



## Standard Test Method for Leaching Solid Material in a Column Apparatus<sup>1</sup>

This standard is issued under the fixed designation D 4874; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method is a standard laboratory procedure for generating aqueous leachate from materials using a column apparatus. It provides a leachate suitable for organic analysis of semivolatile and nonvolatile compounds as well as inorganic analyses.

1.2 The column apparatus is designed and constructed of materials chosen to enhance the leaching of low concentrations of semivolatile and nonvolatile organic constituents as well as to maximize the leaching of metallic species from the solid. Analysis of column effluent provides information on the leaching characteristics of material under the conditions used in the test.

1.3 This test method provides for the passage of an aqueous fluid through materials of known mass in a saturated up-flow mode.

1.4 It is intended that the sample used in the procedure be physically, chemically, and biologically representative of the material.

1.5 This test method does not produce results that can be used as the sole basis for (1) engineering design of a disposal site, or (2) the characterization of wastes based on their leaching characteristics.

1.6 *This test method has the following limitations:*

1.6.1 Maximum particle size is 10 mm (0.4 in.). Particle size reduction is not recommended. Large-diameter material (cinders, rocks, and so forth) should be removed prior to packing the column to ensure adequate compaction.

1.6.2 Test materials containing densely immiscible organic material may result in phase separation and lead to column plugging.

1.6.3 This test method does not differentiate between dissolved constituents and sub-70- $\mu\text{m}$  particulates that pass through the pores of the end plates.

1.6.4 This test method is not applicable to the leachability characterization of materials with regard to volatile compounds.

1.6.5 This test method is not applicable to the characterization of materials that dissolve in water to a degree that

significantly impacts the void volume in the column or the determination of the specific gravity of the material.

1.7 Application of this test method to materials with initial low permeability, or to those that lose permeability over the course of the test, may result in long testing periods.

1.8 The values stated in SI units are to be regarded as the standard. The values given in parentheses are in approximate inch-pound equivalents.

1.9 *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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

C 819 Test Method for Specific Surface Area of Carbon or Graphite<sup>2</sup>

D 422 Test Method for Particle Size Analysis of Soils<sup>3</sup>

D 698 Test Method for Laboratory Compaction Characteristics of Soil Using Standard Effort (12 400 ft-lbf/ft<sup>3</sup>) (600 kN-m/m<sup>3</sup>)<sup>3</sup>

D 854 Test Method for Specific Gravity of Soils<sup>3</sup>

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water<sup>4</sup>

D 1129 Terminology Relating to Water<sup>4</sup>

D 1293 Test Methods for pH of Water<sup>4</sup>

D 1498 Practice for Oxidation-Reduction Potential of Water<sup>4</sup>

D 1888 Test Methods for Particulate and Dissolved Matter in Water<sup>5</sup>

D 2216 Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock<sup>3</sup>

D 2434 Test Method for Permeability of Granular Soils (Constant Head)<sup>3</sup>

D 3370 Practices for Sampling Water<sup>4</sup>

D 3694 Practice for Preparation of Sample Containers and for Preservation of Organic Constituents<sup>6</sup>

D 4253 Test Methods for Maximum Index Density and Unit

<sup>1</sup> 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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<sup>2</sup> *Discontinued*, see 1988 Annual Book of ASTM Standards, Vol 15.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 04.08.

<sup>4</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>5</sup> *Discontinued*, see 1990 Annual Book of ASTM Standards, Vol 11.01.

<sup>6</sup> Annual Book of ASTM Standards, Vol 11.02.

Weight of Soils Using a Vibratory Table<sup>3</sup>  
 E 691 Practice for Conducting an Interlaboratory Study to  
 Determine the Precision of a Test Method<sup>7</sup>

**3. Terminology**

3.1 *Definition:*

3.1.1 *reagent water*—as defined in SW-846, Method 1311, 5.2.<sup>8</sup>

3.1.2 *void volume*—the volume between the solid particles in a bed of granular material. Also called the interstitial volume.

3.2 Several terms used in this test method are defined in Terminology D 1129.

**4. Significance and Use**

4.1 This test method is intended to provide an aqueous leaching of a material in a dynamic partitioning manner.

4.2 Specific operating conditions for the column can be selected to satisfy the objectives of individual studies. An understanding of the fundamental principles governing column leaching is necessary in the selection of appropriate conditions. References (1-3)<sup>9</sup> provide useful information on these principles.

4.3 The column apparatus is constructed of materials that permit the generation of a leachate which is suitable for low concentration organic analysis for semivolatile and nonvolatile compounds as well as inorganic species.

**5. Apparatus (See Fig. 1)**

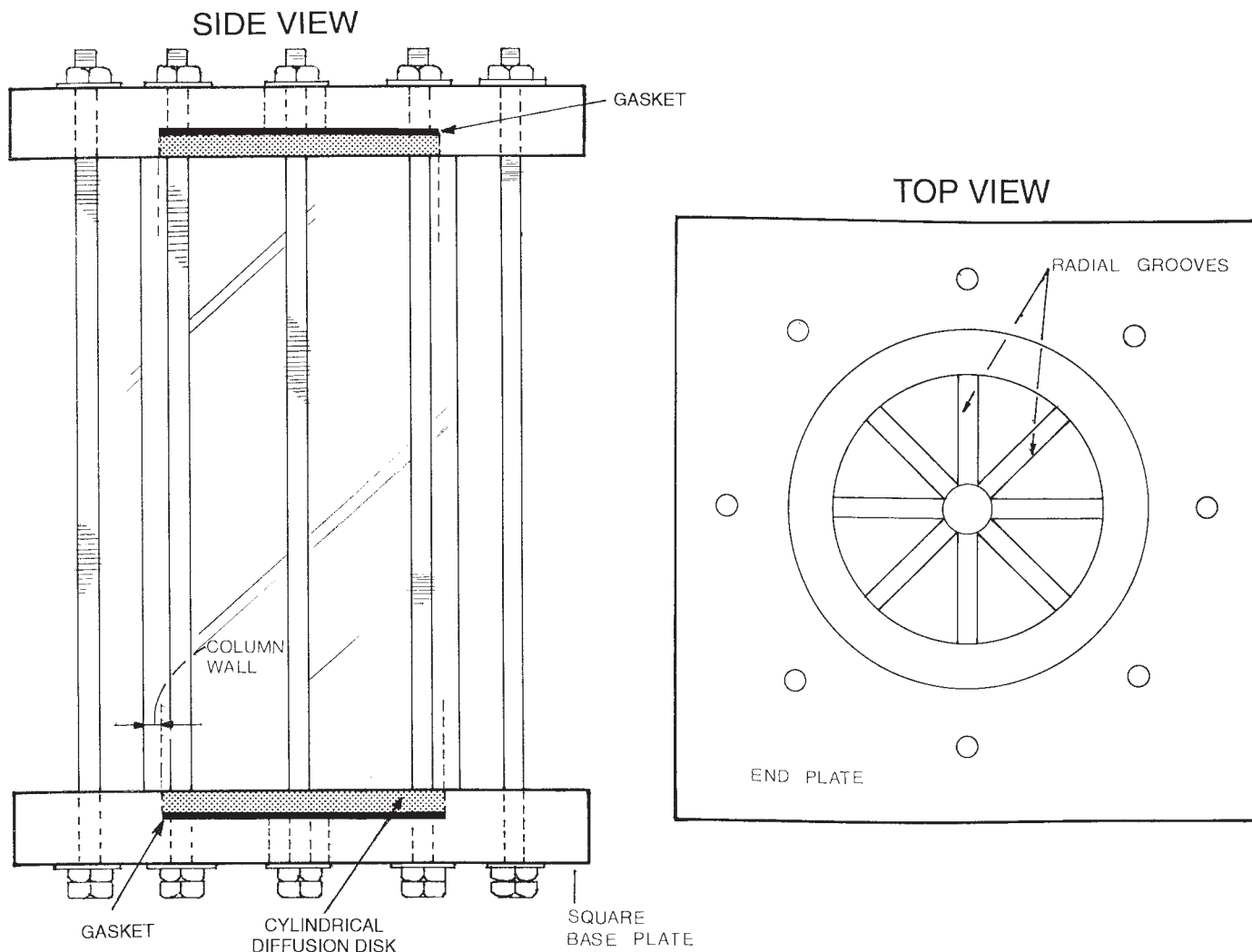
5.1 *Columns:*

5.1.1 The column body is constructed of glass pipe, 300 mm (12 in.) in length, with an inside diameter of 100 mm (4 in.). The cylinder wall must be of sufficient thickness, approximately 6 mm (1/4 in.), to withstand the operating pressure.

<sup>7</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>8</sup> SW-846, 3rd ed., Method 1311, Available from USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

<sup>9</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.



**FIG. 1 Column Apparatus**

5.1.2 End plates are constructed of stainless steel. They are attached by means of eight, 6-mm (1/4-in.) threaded rods or any other means which ensures a leakproof seal.

5.1.3 Gaskets, one at each end of the column, are constructed of chemically inert materials, and are as thin as possible while still providing a good seal. The gasket diameter should overlap the inside column diameter by 3 mm (1/8 in.) to prevent the gasket from being forced out while under pressure. Techniques other than gaskets for providing a seal between the column and end plates are allowed provided the technique used is specified in the report.

5.1.4 Flow distribution disks must be constructed of sintered stainless steel, with a nominal pore diameter of 70  $\mu\text{m}$ . The disk thickness shall be 6 mm (1/4 in.), with a diameter equal to the inside diameter of the column, approximately 100 mm (4 in.). The disk shall have eight evenly spaced grooves; each of which is 3 mm (1/8 in.) wide, 3 mm deep and 50 mm (2 in.) long. These grooves shall be positioned in a ray originating from the center of the disk.

5.1.5 Tubing used in the apparatus shall be of inert materials, for example, glass, stainless steel, polytetrafluoroethylene lined. The outer diameter shall be 6 mm (1/4 in.).

5.1.6 Substitution of materials of construction of the column or any of its parts is acceptable, as long as it is demonstrated that levels of contamination for analyte(s) of interest are equal to or less than those specified. Any modification of the apparatus as described in this test method must be justified, documented, and delineated in the report.

5.2 *Pressurized Reservoir Vessel*, which is used to contain the leaching fluid, is constructed with requirements similar to those of the leaching column with the following exceptions: (1) no diffusion disks are used, and (2) it is equipped with a top port for refilling the fluid.

5.3 *Balance*, 10-kg capacity, with a 1-g sensitivity.

5.4 *Compressed Gas Source*, prepurified nitrogen or argon with a two-stage delivery regulator (0 to 350 kPa) (0 to 50 psig), and a pressure gage capable of measuring the pressure in the head space of the liquid reservoir to within  $\pm 7$  kPa (1 psig).

## 6. Reagents and Materials

6.1 American Chemical Society (ACS) Reagent grade chemicals or equivalent are preferred.<sup>10</sup>

6.2 Other grades of chemicals may be used, provided that the reagent is of sufficiently high purity to permit its use without compromising the objectives of the testing.

6.3 Demonstration of acceptability through reagent blank data at or below the quantitation limits for all analytes of interest is required.

6.4 Unless otherwise indicated, references to reagent water mean water as defined in SW-846, Method 1311, 5.2. See reagent water under the terminology section of this test method (3.1.1).

NOTE 1—Reagent water is defined in SW-846 as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest.

## 7. Safety Precaution

7.1 General operating pressure should not exceed 275.8 kPa (40 psig) with materials as defined herein. Other inert materials are available that can be used to manufacture the column which will withstand pressures above 275.8 kPa (40 psig).

## 8. Sampling

8.1 Sampling must be performed so as to obtain a representative sample of the material.

8.2 Where no specific sampling methods are available, sampling methods for materials of physical form similar to the material shall be used.

8.3 A minimum sample of 5000 g, or three column volumes, whichever is larger, shall be sent to the laboratory for each column.

8.4 Samples must be kept in closed containers appropriate to the sample type and otherwise protected if necessary prior to testing to prevent sample contamination or constituent change or loss. Where it is desired to test biologically or chemically active samples in their existing state, any sample storage required should be at 4°C (Practice D 3370) and the leaching should be started within 8 h of sample collection. Where appropriate, the tester may modify a test portion before leaching to simulate the results of biological or chemical activity in the field. Record the storage conditions, sampling procedures, handling practices, and any abnormal sampling conditions in the report.

## 9. Preparation of Apparatus

9.1 The assembled apparatus is shown in Fig. 1.

9.2 *Column Preparation:*

9.2.1 Before use, clean all parts of the test apparatus that will contact the waste material, leaching fluid, or product leachate.

9.2.1.1 Clean the sintered disks by boiling them for 15 min in reagent water, followed by a backflush with reagent water. Then saturate the disks with concentrated sulfuric acid and soak until all residues are removed. Then vacuum or pressure remove the excess acid with reagent water. Next, pump or draw either acetone or methanol through the disks, followed by either hexane or methylene chloride. Permit disks to air dry.

9.2.1.2 Clean the column apparatus by washing it with a nonionic surfactant soap and water. Rinse with tap water and follow with a reagent water rinse. Then rinse it with either acetone or methanol, followed by a rinse with either hexane or methylene chloride. Permit the apparatus to air dry.

9.3 Assemble the apparatus as depicted in Figs. 1 and 2. Weigh the dried, clean empty column, including end caps and other fittings necessary to contain the waste, and record the mass. This is the tare of the apparatus. Record the inside diameter and height of that part of the column to be filled with waste (that is, the column cylinder). If, following column saturation, the tester wishes to check the degree of saturation,

<sup>10</sup> *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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

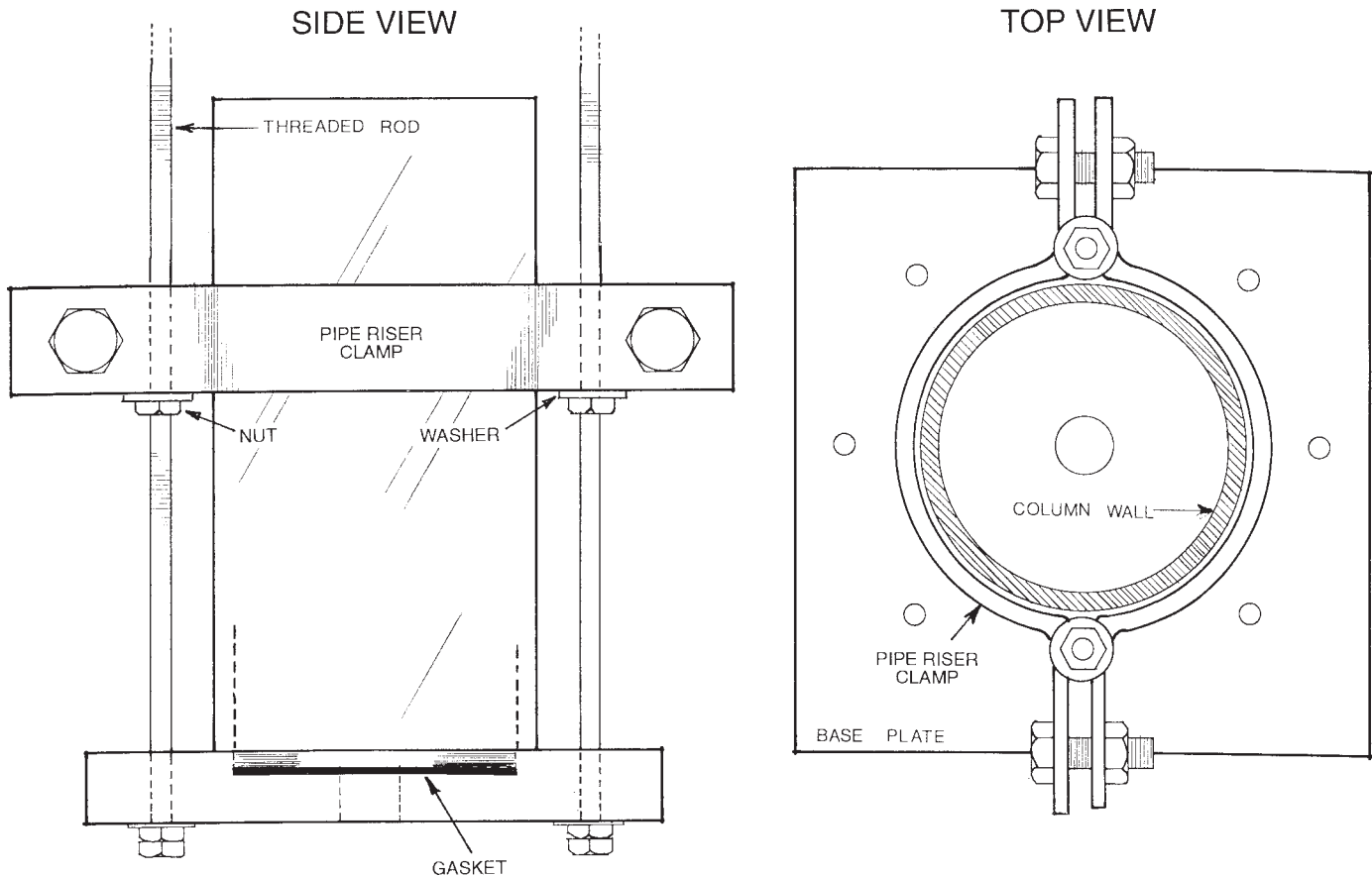


FIG. 2 Packing Configuration

it will first be necessary to determine the mass of water that can be contained in the porous flow distribution disks, end plates, and fittings.

## 10. Procedure

### 10.1 Preconditioning:

10.1.1 Prepare a test portion of waste in a manner that simulates the state the waste is in or will be in as it undergoes leaching in the field. Preparation of the test portion may include such factors as curing, and adjustment of moisture content and density. For such adjustments, the following procedures can be used where appropriate.

10.1.2 *Moisture Content*— Adjust the moisture content to that defined in the disposal scenario by dewatering or adding reagent water to the material. Increase moisture content to that defined in the disposal scenario by addition of reagent water. Note and record the volume of reagent water added. If the concentration of any trace analyte of concern found in the reagent water exceeds the reporting (quantitation) limit for the volume added, note this on the report form. Decrease moisture content by determining scenario temperature (normally less than 60°C) and drying the material at the temperature for a specified time interval. Determine actual moisture content of the waste as it is placed in the column using Test Method D 2216 and an appropriate drying temperature. Note and record the drying temperature used. Record the moisture content.

10.1.3 *Density*—Density is to be adjusted by vibration (see Test Method D 4253) or compaction (see Test Method D 698) to the anticipated field density. Pack the waste material in the column so that uniform density is achieved. This can be checked by visual observation of the waste in the transparent column.

10.1.4 *Curing*—Aging may be necessary for those materials that undergo physical or chemical changes with time in order to obtain a specimen (test portion) representative of the desired conditions. Curing may be accomplished in the field before testing in the column to produce a physical and chemical state representative of the waste as it undergoes leaching in the field. Note and record field conditions of temperature, humidity, and atmospheric pressure. Record the duration of the curing process and report with the test data.

10.1.5 *Particle Size*— Ensure that the particle size distribution of the waste as placed in the column is representative of that expected in field placement. Interpretation of results is based in part on a knowledge of the particle size distribution and surface area of the material. Maximum particle diameter must not exceed  $\frac{1}{10}$  of the inside diameter of the column. Particle size reduction is not recommended. An evaluation of the particle characteristics of size distribution and surface area may be useful. Test Methods D 422 and C 819 should be used to evaluate these properties of the waste.

### 10.2 Column Filling:

10.2.1 Fill the column by compacting or vibrating the waste in five approximately equal layers. Approximate the expected field density. Scarify the top surface of each layer before adding the next increment. Do not scarify the top layer after final compaction. Fill the column to its final height, ensuring that the final height of the compacted material is equal to the height of the leaching column.

10.2.2 After filling the column, determine the mass, including the same fittings as in the determination of the tare. Record the total mass.

### 10.3 Leaching:

10.3.1 The leaching process is conducted in a continuous up-flow mode.

10.3.2 *Saturation*—After assembling the column as shown in Fig. 1, saturate the column with reagent water by the method outlined in Test Method D 2434 for determining permeability coefficients using a constant head permeameter. In this case, the column assembly replaces the constant head permeameter of that procedure. If it is determined that it is not possible to saturate the waste using Test Method D 2434, it may be necessary to increase the vacuum used in the procedure or attempt to saturate the column under pressure, or both, to promote the dissolution of gasses.

10.3.3 Water used to saturate the waste should be of the same quality as that to be used to leach the waste in the column. Collect a sample of this water at the time of saturation and analyze for the same properties and constituents as will be measured in the column effluent. An appropriate sample container, similar to that used for sample collection should be chosen and cleaned. Sample container guidance is given in 10.6.1. The test portion will be collected using the procedures for leachate collection.

10.3.4 *Determination of the Void Volume*—Void volume is estimated from the measurement of specific gravity of the solids in accordance with Test Method D 854, the mass of the wet solids placed into the column, the moisture content of the material in accordance with Test Method D 2216, and the volume of the column. Although the drying temperatures used in Test Methods D 854 and D 2216 can be selected on the basis of the specific characteristics of the material being dried, the drying temperatures used in the two procedures must be the same for the data to be used for porosity and void volume calculations.

10.3.4.1 Measure the mass of the sample material which fills the column apparatus. Note and record the moisture content of the material, as well as the specific gravity and any other specific information relevant to the material.

10.3.4.2 Calculate the void volume using the following equation:

$$V_p = V_c \{M / [(1 + w) \times S \times D]\} \quad (1)$$

where:

$V_p$  = void volume in the column,  $\text{cm}^3$ ,

$V_c$  = volume of the column,  $\text{cm}^3$ ,

$M$  = as-packed mass of the material, including moisture, contained in the column,

$w$  = moisture content of the material contained in the column as a mass ratio from Test Method D 2216, g water/g solids

$S$  = specific gravity of the material as determined in Test Methods D 854, unitless, and

$D$  = density of water,  $\text{g}/\text{cm}^3$ .

10.3.5 Estimate the degree of saturation by calculating the porosity  $n$  of the material using the following equation:

$$n = V_p / V_c \quad (2)$$

Calculate the mass gain in the saturated material. Add the value of the mass gain to the mass of the water in the packed column. Compare the value to the calculated void volume. The ratio is the degree of saturation. The degree of saturation can be estimated by determining the mass gain due to the water added to the waste during the saturation process, adding this mass gain to the mass of water in the packed column before saturation, and comparing this to the void volume just calculated. The mass gain can be determined by subtracting the mass of the packed, unsaturated column plus the mass of the water retained in the end plates, flow distribution disks, and fittings, from the mass of the saturated column.

### 10.4 Effluent Flow:

10.4.1 Once the column is saturated, begin the testing period. Adjust the pressure or hydraulic head, or both, so that one complete void volume exchange rate is accomplished in  $24 \pm 3$  h. Maximum operating column pressure, for apparatus specified, should not exceed 275 kPa (40 psig). Read and record operating pressure, and effluent start time. If some test period other than  $24 \pm 3$  h (an alternate void volume period) is warranted, record justification, time interval required, operating conditions, and effluent start time. Collect all column effluents as discrete void volume increments.

10.4.2 *Operating Requirements*—The column must be operated continuously. Note and record stop and restart times for all cessations. Determine total stoppage time for each occurrence per day and total daily stoppage. Cease test if, on any one day, total stoppage exceeds 1 h.

10.4.3 No type of channeling is permitted. When channeling is noted, note the time and cease the test. Determine if samples generated previously are usable. Document rationale for acceptance/rejection of previous sample effluent. Columns shall be protected from extraneous light except during column checks and sample collection intervals. Note and record all exposure periods.

10.4.4 Columns are to be run at ambient temperature, under normal test conditions. Sufficient pressure is required to maintain flow at the specified rate which will yield a void volume during the specified time interval. Temperature and pressures must be read and recorded periodically. For safety reasons, do not operate this equipment above 275 kPa (40 psig).

### 10.5 Leachate Collection:

10.5.1 The column must be inspected periodically and adjustments made if necessary to maintain the desired operating conditions.

10.5.2 Sufficient leachate from void volume periods shall be collected to establish a basis for judgment on completeness of

the test. Minimally, it is recommended that Void Volumes 1, 2, 4, and 8 be collected and analyzed. Additional void volumes may provide additional useful data. The minimum number and sequence numbers of void volumes that will be collected and analyzed must be identified in advance of any collection activity.

10.5.3 Void volumes are sampled by collecting all column effluent generated over a  $24 \pm 3$  h or other period corresponding to the void volume of interest.

10.5.4 Minimize contact of the leachate with the atmosphere. Fill effluent collection vessel head space with pre-purified nitrogen or argon. Protect vessel head space with a water trap.

10.5.5 Collect effluent in appropriate containers. Containers shall be cleaned in an appropriate manner. Containers must be free from contamination for those parameters of interest in the leachate. Follow the procedures outlined in Practice D 3694 or Ref (4), or both.

#### 10.6 *Leachate Sample Preparation:*

10.6.1 Sample containers must be constructed of materials that do not alter the sample quality. Follow the procedures outlined in Practice D 3694 or Ref (4), or both.

10.6.2 Sample containers must be cleaned according to Practice D 3694, methods contained in Ref (4), or as appropriate for the constituents of interest.

10.6.3 If collected leachates from void volume periods are stirred or agitated before filtration or analysis, this must be noted in the report. Minimize contact between the liquid and air during this mixing. If there are solids visible in the collected leachate before or after mixing, this must be noted in the report.

10.6.4 If filtration of the leachate is desired prior to chemical analyses, then such filtration steps must be noted. Analyze the filtered material separately to ensure that the suspended solids did not contain constituents that are of environmental significance. If suspended solids are found to contain analytes of significance, then redo the column leach test, and analyze the leachate without filtration.

10.6.5 If samples are filtered before analysis, the method of filtration must be noted in the report. Filtration must be completed within 1 h of sample collection. Contact with air must be minimized.

10.6.6 Samples must be stored under refrigeration at 4°C. Sample container head space must be minimized. A tight seal between the cap and the container is expected. Guidance on preservation techniques can be found in Practices D 3694 and D 3370 as well as in Ref (4).

10.7 *Sample Labeling*—At a minimum, the information regarding column source, collection date, collection time, void volume number, sampler's name, sampler's signature, and preservatives used must be recorded.

10.8 *Characterization of the Leachate*—The minimum test portion for characterization is equal to the volume of leachate produced during the void volume period. Suggested analyses for characterization of leachate include: pH, Test Methods D 1293; Conductivity, Test Methods D 1125; Redox Potential, Practice D 1498; Total Solids, Test Method D 1888; and any other analyses that will further characterize the material and provide interpretive guidance.

## 11. Report

11.1 The report must include at least the following information:

11.1.1 Project identification and description,

11.1.2 Description of waste as sampled,

11.1.3 Sampling procedures, waste sample handling, and storage procedures including the date and time of sampling,

11.1.4 All procedures used to condition the waste for placement in the column including curing and moisture adjustment procedures,

11.1.5 Permeability, particle size, and surface area information where available, including disposal scenario specifications; and a complete description of material as sampled,

11.1.6 Temperature used for drying in moisture determination and specific gravity procedures if different than  $110 \pm 5^\circ\text{C}$ ,

11.1.7 Materials used to construct the column apparatus if different than those specified in the test method,

11.1.8 Column washing procedures,

11.1.9 Waste moisture content as sampled and as packed in the column,

11.1.10 Characteristics of the waste as placed in the column including specific gravity of the solids, porosity, and void volume,

11.1.11 Mass of the waste placed in the column,

11.1.12 Saturation procedures used if different than Test Method D 2434, and date and time that column saturation began and ended,

11.1.13 Date and time that leaching began,

11.1.14 Temperature and pressure of column operation at the beginning of the test, and at each time samples are collected or flow rates are adjusted,

11.1.15 Means used to prevent exposure of column contents to light,

11.1.16 Total time for collection of each void volume, the measured volume of the leachate collected during the period, the date and time when measured, any corrections made to flow rates, and quantities of effluent collected between flow rate determinations,

11.1.17 Date, time, and duration of all flow stoppages,

11.1.18 Observations of unusual conditions in the column, such as density variations, cracks, gas-filled spaces, or evidence of fluid channeling along column walls,

11.1.19 Date and time that the tester began and finished collecting each void volume and the number of void volumes collected,

11.1.20 Type and material of construction of sampling containers and methods used to clean sample containers,

11.1.21 Steps taken to avoid sample contact with air where such steps are called for in the test method,

11.1.22 Results of measurements of index properties and any other analyses performed on unfiltered samples within 1 h of collecting the void volume from the column. The results of the analyses of that water used to saturate and leach the waste must also be included, and

11.1.23 All analytical data requested in disposal scenario with complete documentation and traceability through laboratory records.

**TABLE 1 Test Method Precision (Percent Relative Standard Deviations)**

NOTE 1—The top number in each set is the pooled within-laboratory relative standard deviation while the bottom number is the overall interlaboratory variability for a single test result in terms of relative standard deviation.

NOTE 2—Because all chemical analyses were performed by a single laboratory, the total variance does not contain an interlaboratory analytical component.

| Element    | Number of Void Volumes of Liquid Having Passed Through the Column at the Time of Sample Collection |                    |        |                     |                    |         | Average <sup>A</sup> |
|------------|--|--------------------|--------|---------------------|--------------------|---------|----------------------|
|            | 1  | 2                  | 4      | 8                   | 16                 | 32      |                      |
| Aluminum   | 61/130   | 22/62 <sup>B</sup> | 44/54  | 30/40               | 29/85              | 10/50   | 35/72                |
| Calcium    | 9/20   | 5/16               | 6/28   | 7/14                | 14/75              | 13/42   | 9/33                 |
| Cobalt     | 25/64 <sup>B</sup>   | ...                | ...    | ...                 | ...                | ...     | ...                  |
| Chromium   | 13/51  | 12/15              | 16/33  | 8/8                 | 17/33 <sup>B</sup> | ...     | 12/17                |
| Copper     | 25/33  | 8/10               | 12/22  | 18/26               | 33/43 <sup>B</sup> | ...     | 16/23                |
| Iron       | 88/140   | 16/18              | 20/26  | 16/18               | 27/40 <sup>B</sup> | ...     | 35/51                |
| Lithium    | 34/50  | 75/130             | 63/120 | ...                 | ...                | ...     | 57/100               |
| Magnesium  | 130/170 <sup>B</sup>   | ...                | ...    | ...                 | ...                | ...     | ...                  |
| Molybdenum | 5/10   | 13/60              | 59/79  | 17/69               | 51/77 <sup>B</sup> | ...     | 24/55                |
| Nickel     | ...  | ...                | ...    | 50/86 <sup>B</sup>  | 36/41              | 21/34   | 61/38                |
| Phosphorus | ...  | ...                | 64/71  | 85/85 <sup>B</sup>  | 67/73 <sup>B</sup> | ...     | 64/71                |
| Potassium  | 24/82  | 28/110             | 63/120 | 93/100 <sup>B</sup> | 48/80              | ...     | 38/104               |
| Silicon    | ...  | ...                | ...    | ...                 | 34/48              | 22/29   | 28/39                |
| Sodium     | 24/87  | 56/100             | 50/86  | 28/39               | 46/59              | ...     | 41/74                |
| Strontium  | 19/42  | 16/18              | 28/52  | 4/22                | 36/75 <sup>B</sup> | ...     | 17/34                |
| Vanadium   | 49/53 <sup>B</sup>   | 10/27              | 30/60  | 16/18               | 24/33 <sup>B</sup> | 14/24   | 18/32                |
| Zinc       | 99/100 <sup>B</sup>  | ...                | ...    | ...                 | ...                | ...     | ...                  |
|            |  |                    |        |                     |                    | Average | 33/54                |

<sup>A</sup> Average values are based only on data without asterisks.

<sup>B</sup> Based on data sets that contain one to three values below detection limits (out of nine values). Those values below detection limits are assigned the detection limit in the calculations. If more than three values are below the detection limit, the data were not analyzed.

## 12. Precision and Bias

12.1 *Precision*—The precision of this test method has been studied for inorganic materials in a three-laboratory study involving triplicate columns at each laboratory. Fly ash was used as a waste material. Because the fly ash was of very low permeability, end caps without radial grooves were used. The column effluents were analyzed by a single laboratory for metals by emission spectroscopy using an inductively coupled plasma source. Those metals that were measured at concentrations above the method detection limits were used to determine the precision of the column extraction method according to Practice E 691. The precision identified in the study was a measure of the ability of the column method to generate effluents of similar quality from a single waste material in tests conducted at different laboratories, or in tests using multiple columns at a single laboratory. A summary of the precision data is contained in Table 1. The complete interlaboratory study is

described in Ref (5). The pooled within-interlaboratory relative standard deviation for metals data averaged 33 %. The overall interlaboratory single test reproducibility in terms of standard deviation was 54 %. This excludes interlaboratory analytical effects because all analyses were performed by a central laboratory. The analytical precision reported by the central laboratory, at concentrations most frequently measured, was seldom greater than 5 % relative standard deviation.

12.2 Precision data for organic constituents are not available.


12.3 *Bias*—It is not possible to determine the bias of this test method because there are no accepted reference effluents generated.

## 13. Keywords

13.1 column leach; leachate; waste leaching technique

## REFERENCES

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