



Designation: ~~D4793-93 (Reapproved 2004)~~ Designation: D 4793 - 09

Standard Test Method for Sequential Batch Extraction of Waste with Water¹

This standard is issued under the fixed designation D 4793; 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 (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is a procedure for the sequential leaching of a waste containing at least five % solids to generate solutions to be used to determine the constituents leached under the specified testing conditions.

1.2 This test method calls for the shaking of a known weight of waste with water of a specified purity and the separation of the aqueous phase for analysis. The procedure is conducted ten times in sequence on the same sample of waste and generates ten aqueous solutions.

1.3 This test method is intended to describe the procedure for performing sequential batch extractions only. It does not describe all types of sampling and analytical requirements that may be associated with its application.

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1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 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.

2. Referenced Documents

2.1 *ASTM Standards:*²

D 75 ~~Practices~~Practice for Sampling Aggregates

D 420 ~~Practice for Investigating and Sampling Soil and Rock for Engineering Purposes~~Guide to Site Characterization for Engineering Design and Construction Purposes

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 1129 Terminology Relating to Water

D 1193 Specification for Reagent Water

D 2216 ~~Test Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures~~Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D 2234/D 2234M ~~Test Method~~Practice for Collection of a Gross Sample of Coal

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D-19D19 on Water

D 3370 ~~Practices for Sampling Water~~Practices for Sampling Water from Closed Conduits

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, see Terminology D 1129.

~~3.2 Symbols:~~3.2 Symbols:

3.2.1 Variables listed in this test method are defined in the individual sections where they are discussed. A list of defined variables is also given in Section 11.

3.2.2 *Explanation of Variables:*

\bar{X}_t = total mean value

\bar{X}_a = analytical mean value (calculated using data from analysis of standards)

¹ This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.04 on Ancillary Topics.

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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.

- S_{tt} = total standard deviation
 S_{ta} = analytical standard deviation
 S_{te} = estimated standard deviation due to the extraction procedure
 S_{ot} = total single operator standard deviation
 S_{oa} = analytical single operator standard deviation
 S_{oe} = estimated single operator standard deviation due to the extraction procedure

4. Significance and Use

4.1 This test method is intended as a means for obtaining sequential extracts of a waste. The extracts may be used to estimate the release of certain constituents of the waste under the laboratory conditions described in this test method.

4.2 This test method is not intended to provide extracts that are representative of the actual leachate produced from a waste in the field or to produce extracts to be used as the sole basis of engineering design.

4.3 This test method is not intended to simulate site-specific leaching conditions. It has not been demonstrated to simulate actual disposal site leaching conditions.

4.4 An intent of this test method is that the final pH of each of the extracts reflects the interaction of the extractant with the buffering capacity of the waste.

4.5 An intent of this test method is that the water extractions reflect conditions where the waste is the dominant factor in determining the pH of the extracts.

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

4.7 This test method has been tested to determine its applicability to certain inorganic components in the waste. This test method has not been tested for applicability to organic substances, volatile matter (see Note 3 in 5.15), or biologically active samples.

4.8 The agitation technique, rate, liquid-to-solid ratio, and filtration conditions specified in the procedure may not be suitable for extracting all types of wastes (see Sections 7, 8, and the discussion in Appendix X1).

5. Apparatus

5.1 *Straightedge* (such as a thin-edged yard stick).

5.2 *Impermeable Sheet*, of glazed paper, oil cloth, or other flexible material of a composition suitable to the analytes of interest.

5.3 *Drying Pans or Dishes*—Two per waste (for example, aluminum tins, porcelain dishes, or glass weighing pans), suitable to the waste being tested and the instructions given in 9.2.

5.4 *Drying Oven*—Any thermostatically controlled drying oven capable of maintaining a steady temperature of $\pm 2^\circ\text{C}$ in a range from 100 to 110°C.

5.5 *Desiccator*, having the capacity to hold the drying pans described in 5.3 and the crucibles described in 5.8.

5.6 *Laboratory Balance*, capable of weighing to 0.1 g.

5.7 *Pipet*, 10-mL capacity.

5.8 *Crucibles*—Two per waste, porcelain, 20-mL capacity each.

5.9 *Analytical Balance*, capable of weighing to 0.1 mg.

5.10 *Large Glass Funnel*.

5.11 *Wash Bottle*, 500-mL capacity.

5.12 *pH Meter*—Any pH meter with a readability of 0.01 units and an accuracy of ± 0.05 units at 25°C is acceptable.

5.13 *Agitation Equipment*, of any type that rotates the extraction vessel in an end-over-end fashion at a rate of $30 \pm 2 \text{ r/min}$ $0.5 \pm 0.03 \text{ Hz}$, such that the axis of rotation is horizontal and it goes through the center of the bottle, (see Fig. 1 and the discussion of agitation in Appendix X1).

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

5.14 *Pressure Filtration Assembly*—A pressure filtration device of a composition suitable to the nature of the analyses to be performed and equipped with a 0.45 or 0.8- μm pore-size filter (see Note 7, pertaining to 9.4).

5.15 *Extraction Vessels*, cylindrical, wide-mouth, of a composition suitable to the nature of the waste and analyses to be performed, constructed of materials that will not allow sorption of constituents of interest, and sturdy enough to withstand the impact of the falling sample fragments. Container size should be selected so that the sample plus extraction fluid occupy approximately 95 % of the container. Containers must have water-tight closure. Containers for samples where gases may be released should be provided with a venting mechanism.

NOTE 2—Suitable container sizes range from 4.0 to 4.5 in. (102 to 114 mm) in diameter and 8.5 to 13.0 in. (216 to 330 mm) in height. 2—Suitable container sizes range from 10 to 11 cm in diameter and 22 to 33 cm in height.

NOTE 3—The venting of 3—Venting the container has the potential to affect the concentration of volatile compounds in the extracts.

5.15.1 Extraction vessels should be cleaned in a manner consistent with the analyses to be performed. See Practices D 3370; Section 13., Section 13.

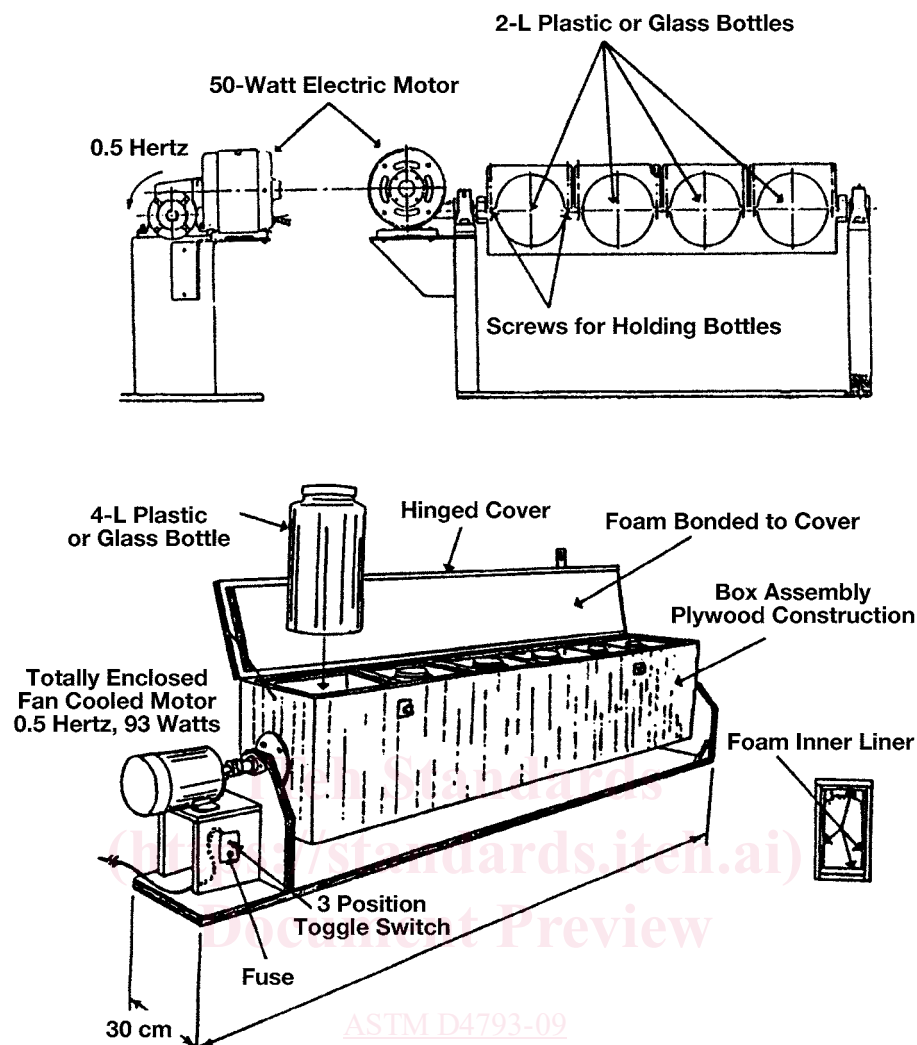


FIG. 1 Extractors

6. Reagents

6.1 *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 American Chemical Society, where such specifications are available.³ 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type IV reagent water at 18 to 27°C (Specification D 1193). The method by which the water is prepared, that is, distillation, ion exchange, reverse osmosis, electro dialysis, or a combination thereof, should remain constant throughout testing.

7. Sampling

7.1 Obtain a representative sample of the waste to be tested using ASTM sampling methods developed for the specific industry where available (see Practices D 75 and D 420 and D 420, Terminology D 653, and Test Method D 2234/D 2234M).

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

7.3 The amount of sample to be sent to the laboratory should be sufficient to perform the solids content determination as specified in 9.2 and to provide 100 g of sample on a dry weight basis for each extraction.

7.4 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 leaching characteristics of the sample. Waste samples should contain a representative distribution of particle sizes.

³ "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."

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

NOTE 4—Information on obtaining representative samples can also be found in *Pierre Gy's Sampling Theory and Sampling Practice*, Volumes I and II, by F. Picard, CRC Press, 1989.

7.5 In order to prevent sample contamination or constituent loss prior to extraction, keep samples in closed containers appropriate to the sample type and desired analysis. See Practices D 3370 for guidance. Record the storage conditions and handling procedures in the report.

7.6 The time between collection and extraction of the sample should be determined by the nature of the sample and the information desired. See Practices D 3370 for guidance. Report the length of time between sample collection and extraction.

8. Sample Preparation

8.1 For free-flowing particulate solid wastes, obtain a sample of the approximate size required in the test by quartering the sample (Section 7) received for testing on an impermeable sheet of glazed paper, oil cloth, or other flexible material as follows:

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

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

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

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

8.1.5 Repeat 8.1.2.

8.1.6 With a straightedge (such as a thin-edged yard stick), one at least as long as the flattened mound of sample, gently divide the sample into quarters. Make an effort to avoid using pressure on the straightedge sufficient to cause damage to the particles.

8.1.7 Discard alternate quarters.

8.1.8 If further reduction of sample size is necessary, repeat 8.1.3-8.1.7. Use a sample size to give 100 g of solid for each extraction. Provide additional samples for determination of solids content. If smaller samples are used in the test, report this fact.

NOTE 5—For other acceptable methods for mixing and subsampling free-flowing solid particulate wastes, see *Pierre Gy's Sampling Theory and Sampling Practice*, Volumes I and II, by F. Picard, CRC Press, 1989. The method of subsampling should be determined by the physical properties of the waste, analytes of interest, and equipment available.

8.2 For field-cored solid wastes or castings produced in the laboratory, cut a representative section weighing approximately 100 g for testing plus samples for determination for solids content. Shape the sample so that the leaching solution will cover the material to be leached.

8.3 For multiphase wastes, mix thoroughly to ensure that a representative sample will be withdrawn. Take samples for determination of solids content at the same time as the test samples.

9. Procedure

9.1 Record the physical description of the sample to be tested, including particle size so far as it is known.

9.2 *Solids Content*—Determine the solids content of two separate portions of the sample as follows:

9.2.1 Dry to a constant weight at $104 \pm 2^\circ\text{C}$ two dishes or pans of size suitable to the solid waste being tested. Cool in a desiccator and weigh. Record the values to ± 0.1 g.

9.2.2 Put an appropriately sized portion of sample of the waste to be tested into each pan. Scale the weight used to the physical form of the waste tested. Use a minimum of 50 g, but use larger samples where particles larger than 10 mm in average diameter are being tested (see Test Method D 2216).

9.2.3 Dry 16 to 20 h at $104 \pm 2^\circ\text{C}$. Record the temperature and time of the drying period.

9.2.4 Cool to room temperature in a desiccator and reweigh. Record the mass to ± 0.1 g.

9.2.5 Repeat steps 9.2.3 and 9.2.4 until constant container-sample masses are obtained. Discard the dried samples following completion of this step.

9.2.6 Calculate the solids content of the sample from the data obtained in 9.2.2 and 9.2.4 as follows:

$$S = A/B \quad (1)$$

where:

A = mass of sample after drying, g,

B = original mass of sample, g, and

S = solids content, g/g.

Average the two values obtained. Record the solids content.

9.3 *Extraction Procedure*—If the entire procedure cannot be conducted without interruption, at least the first four extraction sequences must be conducted without interruption.

9.3.1 Determine the mass of the extraction vessel to be used in the extraction procedure to the nearest 0.1 g. Record the mass of the extraction vessel, $M_{v,1}$. Use one extraction vessel per waste throughout the sequence of extractions.

9.3.2 Add 100 g (weighed to ± 0.1 g) of solid waste on a dry weight basis to the extraction vessel. Calculate the amount of waste

as received to add using the following equation:

$$M = \frac{100}{S} \quad (2)$$

where:

M = mass of waste as received to add to the extraction vessel to give 100 g (weighed to ± 0.1 g) of solid waste.

9.3.2.1 If a mass of solid waste on a dry weight basis other than 100 g is used, (Eq 2) through (4) must be modified to reflect the use of a mass other than 100 g. Replace 100 in these equations with the mass used. Use of a mass other than 100 g is not recommended.

9.3.3 Add a volume in millilitres, V_{vt} , of test water (see 6.2) to the extraction vessel determined using the following equations:

$$M_{sw} = M - 100 \quad (3)$$

where:

M_{sw} = mass of moisture in the sample added to the extraction vessel, g.

$$V_{vt} = (20)(100) - M_{sw} \quad (4)$$

9.3.4 Agitate continuously for 18 ± 0.25 h at 18 to 27°C . Record the agitation time and temperature.

9.3.5 Open the extraction vessel. Observe and record any visible physical changes in the sample and leaching solution. Record the pH of the waste/leaching solution slurry.

9.4 *Filtration*—Transfer as much of the waste/leaching solution as possible through a large glass funnel to a pressure filtration device equipped with a 0.45 or $0.8\text{-}\mu\text{m}$ filter. Transfer the mixed slurry. Do not decant. Invert the extraction vessel over the filtration device and allow the liquid to drain from the solid remaining in the extraction vessel for 1 min. It is important to achieve as complete a transfer of fluid from the extraction vessel to the filtration device as possible. Pressure filter the liquid through the filter using nitrogen gas. After the extract has passed through the filter, continue running nitrogen gas through the filtration device at 30 psi for 3 min. The filtrate obtained is the extract mentioned in this test method (see 9.5, 10.8, and 10.9). Determine the volume of the filtrate collected and report it as V for that extraction step. Measure the pH of the extract immediately, remove the volume of filtrate necessary for determination of total dissolved solids content in 9.5, and then preserve the extract in a manner consistent with the chemical analyses or biological testing procedures to be performed (Practices D 3370, Section 15).

NOTE 6—It is recommended that all filtrations be performed in a hood.

NOTE 7—Analytical results may be affected by the type of filter used. If a $0.8\text{-}\mu\text{m}$ filter pore size is used, the resulting extract should be digested prior to elemental analysis. If the filter is composed of material that may contaminate the extract during filtration, the filter should be washed in the filtration device in a manner consistent with the chemical analyses or biological testing procedures to be performed on the extract. For example, for elemental analysis of the extract, if a filter composed of borosilicate glass fiber is used, it should be washed in the filtration device with a dilute acid solution and rinsed with approximately 2 L of water prior to filtration to prevent contamination.

NOTE 8—Prefilters can be used only if it is absolutely necessary (if the filtrate for analysis or testing cannot be obtained unless a prefilter is used) due to loss of sample trapped in the pores of the prefilter and the possibility of the prefilter disintegrating during rinsing.

9.5 *Total Dissolved Solids Content (TDS)*—Transfer a 10.0-mL sample of the extract to each of two preweighed crucibles (weighed to ± 0.1 mg), previously dried at $110 \pm 2^\circ\text{C}$. Place the samples in a drying oven at $110 \pm 2^\circ\text{C}$ for 3 h. Record the drying oven temperature and drying time. Remove the crucibles and let cool in a desiccator. Reweigh the crucibles and record their weights to ± 0.1 mg.

NOTE 9—Only one drying is performed to limit the contact time between the solid and the rinse water in the extraction vessel prior to the next extraction step (see 9.6 and Section 10).

9.6 Quantitatively transfer the damp solid from the filter back to the original extraction vessel, including the filter. Use water (see 6.2) from a pre-weighed wash bottle to assist in this transfer and to rinse the filtration device. No more than 500 mL of water should be used for rinsing. Use the smallest volume of wash water possible to achieve a thorough transfer. Using tweezers or a similar device, recover the filter and rinse the adhering solid into the extraction vessel with water from the pre-weighed wash bottle. Do not leave the filter in the extraction vessel. Reweigh the wash bottle to determine the amount of water used in the transfer. Record this value as M_w . Weigh the extraction vessel following the transfer described above and record this value as M_v . The extraction vessel may be sealed until a feasible time for addition of new extraction fluid. This is to enable filtration during the next sequence at a reasonable time during the day. If the slurry is stored for longer than 6 h in the extraction vessel prior to the addition of new extraction fluid, the data generated by the analysis of the extracts should be plotted to check for perturbation of the data curve.

10. Calculation

10.1 Calculate the total dissolved solids contents, TDS, in milligrams per litre of the filtrate using the following equation:

$$TDS = (M_{sc} - M_c)(100) \quad (5)$$

where:

M_{sc} = mass of the crucible and dried solids, mg, and
 M_c = mass of the crucible, mg.

10.2 Calculate the mass of the solid in grams lost through dissolution, M_d , using the following equation:

$$M_d = (TDS)(V)(0.001) \quad (6)$$

where:

V = volume of filtrate collected in that extraction, L, and

M_d = mass loss through dissolution.

10.3 Calculate the mass of the solid corrected for TDS remaining for the next extraction step, M_s , using the following equation:

$$M_s = M_s^{e-1} - M_d \quad (7)$$

where:

M_s^{e-1} = mass of the solid extracted in the current extraction step, g.

NOTE 10—For example, in beginning the first extraction, M_s^{e-1} will equal 100 g, and to calculate the mass of solid remaining for the second extraction step, M_s will equal $100\text{ g} - M_d$.

10.4 Calculate the combined mass of the solid and the residual liquid in the extraction vessel, M_{sl} , using the following equation:

$$M_{sl} = M_v - M_{v1} - M_w \quad (8)$$

10.5 Calculate the mass of liquid adhering to the solids in the extraction vessel, M_l , using the following equation:

$$M_l = M_{sl} - M_s \quad (9)$$

10.6 Calculate the volume in millilitres of new test water to be added to the extraction vessel, Test Water Volume, TWV, using the following equation:

$$TWV = [(M_s)(20)] - M_l - M_w \quad (10)$$

10.7 Add to the extraction vessel the amount of new test water, TWV, determined in 10.6, and repeat 9.3.4 through 10.7 so that ten extractions are done in sequence.

NOTE 11—This procedure assumes that the amount of waste that is trapped in the filters after rinsing is negligible.

10.8 Analyze the extracts for specific constituents or properties or use the extracts for biological testing procedures as desired using appropriate ASTM test methods. Where no appropriate ASTM test methods exist, other methods may be used and recorded in the report. Where phase separation occurs during the storage of the extracts, appropriate mixing should be used to ensure the homogeneity of the extracts prior to their use in such analyses or testing.

10.9 *Compensation for Carry-Over* —For each constituent in each of the extracts generated in the extraction sequence, the contribution to concentration from the residual liquid from the previous extraction step, C_j , can be calculated using the following equation:

$$C_j = [M_{il}/20(M_s^{e-1})][C_i] \quad (11)$$

where:

C_i = concentration of the constituent in the filtrate from the previous extraction step,

M_{il} = M_1 from the previous extraction step, and

M_s^{e-1} = mass of solid extracted in the current extraction step (see Note 10).

11. Definition of Variables

11.1 The following variables must be determined when performing the sequential batch extraction procedure:

11.1.1 *Solids Content Determination* :

A = mass of the sample after drying in the determination of the solids content of the waste to be extracted, g,

B = original mass of the sample prior to drying in the determination of the solids content of the waste to be extracted, g, and

S = solids content of the waste to be extracted, g/g.

11.1.2 *First Extraction Step*:

M = mass of waste as received added to the extraction vessel to give 100 g (weighted to ± 0.1 g) of solid on a dry weight basis for the first extraction step, g,

V_{vl} = volume of test water to be added for the first step in the extraction procedure, mL, and

M_{sw} = mass of the moisture in the sample to be extracted in the first extraction step, g.

11.1.3 *TDS Determination*: