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Standard Guide for General Planning of Waste Sampling[1]

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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 must 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.

Currently, Subcommittee D34.01 is working on practices for sampling wastes from a variety of different sampling locations and situations. Also in progress is a practice for containerization, preservation, and holding times for waste samples. As these documents are approved by ASTM, reference to these standards will be made in this general guide on waste sampling. Further, Subcommittee D34.01 recommends this guide be used in conjunction with the new waste sampling practices when available in print by ASTM.

1. Scope

1.1 This guide provides information for formulating and planning the many aspects of waste sampling (see 1.2) which are common to most waste sampling situations.

1.2 The aspects of sampling which this guide addresses are as follows:

	Section
Safety plans	4
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Preservation and containerization	8
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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 assure that the procedures used are proper and adequate.

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

2. Referenced Documents

2.1 *ASTM Standards:*

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process[2]

2.2 *Other Document:*

EPA-SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods[3]

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.

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 where safety hazards are unknown. In such cases, the user must use other more appropriate procedures.

4. Hazards

4.1 Proper safety precautions must always be observed when sampling wastes. Persons collecting samples must be

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] Available from Superintendent of Documents, U.S. Printing Office, Washington, DC 20402.

aware that the waste can be a strong sensitizer and can be 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 "Dangerous Properties of Industrial Materials" the "March Index," the "Condensed Chemical Dictionary," and the "Toxic and Hazardous Industrial Chemicals Safety Manual for Handling and Disposal with Toxicity and Hazardous 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; when vapors, gases, or airborne particulates may be generated; or when 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 due to 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 must be selected based on professional experience and judgment until the hazards can be better characterized.

4.2.3 *Level A Protection—Personnel Protective Equipment:*

(a) (*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) (*b*) Fully encapsulating chemical-resistant suit,

(c) (*c*) Coveralls,[4]

(d) (*d*) Long cotton underwear,[4]

(e) (*e*) Gloves (outer), chemical-resistant,

(f) (*f*) Gloves (inner), chemical-resistant,

(g) (*g*) Boots, chemical-resistant, steel toe and shank. (Depending on suit construction, worn over or under suit boot),

(h) (*h*) Hard hat[4] (under suit),

(i) (*i*) Disposable protective suit, gloves, and boots[4] (worn over fully encapsulating suit), and

(j) (*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—Personnel Protective Equipment*:

(a) (*a*) Pressure-demand, self-contained breathing apparatus (MSHA/NIOSH approved),

(b) (*b*) Chemical-resistant clothing (overalls and long-sleeved jacket; coveralls; hooded, one- or two-piece chemical-splash suit; disposable chemical-resistant coveralls),

(c) (*c*) Coveralls,[4]

(d) (*d*) Gloves (outer), chemical-resistant,

(e) (*e*) Gloves (inner), chemical-resistant,

(f) (*f*) Boots, chemical-resistant, steel toe and shank,

(g) (*g*) Boots (outer), chemical-resistant (disposable, worn over permanent boots),[4]

(h) (*h*) Hard hat (face shield),[4] and

(i) (*i*) Two-way radio communications (intrinsically safe).

4.2.4.1 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, etc. 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.

4.2.4.2 For initial site entry and reconnaissance at an open

[4] Equipment is optional.

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—Personnel Protective Equipment*:

(a) (*a*) Full-face, air purifying, canister-equipped respirator (MSHA/NIOSH approved),

(b) (*b*) Chemical-resistant clothing (coveralls; hooded, two-piece chemical splash suit; chemical-resistant hood and apron; disposable chemical-resistant coveralls),

(c) (*c*) Coveralls,[4]

(d) (*d*) Gloves (outer), chemical-resistant,

(e) (*e*) Gloves (inner), chemical-resistant,[4]

(f) (*f*) Boots, chemical resistant, steel toe and shank,

(g) (*g*) Boots (outer), chemical-resistant (disposable, worn over permanent boots),[4]

(h) (*h*) Hard hat (face shield),[4]

(i) (*i*) Escape mask[4], and

(j) (*g*) Two-way radio communications (intrinsically safe).

4.2.5.1 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.

4.2.5.2 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—Personnel Protective Equipment*:

(a) (*a*) Coveralls,

(b) (*b*) Gloves,[4]

(c) (*c*) Boots/shoes, leather or chemical-resistant, steel toe and shank,

(d) (*d*) Boots, chemical-resistant (disposable worn over permanent boots),[4]

(e) (*e*) Safety glasses or chemical splash goggles,[4]

(f) (*f*) Hard hat (face shield),[4] and

(g) (*g*) Escape mask.[4]

4.2.6.1 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. above the sampling point. Follow manufacturers 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. Developmentof 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, 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, etc.

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:

(a) (*a*) U. S. Department of Agriculture

(b) (*b*) Soil Conservation Service (USDA-SCS).

(c) (*c*) U. S. Geological Survey.

(d) (*d*) U. S. Forest Service.

(e) (*e*) National Air and Space Administration (NASA).

(f) (*f*) National Oceanic and Atmospheric Administration (NOAA).

(g) (*g*) National Weather Service.

(h) (*h*) Corps of Engineers.

(i) (*i*) 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 must be sampled. Each situation is different and requires the best judgement 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 must 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, or because 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 judgement, a coordinate system, or a grid system.

5.5.1 *Judgement 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 if 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, etc. 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 when 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 judgement. 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):[3]

$$n = (t^2_{0.80} S^2)/d^2 \quad (1)$$

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, σ^2;
S^2 = preliminary estimate of σ^2 obtained from previous samplings, a pilot sampling effort or other information such as the likely range of the population values;
d = 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 a goodness of fit test should be performed to determine if the assumption of a normal distribution is reasonable. The Lilliefors goodness of fit test, as it applies to the pilot sampling presented here, is described in the Appendix. This test involves examining the data from a sampling and analysis program in order 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 adequately 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:

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

$$s^2 = \frac{\sum_{i=1}^{n} X_i{}^2 - (\sum_{i=1}^{n} X_i)^2/n}{n-1}$$

$$= \frac{35\ 916.00 - 35\ 721.00}{3} = 65.00$$

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,

$$t_{0.80} = 1.638$$

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

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

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.

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

Degrees of Freedom, $(n-1)^A$	Tabulated t Value^B
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
30	1.310
40	1.303
60	1.296
120	1.289
	1.282

^{A} Degrees of freedom, *df*, are equal to the number of samples, *n*, collected from a solid waste less one.

^{B} 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).

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 must be consistent with the objectives of the sampling. The sampling plan must 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 prior to 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.

6.3.1.2 The sampling plan should normally include a minimum of one field blank for each procedure for each sampling event. These samples can be submitted blind to the laboratory to challenge their analytical system or can be shipped with the instruction to hold them unless there is a reason to suspect sample contamination. The submission of blind field blanks would normally be reserved for those situations where the competency of the analytical laboratory was unproven (that is, where a new laboratory was being utilized).

6.3.2 *Split Samples*:

6.3.2.1 Split samples are used to challenge the analytical laboratory performance. Split samples are also used when two different parties are sampling the same site and verification of analytical results is necessary. A split sample is prepared by subsampling a homogenous sample into two or more portions and submitting each portion separately to the analytical laboratory.

6.3.2.2 For liquid matrixes, the material should be placed in a large, clean container and stirred or swirled to ensure thorough mixing of the medium prior to subsampling. For solid media, a sufficient quantity must be removed, mixed with clean utensils, and subsampled. Sufficient mixing of the sample

should be accomplished to ensure that stratification of analytes is avoided.

NOTE 4—**Caution:** If volatile organics are a concern, homogenizing in open containers will likely result in losses of volatiles.

6.3.2.3 Split samples are treated as separate study samples, carried through the entire sample handling procedures, and submitted to the analytical laboratory without distinguishing identification. Split samples are an indication of the precision of the analytical procedures. For comments on sampling precision, see 5.6; the definition of acceptable levels of precision is the responsibility of the user.

6.3.2.4 Where feasible, each sampling event should include a minimum of one split sample for each type of media or location sampled. Where the data are intended for demonstration of data quality to an outside agency, replicates should be included at a greater frequency, up to 10 % of the total number of samples collected.

6.3.3 *Field Rinsates*—Field rinsates are samples collected in the field by filling a sample collection vessel, such as a well bailer with reagent water or other blank matrix, and then transferring this water to the proper sample bottles. It may be necessary in some instance to fill the collection vessel a number of times to ensure enough water is collected for analysis. The purpose of a field rinsate is to ensure that sampling equipment cleaned in the field is not cross contaminating samples through improper cleaning techniques. These types of samples should be taken at least once for each procedure for each sampling event when field cleaning is performed. If only one such sample is taken it should be collected just prior to the last sample.

6.3.4 *Field Spikes*:

6.3.4.1 Field spikes are samples collected in the field and spiked with compounds of interest or related compounds. These samples are used to check on the potential for loss of analyte on shipping and for recovery of analytes from a particular medium. The field spike is prepared by adding a known amount of the spiking material to a known amount of the matrix and mixing thoroughly. Where a liquid medium is to be collected, the spiking material may be added to the collection container at the laboratory and the sample medium added to the container. For a solid matrix, the material should be added in the field and thoroughly mixed through the matrix prior to closing and sealing the container.

6.3.4.2 Field spikes are normally not required. Instances when a field spike may be desired include where preservation techniques are in question and the integrity of the analytes at the laboratory is not known, when there is a question concerning matrix effects, and when the results from the analytical laboratory for a particular analyte or class of analytes is in question.

6.3.4.3 Field spikes should be submitted blind to the laboratory in the same manner as outlined for the split samples. These samples should be carried through all stages of the sampling and sample handling process as the actual study samples to ensure that they truly indicate the integrity of the samples collected.

7. General Sampling Consideration

7.1 Sampling equipment must be selected that is chemically compatible with the type of waste and type of analyses. Generally, plastic sampling equipment is not suitable for waste containing or to be analyzed for organic parameters. Stainless steel, glass, and plastic are generally acceptable for most samples to be analyzed for inorganics. It is up to the user to ensure that the equipment will not contaminate or bias the analyses.

7.2 The sampling equipment must be capable of extracting a sample from the desired location, depth, or point and at the same time provide protection from cross-contamination during sampling. For instance, one very common problem is extracting a sludge sample from beneath a top layer of wastewater or sludge without contaminating the sample with the overlying wastewater or sludge. This situation, as well as many others, requires special equipment. The collector is therefore faced in many instances with having to fabricate the needed equipment.

7.3 Recommended sampling procedures are for collection of samples from the edge of ponds or lagoons or from piers or catwalks. Sampling from boats is not recommended and should be attempted only if the collector knows the waste poses no real health problem and every possible safety measure has been taken.

7.4 Tanks and drums containing unknown substances also pose potential health risks for the collector due to the possibility of fire, explosion, or the release of deadly gases upon opening. Therefore, it is recommended that in these situations only spark-proof, remote opening devices be used and only fully trained and experienced personnel attempt to do this.

7.5 Representative samples are intended to reflect the true makeup of the population. Composite sampling is one way to help achieve representativeness in a cost- and resource-efficient way. Frequently overlooked but important problems with composite sampling of waste materials include the following:

7.5.1 Loss of volatile components during the mixing process,

7.5.2 Reactivity of dissimilar materials combined into a single composite,

7.5.3 Collecting the correct number and size of aliquots to form the composite, and

7.5.4 Properly homogenizing and subsampling to reduce the amount of material sent to the laboratory.

7.6 The laboratory should provide guidance to the field sampling team to avoid losses due to volatilization or reactivity. Guidelines based on geostatistical principles for forming the composite from individual aliquots should be provided to sampling personnel. Sample homogenization and subsampling in the field should only be attempted by qualified personnel using appropriate equipment. If these are not available, the individual aliquots should be sent to the laboratory for processing. All composite samples or aliquots, or both, intended for forming a composite should be clearly marked so that the laboratory can mix and subsample appropriately.

7.6.1 If low chemical concentrations of a parameter to be tested are expected, take a large volume of sample.

7.6.2 If high chemical concentrations of a parameter to be tested are expected, smaller volumes will suffice.

7.7 When possible, it is recommended that sampling proceed from the least contaminated to the most contaminated areas to reduce problems of cross contamination.

8. Preservation and Containerization of Samples

8.1 Water sample preservation techniques cannot be used for waste samples but are sometimes confused with them.

NOTE 5—Subcommittee D34.01 on Sampling and Monitoring is currently balloting a draft practice on preservation of waste and when approved by the ASTM membership, it will be referenced here. Container specifications for waste samples are found in the same draft practice mentioned above and will be similarly referenced.

9. Cleaning Equipment

9.1 All sampling equipment must be cleaned before use and if possible, it is preferable to have them lab cleaned. Improper cleaning causes cross-contamination of samples. Use of disposable samplers is a very simple way of eliminating the cleaning problem. When samplers are lab cleaned, it is equally important to protect the samples from contamination by wrapping, packaging or containerizing in clean, noncontaminating material.

9.2 Wash equipment cleaned in the lab with a warm detergent solution, rinse several times with tap water, rinse with deionized water and air dry. If organic analyses are to be run, wash with a warm detergent solution (it may first be necessary to wipe with an absorbent cloth to eliminate residues), rinse several times with tap water, rinse with deionized water, rinse with an appropriate organic solvent (the solvent should be, if possible, the extracting solvent) and oven dry at 105°C for at least an hour.

9.3 Cleaning procedures in the field are the same except that tap water rinses, solvent rinses, and drying may not be practical. The user must be careful in using a solvent rinse without oven drying as it may permeate other samples if shipped in the same container or it may react with constituents in the waste. It should be kept in mind that field cleaning requires the capability to carry a large amount of water.

10. Packaging and Package Marking

10.1 An indelible label should be secured to the container identifying the sample. The label should contain or reference the following information:

10.1.1 Name and location of site,

10.1.2 Date and time of sampling,

10.1.3 Location of sampling,

10.1.4 Sample number,

10.1.5 Description and disposition of sample,

10.1.6 Name of sampling personnel,

10.1.7 If possible, full weight of sample and container upon shipping,

10.1.8 Type of preservative, and

10.1.9 Analytical requirements.

10.2 Pack the sample container securely in a shipping container. Generally, if the sample is to be analyzed for volatile organics, it should be packed in ice and cooled to 4°C (See Section 8). A minimum-maximum thermometer packed with the sample container is valuable where knowledge of temperature extremes is critical.

10.3 For shipping purposes, samples must be classified as environmental or hazardous material (waste) samples. In general, environmental samples are collected offsite (for example, from streams, ponds, or wells) and are not expected to be grossly contaminated with high levels of hazardous materials. Waste samples (for example, material from drums or bulk storage tanks; obviously contaminated lagoons, ponds, soils; and leachates from hazardous waste sites) are considered hazardous. Hazardous materials are subject to shipping regulation (for example, International Air Transportation Association (IATA) or U.S. Department of Transportation (USDOT)) and should be packaged and shipped by a person trained in those requirements. If the substance in the sample is known or can be identified, package, mark, and ship according to the specific instructions for that material. Further detail on the shipment of hazardous materials is beyond the scope of this guide, and professional assistance should be sought.

10.4 Make arrangements for handling, logging in, adequate storage, and analysis of the sample at its destination.

10.5 All information pertinent to a field survey or sampling activity must be recorded in a logbook. This should be bound, preferably with consecutively numbered waterproof pages. Where the same information is routinely collected, preprinted logbooks are suggested. Information should be recorded in indelible ink, all entry errors should be crossed out with a single line and initialed, and all entries should be dated and signed at least daily. Entries in the logbook should include information such as the folowing:

10.5.1 Identification of sampling plan (by reference),

10.5.2 Location of sampling,

10.5.3 Name and address of field contact,

10.5.4 Producer of waste and address,

10.5.5 Type of process producing waste (if known),

10.5.6 Type of waste (for example, sludge, wastewater),

10.5.7 Suspected waste composition, including concentrations,

10.5.8 Number and volume of samples taken,

10.5.9 Purpose of sampling (for example, surveillance, contract number),

10.5.10 Description of sampling point and sampling methods,

10.5.11 Date and time of collection,

10.5.12 Preservation used, if any (include ice),

10.5.13 Analytical parameter to be measured,

10.5.14 Unique sample identification number(s),

10.5.15 Sample destination, how transported, and name of transporter,

10.5.16 References, such as maps or photographs of the sampling site,

10.5.17 Field observations (for example, ambient temperature, wind conditions, or other site specific conditions),

10.5.18 Field measurements (for example, pH, explosivity) and results, and

10.5.19 Names of personnel on sampling team.

10.6 Record sufficient information so that anyone could reconstruct the sampling without reliance on the collector's memory. The contents of the logbook should be specified in the sampling plan, a standard practice or similar document. The

logbook must be protected and kept in a safe place. Additional detail may be needed than listed above depending on the technical objectives of the sampling project.

11. Chain-of-Custody Procedure

11.1 A chain-of-custody procedure should be developed for samples that may be used in legal proceedings. It is recommended that legal counsel be consulted to assist in developing an individual chain-of-custody record. Chain-of-custody procedures are used to ensure sample integrity and to ensure the sample will provide legally and technically defensible data.

11.2 Samples should be collected in accordance with sampling procedures designated in the sampling plan if the sample is to maintain its legal integrity.

11.3 A legal seal should be attached to the immediate sample container in a manner that the sample cannot be opened without breaking the seal. The seal should have a unique number written across it or the signature of the sampler. Be sure to record the unique legal seal number in the log book.

11.4 The sample should be kept in view or within limited access or locked storage until custody is relinquished and formal documentation of the transfer is completed. The collector should initiate documents at the source of the sample and start the chain-of-custody procedure. Documentation should include the following:

11.4.1 Sample number and legal seal numbers,

11.4.2 Site name and location,

11.4.3 Date and time sampled,

11.4.4 Date sent to laboratory,

11.4.5 Name of sampler,

11.4.6 Information describing source of sample and sample,

11.4.7 Sampling method,

11.4.8 Preservation techniques,

11.4.9 Condition of legal seal upon delivery and name of receiving person,

11.4.10 Method of shipment,

11.4.11 Dates of all activities, and

11.4.12 Signature of persons delivering and receiving samples.

APPENDIX

(Nonmandatory Information)

X1. THE LILLIEFORS TEST FOR GOODNESS OF FIT

X1.1 The data consist of a random sample $X_1, X_2, \ldots, X_n$ of size, n, associated with some unknown distribution function, denoted by $F(x)$. Compute the sample mean:

$$\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_i \qquad \text{(X1.1)}$$

for use as an estimate of μ, and compute:

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n} (X_i - \bar{X})^2} \qquad \text{(X1.2)}$$

as an estimate of σ. Then compute the "normalized" sample values Z_i, defined as follows:

$$Z_i = \frac{X_i - \bar{X}}{s} \; i = 1, 2...., n \qquad \text{(X1.3)}$$

The test statistic is computed from the Z_i's instead of from the original random sample.

X1.2 *Assumptions*—The sample is a random sample.

X1.3 *Hypotheses:*

X1.3.1 H_0—The random sample has the normal distribution, with unspecified mean and variance.

X1.3.2 H_1—The distribution function of the X_i's is non-normal.

X1.4 *Test Statistic*—Ordinarily the test statistic is the usual two-sided Kolmogorov test statistic, defined as the maximum vertical distance between the empirical distribution function of the X_i's and the normal distribution function with mean $\bar{X}$ and standard deviation s, as given by Eq X1.1 and X1.2. However, the following method of computing the test statistic is slightly easier, and is equivalent to the method indicated in X1.1.

X1.4.1 Draw a graph of the standard normal distribution function, and call it $F'(x)$. Table X1.1 may be of assistance. Also draw a graph of the empirical distribution function of the normalized sample, the Z_i's defined by Eq X1.3, using the same set of coordinates as was used above for $F'(x)$. Find the maximum vertical distance between the two graphs, $F'(x)$ and the empirical distribution function which we shall call $S(x)$. This distance is the test statistic. That is, the Lilliefors test statistic T_2 is defined by the following equation:

$$T_2 = \sup | F'(x) - S(x) | \qquad \text{(X1.4)}$$

X1.5 *Decision Rule*—Reject H_o at the approximate level of significance of α if T_2 exceeds the $1 - \alpha$ quantile as given in Table X1.2.

X1.5.1 The same data used to calculate n, the number of samples required, in Section 5 will be used to illustrate the Lilliefors test.

X1.5.2 Barium levels in four sludge samples were 86, 90, 98, and 104 ppm. The sample values, X_i, are arranged from smallest to largest, and converted to Z_i by subtracting $\bar{X} = 94.50$, and dividing by $S = \sqrt{65.00} = 8.06$.

X_i	Z_i
86	−1.05
90	−0.56
98	0.43
104	1.18

X1.5.2.1 The null hypothesis of normality is tested with the Lilliefors test statistic

TABLE X1.1 Normal Distribution[A]

NOTE 1—This table was abridged from Tables 3 and 4 by Pearson, E. S., and Hartley, H. E., "Biometric Tables for Statisticians," Cambridge University Press, Cambridge, England, pp. 111–112, 1962.

w_p	P	w_p	P	w_p	P
−3.7190	0.0001	−0.4677	0.32	0.5244	0.70
−3.2905	0.0005	−0.4399	0.33	0.5534	0.71
−3.0902	0.001	−0.4125	0.34	0.5828	0.72
−2.5758	0.005	−0.3853	0.35	0.6128	0.73
−2.3263	0.01	−0.3585	0.36	0.6433	0.74
−2.1701	0.015	−0.3319	0.37	0.6745	0.75
−2.0537	0.02	−0.3055	0.38	0.7063	0.76
−1.9600	0.025	−0.2793	0.39	0.7388	0.77
−1.8808	0.03	−0.2533	0.40	0.7722	0.78
−1.7507	0.04	−0.2275	0.41	0.8064	0.79
−1.6449	0.05	−0.2019	0.42	0.8416	0.80
−1.5548	0.06	−0.1764	0.43	0.8779	0.81
−1.4758	0.07	−0.1510	0.44	0.9154	0.82
−1.4395	0.075	−0.1257	0.45	0.9542	0.83
−1.4051	0.08	−0.1004	0.46	0.9945	0.84
−1.3408	0.09	−0.0753	0.47	1.0364	0.85
−1.2816	0.10	−0.0502	0.48	1.0803	0.86
−1.2265	0.11	−0.0251	0.49	1.1264	0.87
−1.1750	0.12	0.0000	0.50	1.1750	0.88
−1.1264	0.13	0.0251	0.51	1.2265	0.89
−1.0803	0.14	0.0502	0.52	1.2816	0.90
−1.0364	0.15	0.0753	0.53	1.3408	0.91
−0.9945	0.16	0.1004	0.54	1.4051	0.92
−0.9542	0.17	0.1257	0.55	1.4395	0.925
−0.9154	0.18	0.1510	0.56	1.4758	0.93
−0.8779	0.19	0.1764	0.57	1.5548	0.94
−0.8416	0.20	0.2019	0.58	1.6449	0.95
−0.8064	0.21	0.2275	0.59	1.7507	0.96
−0.7722	0.22	0.2533	0.60	1.8808	0.97
−0.7388	0.23	0.2793	0.61	1.9600	0.975
−0.7063	0.24	0.3055	0.62	2.0537	0.98
−0.6745	0.25	0.3319	0.63	2.1701	0.985
−0.6433	0.26	0.3585	0.64	2.3263	0.99
−0.6128	0.27	0.3853	0.65	2.5758	0.995
−0.5828	0.28	0.4125	0.66	3.0902	0.999
−0.5534	0.29	0.4399	0.67	3.2905	0.9995
−0.5244	0.30	0.4677	0.68	3.7190	0.9999
−0.4959	0.31	0.4959	0.69		

[A] The entries in this table are quantiles w_p of the standard normal random variable W, selected so $P(W<w_p) = p$ and $P(W>w_p) = 1 - p$.

$$T_2 = \sup | F'(x) - S(x) | \quad \text{(X1.5)}$$

where $F'(x)$ is the standard normal distribution function and $S(x)$ is the empirical distribution function of the Z_i's. Fig. X1.1 shows the curves representing $F'(x)$ (generated from Table X1.1) and $S(x)$. The maximum vertical distance between $F'(x)$ and $S(x)$ is seen from Fig. X1.1 to occur at $x = -1.05$, where $S(x)$ equals 0 and $F'(x)$ equals 0.15, and so T_2 equals 0.15.

X1.5.2.2 The Lilliefors test calls for rejection of H_o at $\alpha = 0.05$ if T_2 exceeds its 0.95 quantile, which is given in Table X1.2 as follows:

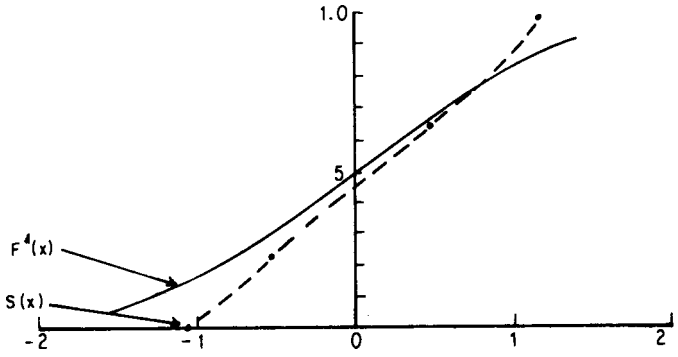
$$W_{0.95} = \frac{0.381}{\sqrt{n}} = \frac{0.381}{\sqrt{4}} = 0.19$$

TABLE X1.2 Quantiles of the Lilliefors Test Statistic[A]

NOTE 1—This table was adapted from Table 1 of Lilliefors, H. W., "On the Kolmogorov-Smirnov Test for Normality with Mean and Variance Unknown," *Journal of American Statistical Association*, Vol 62, pp. 399–402, 1967.

	$p = 0.80$	0.85	0.90	0.95	0.99
Sample size $n = 4$	0.300	0.319	0.352	0.381	0.417
5	0.285	0.299	0.315	0.337	0.405
6	0.265	0.277	0.294	0.319	0.364
7	0.247	0.258	0.276	0.300	0.348
8	0.233	0.244	0.261	0.285	0.331
9	0.223	0.233	0.249	0.271	0.311
10	0.215	0.224	0.239	0.258	0.294
11	0.206	0.217	0.230	0.249	0.284
12	0.199	0.212	0.223	0.242	0.275
13	0.190	0.202	0.214	0.234	0.268
14	0.183	0.194	0.207	0.227	0.261
15	0.177	0.187	0.201	0.220	0.257
16	0.173	0.182	0.195	0.213	0.250
17	0.169	0.177	0.189	0.206	0.245
18	0.166	0.173	0.184	0.200	0.239
19	0.163	0.169	0.179	0.195	0.235
20	0.160	0.166	0.174	0.190	0.231
25	0.142	0.147	0.158	0.173	0.200
30	0.131	0.136	0.144	0.161	0.187
Over 30	0.736	0.768	0.805	0.886	1.031
	$\sqrt{n}$	$\sqrt{n}$	$\sqrt{n}$	$\sqrt{n}$	$\sqrt{n}$

[A] The entries in this table are the approximate quantiles w_p of the Lilliefors test statistic T_2. Reject H_O at the level α if T_2 exceeds $w_{1-\alpha}$ for the particular sample size n.



NOTE 1—Because T_2 equals 0.15, and is less than 0.19, the null hypothesis is accepted. This means that the normal distribution is a reasonable approximation of the true unknown distribution.

FIG. X1.1 Graphs of *F*′(*x*) and *S*(*x*) from Example