality assurance of field sampling: (1) field blanks, (2) split samples, (3) field rinsates, and (4) field spikes. The selection of the types of quality control samples to be used should be made before the sampling event and included in the sampling plan. The nature of the sampling, the intended uses of the data, and the material being sampled all impact upon the selection of quality control samples to be used in an event.

## 6.3.1 Field Blanks:

6.3.1.1 Field blanks are samples prepared in the laboratory using reagent water or other blank matrix and sent with the sampling team. These samples are exposed to the sampling environment and returned with the samples to the laboratory for analysis. The purpose of the field blank is to verify that none of the analytes of interest measured in the field samples resulted from contamination of the samples during sampling.