



Designation: D 4793 – 93

Standard Test Method for Sequential Batch Extraction of Waste with Water¹

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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.

1.4 *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:

D 75 Practices for Sampling Aggregates²

D 420 Practice for Investigating and Sampling Soil and Rock for Engineering Purposes³

D 653 Terminology Relating to Soil, Rock, and Contained Fluids³

D 1129 Terminology Relating to Water⁴

D 1193 Specification for Reagent Water⁴

D 2216 Test Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures³

D 2234 Test Method for Collection of a Gross Sample of Coal⁵

D 2777 Practice for Determination of Precision and Bias of Methods of Committee D-19 on Water⁴

D 3370 Practices for Sampling Water⁴

¹ This test method is under the jurisdiction of ASTM Committee D-34 on Waste Management and is the direct responsibility of Subcommittee D34.02 on Physical and Chemical Characterization.

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² Annual Book of ASTM Standards, Vol 04.03.

³ Annual Book of ASTM Standards, Vol 04.08.

⁴ Annual Book of ASTM Standards, Vol 11.01.

⁵ Annual Book of ASTM Standards, Vol 05.05.

3. Terminology

3.1 Definitions:

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

3.2 Symbols: 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
X_a	= analytical mean value (calculated using data from analysis of standards)
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.

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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 ± 2 r/min 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.

NOTE 3—The venting of 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.

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.⁶ 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, electrodialysis, 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, Terminology D 653, and Test Method D 2234).

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.

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

⁶ "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."

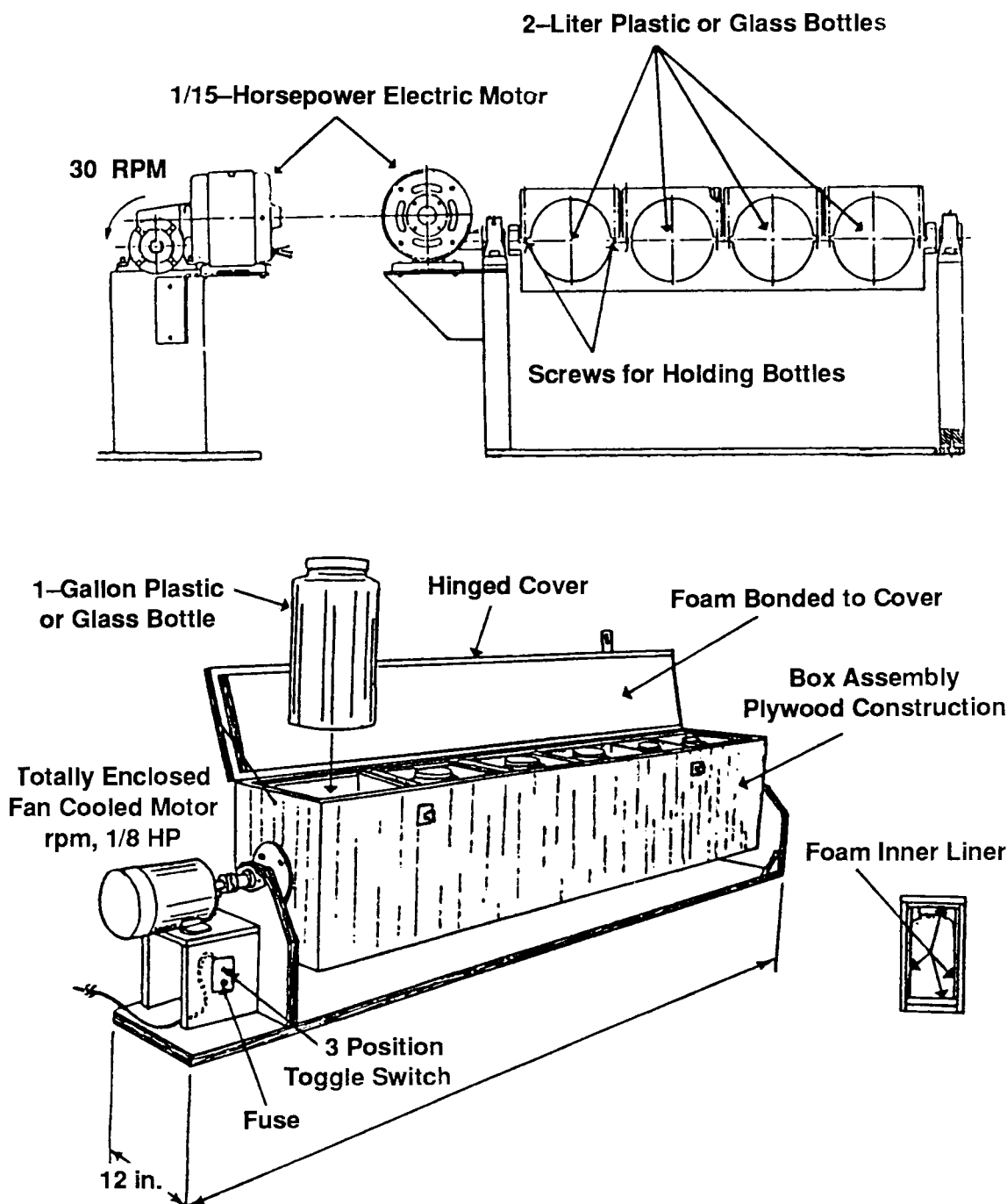
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FIG. 1 Extractors

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 straight-edge 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.