Designation: D 5233 – 92 (Reapproved 2004)

Standard Test Method for Single Batch Extraction Method for Wastes[1]

1. Scope

1.1 This test method is applicable to the extraction of samples of treated or untreated solid wastes or sludges, or solidified waste samples, to provide an indication of the leaching potential.

1.2 This test method is intended to provide an extract for measurement of the concentration of the analytes of concern. The measured values may be compared against set or chosen acceptance levels in some applications.

1.3 If the sole application of the test method is such a pass/fail comparison and a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that the chosen acceptance concentration levels could not possibly be exceeded, the test method need not be run.

1.4 If the sole application of the test method is such a pass/fail comparison and an analysis of any one of the liquid fractions of the extract indicates that the concentration of the target analyte is so high that, even after accounting for dilution from the other fractions of the extract, it would be equal to or above an acceptance concentration level, then the waste fails the test. In such a case it may not be necessary to analyze the remaining fractions of the extract.

1.5 This test method is intended to provide an extract suitable for the measurement of the concentration of analytes that will not volatilize under the conditions of the test method.

1.6 Presence of volatile analytes may be established if an analysis of the extract obtained using this test method detects the target volatile analyte. If its concentration is equal to or exceeds an acceptance level for that analyte, the waste fails the test. However, extract from this test method shall not be used to determine the concentration of volatile organic analytes.

1.7 This test method is intended to describe only the procedure for performing a batch extraction. It does not describe all of the sampling and analytical requirements that may be associated with the application of this test method.

1.8 The values stated in either SI or inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.9 *This standard does not purport to address all of the safety problems, 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 a specific precautionary statement, see Note 8.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 75 Practices for Sampling Aggregates
D 420 Practice for Investigating and Sampling Soil and Rock for Engineering Purposes
D 653 Terminology Relating to Soil, Rock, and Contained Fluids
D 1129 Terminology Relating to Water
D 1193 Specification for Reagent Water
D 2234 Test Method for Collection of a Gross Sample of Coal
D 3370 Practices for Sampling Water
E 122 Practice for Choice of Sample Size to Estimate A Measure of Quality for a Lot or Process
ES 16 Practice for the Generation of Environmental Data Related to Waste Management Activities[3]

3. Terminology

3.1 Definitions—For definitions of terms used but not defined in this test method, see Terminology D 1129.

4. Summary of Test Method (See Fig. 1)

4.1 For wastes containing less than 0.5 % dry solid material, the filtrate of the waste, after filtration through a 0.6 to 0.8-μm glass fiber filter, is defined as the method extract. Extraction of the solid is not required for such wastes.[4,5]

4.2 For wastes containing greater than or equal to 0.5 % dry solid material, the liquid, if any, is separated from the solid

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[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Disposal and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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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] *Annual Book of ASTM Standards*, Vol 11.04 (see 1991 edition).

[4] *Federal Register*, Vol 55, No. 61, March 29, 1990. Toxicity Characteristics Revisions, Final Rule.

[5] *Federal Register*, Vol 55, No. 126, June 29, 1990. Toxicity Characteristic Revisions, Final Rule, Corrections.

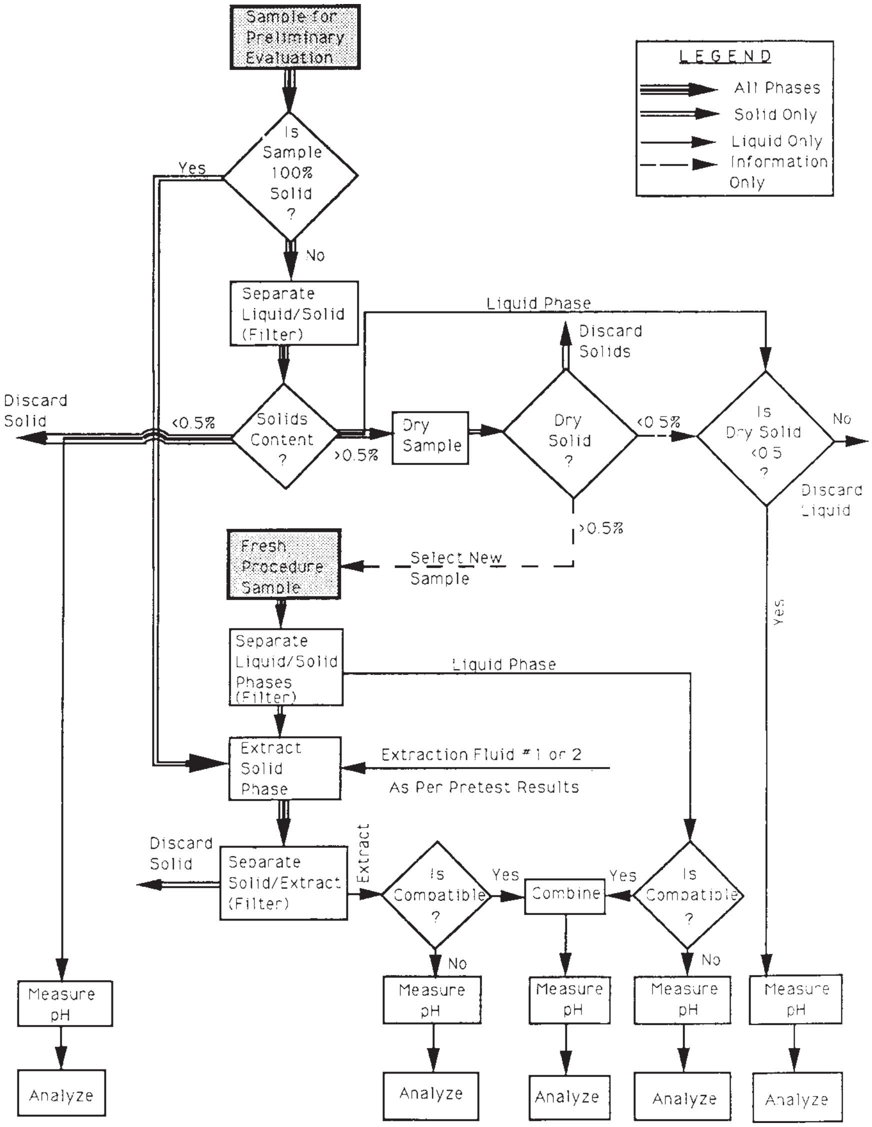

FIG. 1 Method Flow Chart

phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid used is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8-µm glass fiber filter.

4.3 If compatible (that is, multiple phases will not form upon combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are combined mathematically to yield a volume-weighted average concentration.

5. Significance and Use

5.1 This test method is intended to generate an extract with a concentration of the target analyte(s) representative of the expected release under the scenario simulated, and which can be compared with concentration levels acceptable in waste disposal, treatment, or production activities.

5.2 The extraction conditions of the test method were chosen to simulate a potential disposal scenario to which the wastes may be exposed.

5.3 One intent of this test method is that the amount of acid in the extraction fluids reflect the acid available from the leachate of a specific landfill where municipal and industrial wastes were co-disposed.[6]

5.4 One intent of this test method is to not allow the pH of the extraction fluid to be lower than that of the leachate of a specific landfill where municipal and industrial wastes were co-disposed. Therefore, the pH of the extraction fluid was chosen with the following considerations:

(1) Not to be less than 4.93 ± 0.05 for the extraction of wastes with an acid neutralization capacity of less than the acid

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[6] Kimmel, T. A., and Friedman, D. A., "Model Assumptions and Rationale Behind the Development of EP III," *ASTM STP 886*, J. K. Petros, et al, Eds., ASTM, Philadelphia, PA, 1986, pp. 36–53.

available in the total volume of extraction fluid used in the method (Extraction Fluid No. 1).

(2) At 2.88 ± 0.05, as defined by the pH of the acid, for the extraction of wastes with an acid neutralization capacity of more than the acid available in the extraction fluid used in the method (Extraction Fluid No. 2).

5.5 The interpretation and use of the results of this test method are limited by the assumptions of a single co-disposal scenario and by the factors affecting the composition of a landfill leachate and chemical or other differences between a selected extraction fluid and the real landfill leachate.

5.6 This test method may be affected by biological changes in the waste, and it is not designed to isolate or measure the effect of such processes.

5.7 This test method produces extracts that are amenable to the determination of both minor and major constituents. When minor constituents are being determined, it is especially important that precautions be taken in sample storage and handling to avoid possible contamination of the samples.

5.8 The agitation technique, rate, liquid-to-solid ratio, and filtration conditions specified in the method may not be suitable for extracting all types of wastes.

5.9 This test method is intended to extract the samples in their original physical state as is, without any size reduction. However, the sample/extractor interaction is expected to correlate with the environmental conditions to which a waste may be exposed.[7]

5.10 The extraction conditions defined by this test method are expected to yield steady-state concentrations, determined by the extraction liquid-to-solid ratio and the duration of the extraction, which may or may not agree with the concentration of an equilibrium.

6. Apparatus and Materials

6.1 *Agitation Apparatus*, capable of rotating the extraction vessel in an end-over-end fashion (see Fig. 2), at 30 ± 2 r/min, such that the axis of rotation is horizontal and passes through the center of the bottle.

NOTE 1—Similar devices may be used having a different axle arrangement if equivalency can be demonstrated.

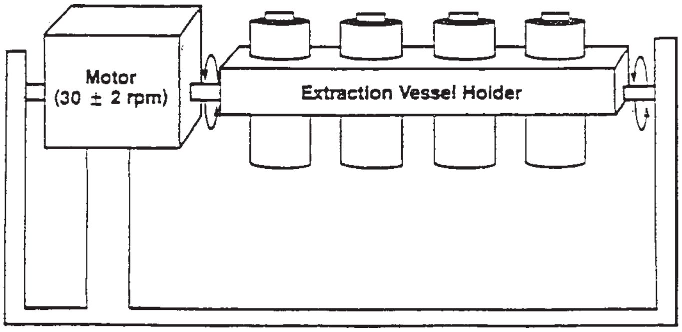

FIG. 2 Rotary Agitation Apparatus

6.2 *Extraction Vessel*—Suitable vessels include cylindrically shaped, minimum 2-L size, with capacity sufficient to hold the sample and the extraction fluid. Head-space is allowed in this vessel. The extraction bottles may be constructed from various plastic materials, depending on the analytes of interest and the nature of the waste. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. The bottles should be sturdy, in order to withstand the impact of the falling sample fragments, and shall have a leak-free seal. The use of polytetrafluoroethylene tape is recommended to ensure a tight seal. Due to their potential for breakage, the use of glass bottles is not recommended.

NOTE 2—Suitable bottles range from 4.0 to 4.5 in. (102 to 114 mm) in diameter and from 8.5 to 13.0 in. (216 to 330 mm) in height.

6.3 *Filtration Device*—It is recommended that all filtrations be performed in a hood. Wastes should be filtered using positive-pressure filtration using a pre-purified grade inert gas such as nitrogen.

6.3.1 *Filter Holder*, capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation (maximum 50 psi or 345 kPa). These devices shall have a minimum internal volume of 300 mL and shall be equipped to accommodate a minimum filter size of 47 mm. (Filter holders having an internal capacity of 2.2 L and equipped to accommodate a 142-mm diameter filter are recommended.)

6.3.1.1 *Materials of Construction*—Filtration devices shall be made of inert materials that will not leach or adsorb the analytes of concern. Glass, polytetrafluoroethylene, or type 316 stainless steel equipment may be used when both organic and inorganic analytes are of concern. Devices made of high-density polyethylene (HDPE), polypropylene (PP), or polyvinylchloride (PVC) may be used when only inorganics are of concern.

6.4 *Filters*, made of borosilicate glass fiber, containing no binder materials, and having an effective pore size of 0.6 to 0.8μ m. Pre-filters must not be used. When inorganic analytes are of concern, the filter shall be acid washed prior to use by rinsing with 1 N nitric acid followed by three consecutive rinses with Type II reagent water as defined in Specification D 1193. (A minimum of 1 L per rinse is recommended.) Glass fiber filters are fragile and should be handled with care.

6.5 *pH Meter*, with a readability of 0.01 unit and an accuracy of ±0.05 unit at 25°C.

6.6 *Laboratory Balance*, accurate to within ±0.01 g. (All weight measurements are to be within± 0.1 g.)

6.7 *Beakers or Erlenmeyer Flasks*, glass 500-mL, and 2-L.

6.8 *Watch-Glass*, with an appropriate diameter to cover the beaker or Erlenmeyer flask.

6.9 *Magnetic Stirrer*.

6.10 *Mold*, cylindrical, made of inert, non-adsorbing and non-contaminating material for casting of laboratory samples.

6.11 *Straightedge*, made of stainless steel.

6.12 *Impermeable Sheet or Glazed Paper*.

6.13 *Volumetric Flask*, 1-L size.

6.14 *Drying Oven*—Any thermostatically controlled drying oven capable of maintaining a temperature between 85 and 115°C within ±5°C.

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[7] *Federal Register*, Vol 53, No. 100, May 24, 1988. Proposed Cage Modification of TCLP.

6.15 *Graduated Pipet*, readable to 0.1 mL.

6.16 *Hot Plate*, equipped for agitation and temperature control capable of maintaining a 50 ± 3°C temperature.

6.17 *Graduated Measuring Cylinder*, with a precision of ±3 %.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Reagent water is defined as water in which an interfering analyte is not observed at or above the method detection limit of the analyte(s) of interest. Type II of Specification D 1193 or equivalent meets the definition of reagent water.

7.3 *Hydrochloric Acid* (HCl), 1 N, made from ACS reagent grade.

7.4 *Nitric Acid* (HNO_3), 1 N, made from ACS reagent grade.

7.5 *Sodium Hydroxide* (NaOH), 1 N, made from ACS reagent grade.

7.6 *Glacial Acetic Acid* (CH_3COOH), ACS reagent grade.

7.7 *Extraction Fluids*—Several batches or multiple volumes of extraction fluids should be prepared in accordance with the number of extractions. The volume needed for an individual extraction is approximately 2 L. The extraction fluids should be monitored frequently for impurities. The pH should be examined prior to extraction to ensure that these fluids were made up accurately. If impurities are found or the pH is not within the specifications, the fluid shall be discarded and fresh extraction fluid prepared.

7.7.1 *Extraction Fluid* No. 1—Add 5.7 mL glacial acetic acid to 500 mL of reagent water, add 64.3 mL of 1 N NaOH, and dilute to a volume of 1 L. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

7.7.2 *Extraction Fluid* No. 2—Dilute 5.7 mL glacial acetic acid with reagent water to a volume of 1 L. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

8. Sampling

8.1 If representative samples of the waste must be tested, use ASTM Sampling methods developed for the specific industry where available (see Practices D 75, D 420, D 3370, Terminology D 653, and Method D 2234).

8.2 All samples shall be collected using an appropriate sampling plan to ensure sample integrity and representativeness (see Practice E 122).

8.3 Where no specific methods are available, sampling methodology for materials of similar physical form shall be used.

8.4 It is important that the sample of the waste be representative with respect to surface area, as variations in surface area would directly affect the extraction characteristics of the sample. Waste samples should contain a representative distribution of particle sizes.

NOTE 3—Information on obtaining representative samples can also be found in *Pierre Gy's Sampling Theory and Practice*.[9]

8.5 Approximately 100 g of solid phase samples are required for each extraction. Preliminary evaluation also requires 100 g of solid phase sample. A larger sample size may be required, depending on the solids content of the waste sample (percent solids; see 10.2.9).

8.6 Enough extract should be generated such that the volume will be sufficient to support all of the analyses required. If the volume of extract generated by the performance of a single extraction will not be sufficient to perform all of the analyses to be conducted, it is recommended that more than one extraction be performed and that the extracts from each extraction be combined and then aliquoted for analysis.

8.7 For the evaluation of solidified or stabilized wastes, or both, samples may be cast in the form of a cylinder that will fit into the extraction apparatus. Such cylinders may be used for the evaluation. The casting may be allowed to cure for 30 days before the extraction procedure is performed. For other monolithic materials, a coring may be produced that will fit into the extraction apparatus. Waste materials to which these casting and coring procedures apply include concrete materials, rock, wood, slag, and so forth.

8.8 Quality control measures may require additional samples.

9. Sample Handling and Preparation

9.1 For free-flowing particulate solid wastes, obtain a sample in accordance with the requirements of Section 8 by quartering the sample received for testing on an impermeable sheet of glazed paper, or other flexible non-contaminating material, as follows:

9.1.1 Empty the sample container into the center of the sheet.

9.1.2 Flatten out the sample gently with a suitable straightedge until it is spread uniformly to a depth at least twice the maximum particle diameter.

9.1.3 Remix the sample by lifting a corner of the sheet and drawing low across to the opposite corner in the manner that the material is made to roll over and over and does not merely slide along. Return that corner to its original position. Continue the operation with each corner, proceeding in a clockwise direction. Repeat this cycle ten times.

9.1.4 Lift all four corners of the sheet toward the center, and holding all four corners together, raise the entire sheet into the air to form a pocket for the sample.

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[8] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

[9] Pitard, F., *Pierre Gy's Sampling Theory and Practice*, Volumes I and II, CRC Press, 1989.

9.1.5 Repeat 9.1.2.

9.1.6 Gently divide the sample into quarters with a straightedge, one at least as long as the flattened mound of sample. Make an effort to avoid using pressure on the straightedge sufficient to cause damage to the particles.

9.1.7 Discard alternate quarters.

9.1.8 If further reduction of the sample volume is necessary, repeat 9.1.3 through 9.1.7. Use a sample volume to give 100 g of solid for each extraction. Provide additional samples for the preliminary evaluation.

9.2 For field-cored solid wastes or castings produced in the laboratory, cut a representative section weighing approximately 100 g for each extraction plus the preliminary evaluation. Take samples for the preliminary evaluation at the same time as the test samples.

9.2.1 If necessary, shape the sample so that its largest dimension would not exceed the radius of the extraction bottle. (The material shall move freely while fully covered with the extraction fluid.)

9.3 Preservatives shall not be added to the samples prior to extraction.

9.4 For multi-phasic wastes, mix thoroughly to ensure that a representative sample will be withdrawn.

9.5 Samples shall be stored at 4°C to minimize changes due to biological processes. If precipitation occurs, the entire sample (including precipitate) of the precipitate existing at room temperature (see 4.1 and 4.2) should be used.

9.6 The method filtrates and extracts should be prepared for analysis and analyzed as soon as possible. Filtrates and extracts or their portions for metallic analyte determinations should be acidified with nitric acid to pH <2 unless precipitation occurs. To minimize losses, filtrates or extracts or their portions for organic contaminant determinations shall not be allowed to make contact with the atmosphere (that is, head-space).

9.7 Sample maximum holding times (days) are given in Table 1.

10. Preliminary Evaluations and Pre-Extraction Procedures

10.1 Perform preliminary method evaluations on a minimum 100-g aliquot of waste. This aliquot may not undergo the actual extraction. These preliminary evaluations include the determination of the following: (*1*) percent solids, (*2*) whether the waste contains dry solids in excess of 0.5 %, and (*3*) which of the two extraction fluids are to be used for extraction of the waste.

10.2 *Preliminary Determination of Percent Solids*—Percent solids is defined as that fraction of the waste sample (as a percent of the total sample w/w) from which no liquid may be forced out by an applied pressure as described below.

10.2.1 If the waste will obviously yield no free liquid when subjected to pressure filtration of this method (that is, 100 % solids), proceed to 10.4.

NOTE 4—Some materials may look like dry solids but may release liquids under pressure, for example, adsorbents, filter cakes, paint, and other sludges. If uncertain, proceed to the filtration step (10.2.2).

10.2.2 If the sample is liquid or multi-phasic, liquid/solid separation to make a preliminary evaluation of percent solids is required. This involves the filtration device described in 6.3, and the procedure is outlined in 10.2.3 through 10.2.9.

10.2.3 Pre-weigh the filter and the container that will receive the filtrate.

10.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

10.2.5 Weigh out a sub-sample of the waste (100-g minimum) and record the weight.

10.2.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

10.2.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. Allow the sample to warm to room temperature in the device before filtering.

NOTE 5—If some waste material (>1 % of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in 10.2.5 to determine the weight of the waste sample that will be filtered.

10.2.7.1 Gradually apply gentle pressure of 1 to 10 psi (7 to 70 kPa), until the pressurizing gas moves through the filter. If this point is not reached below 10 psi (69 kPa), and if no additional liquid has passed through the filter in any 2-min interval, slowly increase the pressure in 10-psi (69-kPa) increments to a maximum of 50 psi (345 kPa). After each incremental increase of 10 psi (69 kPa), if the pressurizing gas has not moved through the filter, and if no additional liquid has passed the filter in any 2-min interval, proceed to the next 10-psi (69-kPa) increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (345 kPa) (that is, filtration does not result in any additional filtrate within any 2-min period), stop the filtration.

NOTE 6—Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

TABLE 1 Sample Maximum Holding Times, Days

Period Compound	From Field Collection to Method Extraction	From the End of Extraction to the Start of Filtration, h	From Method Extraction to Analytical Extraction	From the Analytical Extraction to the Chemical Analysis	Total Time, Days
Organics	14	2	7	40	61
Mercury	28	2	NA	28	56
Inorganics except Mercury	180	2	NA	180	360

10.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE 7—Some wastes, such as oily and some paint wastes, will obviously contain some material that appears to be liquid. Even after applying the pressure filtration as outlined in 10.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as solid. Do not replace the original filter under any circumstances. Use only one filter.

10.2.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see 10.2.3) from the total weight of the container plus filtrate. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in 10.2.5 or 10.2.7. Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{percent solids} = \frac{\text{weight of solid (10.2.9)}}{\text{total weight of waste (10.2.5 or 10.2.7)}} \times 100 \tag{1}$$

10.3 If the percent solids determined in 10.2.9 is equal to or greater than 0.5 %, proceed with the filtered solid phase to 10.3.1. If the percent solids determined in 10.2.9 is less than 0.5 %, the initial liquid phase becomes the method extract. Measure the pH and proceed with this method extract to 11.16.

10.3.1 Remove the solid phase and filter from the filtration apparatus.

10.3.2 Dry the filter and solid phase at 100 ± 20°C until two successive weighings yield the same value within 1 % of the last measurement. Record the final weight.

NOTE 8—**Caution:** Take care to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or another appropriate device.

10.3.3 Calculate the percent dry solids as follows:

$$\text{percent dry solids} = \frac{(\text{weight of dry waste} + \text{filter}) - \text{tared weight of filter}}{\text{initial weight of waste (10.2.5 or 10.2.7)}} \tag{2}$$

10.3.4 If the percent dry solids is less than 0.5 %, the initial liquid phase becomes the method extract. Proceed with the extract to 11.16. Measure the pH of the method extract. If the percent dry solids is greater than or equal to 0.5 %, return to the beginning of this section and filter a small, fresh portion of the waste to determine the appropriate extraction fluid (10.4). Since the original sample was destroyed by drying and the preliminary evaluation has been completed, proceed to 11.3 to perform the extraction procedure on a fresh sample.

10.4 *Determination of Appropriate Extraction Fluid* (See Fig. 3)—If the dry solids content of the waste is greater than or equal to 0.5 %, perform the determination of the appropriate fluid (7.7) to be used for the extraction as follows:

10.4.1 Weigh out a small sub-sample of the solid phase of the waste; reduce the solid, if necessary, to a fragment size of approximately 1 mm in diameter or less; and transfer 5.0 g of the solid phase of the waste to a 500-mL beaker or an Erlenmeyer flask.

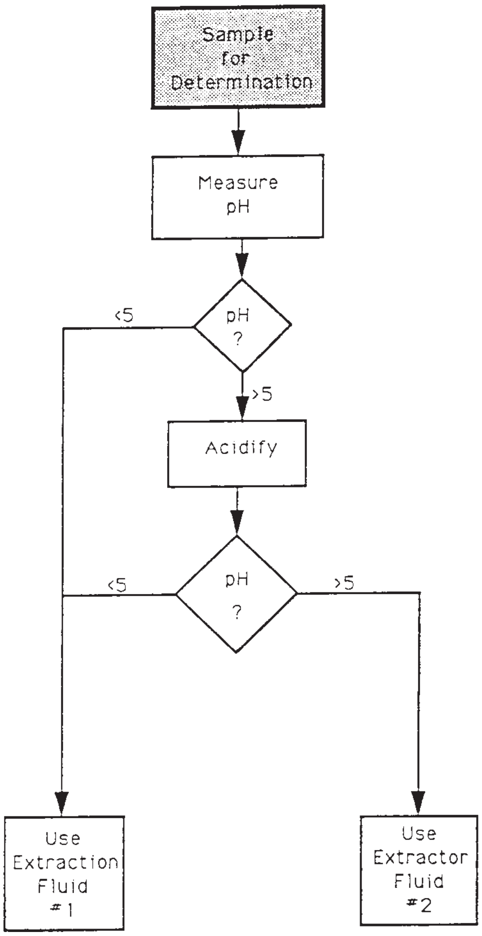

FIG. 3 Determination of the Extraction Fluid

10.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watch-glass, and stir vigorously for 5 min using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use Extraction Fluid No. 1. Proceed to Section 11.

10.4.3 If the pH from 10.4.2 is >5.0, add 3.5 mL 1 N HCl, slurry briefly, cover with a watch-glass, heat to 50°C, and hold at 50°C for 10 min.

10.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use Extraction Fluid No. 1. If the pH is >5.0, use Extraction Fluid No. 2. Proceed to Section 11.

10.5 If the aliquot of the waste used for the preliminary evaluation (10.2 through 10.4) was determined to be 100 % solids at 10.2.1, it can be used for Section 11 extraction (assuming that at least 100 g remains). The aliquot subjected to the procedure in 10.2.7 might be appropriate for use in Section 11 if an adequate amount of solids (as determined by 10.2.9) was obtained. The amount of solids necessary is also dependent on whether a sufficient amount of extract will be produced to support the analyses for the target analytes. If an adequate amount of solids remains, proceed to 11.11.

11. Extraction

11.1 A sample size of minimally 100 g (solid and liquid phases) is required. A larger sample size may be appropriate in some cases, depending on the solid contents of the waste sample (percent solids; see 10.2); whether the initial liquid phase (filtrate) will be miscible with the aqueous extract of the solid; and whether inorganics, semivolatile organics, pesticides and herbicides are all analytes of concern. If the amount of extract generated by a single extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

11.2 If the waste will obviously yield no liquid when subjected to pressure filtration (that is, is 100 % solid; 10.2), weigh out a sub-sample of the waste (100 g minimum) and proceed to 11.10.

11.3 If the sample is liquid-like or multi-phasic, liquid-solid separation is required. This involves the filtration device described in 6.3 and is outlined in 11.4 through 11.9.

11.4 Pre-weigh the container that will receive the filtrate.

11.5 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if inorganics are of concern (see 6.4).

NOTE 9—Acid washed filters may be used for all extractions, even when inorganics are not of concern.

11.6 Weigh out a sub-sample of waste (100-g minimum) and record the weight. If the waste contains <0.5 % dry solids (10.3), the liquid portion of the waste, after filtration, is defined as the method extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required. For wastes containing >0.5 % dry solids (10.2 or 10.3), use the percent solids information obtained in 10.2 to determine the optimum sample size (100-g minimum) for filtration. Sufficient solid should be generated by filtration to support the analyses to be performed on the method extract.

11.7 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

11.8 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder. Spread the waste sample evenly over the surface of the filter. Allow the sample to warm to room temperature in the device before filtering.

NOTE 10—If some waste material (>1 % of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in 10.2.5 to determine the weight of the waste sample that will be filtered.

11.8.1 Gradually apply gentle pressure of 1 to 10 psi (7 to 70 kPa), until the pressurizing gas moves through the filter. If this point is not reached below 10 psi (69 kPa), and if no additional liquid has passed through the filter in any 2-min interval, slowly increase the pressure in 10-psi (69-kPa) increments to a maximum of 50 psi (345 kPa). After each incremental increase of 10 psi (69 kPa), if the pressurizing gas has not moved through the filter, and if no additional liquid has passed the filter in any 2-min interval, proceed to the next 10-psi (69-kPa) increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (345 kPa) (that is, filtration does not result in any additional filtrate within any 2-min period), stop the filtration.

NOTE 11—Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

11.9 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Measure the pH of the filtrate. Measure the volume of the filtrate (V_1) if the data are to be combined mathematically. Use a graduated measuring cylinder for the volume measurement. The liquid phase may now be analyzed (see 11.13) or stored at 4°C until the time of analysis.

NOTE 12—Some wastes, such as oily and some paint wastes, will obviously contain some material that appears to be liquid. Even after applying the pressure filtration as outlined in 10.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as solid. Do not replace the original filter under any circumstances. Use only one filter.

11.10 If the waste contains <0.5 % dry solids (see 10.3), proceed to 11.14. If the waste contains >0.5 % solids (see 10.2 or 10.3), proceed to 11.11.

11.11 Quantitatively transfer the solid residue retained by the filter, or the solid sample if it did not require filtration, into an extractor bottle. Include the filter if it was used to separate the initial liquid from the solid phase.

11.12 Determine the amount of extraction fluid to add to the extractor as follows:

$$\text{weight of extraction fluid} = \frac{20 \times \text{percent solids (10.2.9)} \times \text{weight of waste filter (11.6 or10.2.9)}}{100} \tag{3}$$

Slowly add this amount of appropriate extraction fluid (see 10.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that polytetrafluoroethylene tape be used to ensure a tight seal), secure in a rotary agitation device, and rotate at 30 ± 2 r/min for 18± 2 h. Ambient temperature (that is, temperature of the room in which the extraction takes place) shall be maintained at 23 ± 2°C during the extraction period.

NOTE 13—As agitation continues, pressure may build up within the extractor bottle for some types of wastes (for example, limed or calcium carbonate-containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be taken under a hood and opened carefully from time to time (for example, after 15 min, 30 min, and 1 h).

11.13 Within 2 h, following the 18 ± 2 h extraction, initiate the separation of the material in the extraction vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in 11.8. For final filtration of the method extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see 6.4) if inorganics are of concern.

11.14 Prepare the method extract as follows:

11.14.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from 11.13 is defined as the method extract. Proceed to 11.15.

11.14.2 If compatible (for example, multi-phase waste will not result upon combination), combine the filtered liquid resulting from 11.13 with the initial liquid phase of the waste obtained in 11.8. This combined liquid is defined as the method extract. Proceed to 11.15.

11.14.3 If the initial liquid phase of the waste, as obtained from 11.8, is not or may not be compatible with the filtered liquid resulting from 11.13, do not combine these liquids. Analyze these liquids, collectively defined as the method extract, and combine the results mathematically, as described in 11.15.

11.15 Following collection of the method extract, the pH of the extract should be recorded. The volume of the extract (V_2) shall be measured if the data are to be combined mathematically. Use a graduated measuring cylinder for volume measurement. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with 1 N nitric acid to pH <2. If precipitation is observed upon the addition of nitric acid to a small aliquot of the extract, the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The method extract shall be prepared and analyzed according to appropriate analytical methods. The method extracts to be analyzed for metals shall be acid-digested except in those instances in which digestion causes a loss of metallic contaminants. If an analysis of the undigested extract reveals that the concentration of any regulated metallic contaminant exceeds the acceptance level, the waste fails the test and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste met the set acceptance level. If the individual phases are to be analyzed separately, determine the volumes of the individual phases (to ± 3 %), using a separatory funnel and graduated cylinder. Conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{final analyte concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2} \qquad (4)$$

where:

V_1 = volume of the initial phase (filtrate, L),
C_1 = concentration of the contaminant of concern in the initial phase (mg/L),
V_2 = volume of the extract (L), and
C_2 = concentration of the analyte of concern in the extract (mg/L).

NOTE 14—If either V_1 or V_2 were not detected, use the appropriate detection limit in the calculation.

11.16 Compare the analyte concentration of the method extract with the applicable comparative values. Refer to Section 13 for quality assurance requirements.

12. Report

12.1 Report the following information:

12.1.1 All pH values for the method extract(s);

12.1.2 The concentration of all analytes of concern as measured or calculated in 11.6 and 11.15, respectively;

12.1.3 The type of the extraction fluid used;

12.1.4 The solids content of the sample; and

12.1.5 The dry solids content of the sample if the solids content is > 0.5 %.

13. Quality Assurance Requirements

13.1 Maintain all data, including quality assurance data, and keep them available for reference or inspection.

13.2 All quality control measures described in the appropriate analytical methods shall be followed. If the analytical quality control requirements are not specified in the appropriate method, follow the requirements in 13.3 and 13.4 and refer to Practice ES 16.

13.3 A minimum of one blank (using the same extraction fluid and equipment as used for the samples) for every ten extractions that have been conducted shall be used as a check to determine whether any memory effects from the extraction equipment are occurring.

13.4 A matrix spike shall be performed for each waste type where the compositions of the waste matrices are significantly different.

NOTE 15—It is recommended that it be assumed that each sample has a significantly different composition unless previous data would indicate otherwise (for example, regular sampling of steady-state process streams). If more than 20 samples of the same waste are being tested, a matrix spike must be performed for every 20 samples.

13.4.1 The matrix spikes are to be added after filtration and combination, if necessary, of the method extract and before preservation.

13.4.2 Matrix spike levels should be established at the appropriate acceptance levels. If the analyte concentration in the method extract is less than one half of the acceptance level, the spike level may be as low as one-half the analyte concentration. However, it shall not be less than the quantitation limit or one-fifth of the acceptance level. In order to avoid differences in the matrix effects, the matrix spikes must be added to the same nominal volume of the method extract as that which was analyzed for the unspiked sample.

13.4.3 The purpose of the matrix spike is to monitor the performance of the analytical method used and to determine whether matrix interference exists. The use of other internal calibration methods, modifications of the analytical methods, or the use of alternate analytical methods may be needed to measure accurately the analyte concentration of the method extract when recovery of matrix spike is below the expected analytical method performance.

13.4.4 Acceptable sample holding times are outlined in 9.7. Exceeding the holding time is not acceptable to establish any compliance with the acceptance levels, but it may be used if the samples are not in compliance.

14. Precision and Bias

14.1 *Precision*—The precision of the procedure in Test Method D 5233 for measuring the sample disintegration, surface area increase, has been evaluated. The fractions retained on 9.5 mm sieve were measured in three laboratories. In each laboratory, duplicate, monolithic samples not passing a 9.5 mm sieve of four high strength and four low strength materials were extracted (see Table 2). The weight of the residual 9.5-mm size fractions was measured and expressed as a percent of the initial sample weight.

TABLE 2 Precision Data

	High Strength	Low Strength
Mean, %	21.0	1.9
Standard deviation,%	3.0	0.63

14.2 *Bias*—The procedure in Test Method D 5233 for measuring extract generation has no bias because the value of extract is defined only in terms of this method.

15. Keywords

15.1 batch; extraction; laboratory; leaching; single; sludge; solid; solidified; testing; waste

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