Designation: D3375 – 16

Standard Test Method for Column Capacity of Particulate Mixed Bed Ion Exchange Materials[1]

This standard is issued under the fixed designation D3375; 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 covers the determination of the performance of particulate mixed bed ion exchange materials in the regenerated form when used for deionization. It is intended for use in testing unused mixed bed materials and samples of regenerated mixed beds from operating units.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.3 *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:*[2]

D1125 Test Methods for Electrical Conductivity and Resistivity of Water
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D1293 Test Methods for pH of Water
D1782 Test Methods for Operating Performance of Particulate Cation-Exchange Materials
D2687 Practices for Sampling Particulate Ion-Exchange Materials

3. Terminology

3.1 *Definitions*—For definitions of terms related to water, refer to Terminology D1129.

4. Summary of Test Method

4.1 This test method consists of exhausting a column of regenerated mixed bed ion exchange material to a specific end point with an influent solution of known composition and volume.

5. Significance and Use

5.1 This test method can be used to evaluate unused mixed bed ion exchange materials for conformance to specifications. When a representative sample of the mixed bed can be obtained from an operating unit, this test method can be used to evaluate the regeneration efficiency by comparison with the same data obtained with new material from the same manufactured lots, or retained samples of the in-place products.

5.2 This test method provides for the calculation of capacity in terms of the volume of water treated to a conductivity end point.

5.3 The test method as written assumes that the cation exchange material has been regenerated to the hydrogen form with acid and the anion exchange material has been regenerated with alkali to the hydroxide or free-base form. In certain applications a cation exchange material in the potassium, ammonium, or other monovalent form may be encountered. Such materials may be tested following this procedure using Test Water A (Test Methods D1782) as the influent and substituting the hardness end point (Test Methods D1782) for the end points prescribed herein.

5.4 In most cases the product tested will be properly mixed and will contain the correct proportions of anion and cation exchange materials. However, if the pH as well as the conductivity of the effluent is measured, the test method will indicate which of the components is present in excess; an acid effluent at breakthrough indicating an excess of regenerated cation exchange groups and an alkaline effluent an excess of regenerated anion exchange groups. In such cases the volumes of the two components obtained in the final backwash will indicate whether this imbalance arises from improper regeneration or from an improper ratio of the two components. It should be noted, however, that not all units are charged with a balanced ratio of anion-exchanging and cation-exchanging

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[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

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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.

groups. Hence, wherever possible, a field sample should be evaluated in comparison with a retained sample of the original charge.

5.5 This test method provides for the calculation of capacity on either a wet weight basis or a volume basis. Although such materials are normally bought and sold in terms of cubic feet, they are actually packaged in wet pounds. Therefore, it is the capacity on a wet weight basis that is directly correlatable to the amount of material in a given shipment.

5.6 Calculation of a volume capacity is based on the exhausted, separated volume of the components after backwashing and resettling the bed. This volume is chosen because it is difficult, if not impossible, to pack a sample of regenerated mixed bed material in a small-diameter column reproducibly.

5.7 This test method may be used to test mixed bed resin cartridges. In such cases the flow rate of test water and the frequency of sampling must be varied to compensate for the approximate volume of resin in the test sample. The test as written assumes a resin volume of approximately 330 mL.

6. Apparatus

6.1 *Test Assembly* (Fig. 1), consisting of the following:

6.1.1 *Column,* transparent supported 2.5 ± 0.25-cm (1 ± 0.1-in.) inside diameter and approximately 150 cm (60 in.) long. The bottom of the column shall be closed and provided with an outlet of about 6-mm inside diameter. Connections shall be provided at the top and the bottom for the admission and removal of the exhausting solution as described in 7.4. Adequate means of regulating and measuring flow shall be provided. Support for the sample shall be provided so that the distance from the sample to the column outlet is at least 5 cm. Calibrate the column in such a manner that the volume readings required by the test method can be made. Make all calibration measurements at 25 ± 5°C.

6.1.2 *Sample Support,* so designed that the distance from the sample to the column outlet is at least 5 cm. A suggested supporting bed utilizes quartz, gravel, glass beads, or other material 1.5 to 3.5 mm in diameter, insoluble in the reagents used, and retained on a corrosion-resistant screen.

6.2 Measuring circuit and in-line conductivity cells as described in Test Methods D1125. A continuous recorder is recommended.

6.3 *pH Meter,* with associated electrodes as described in Test Methods D1293. A continuous recorder is recommended.

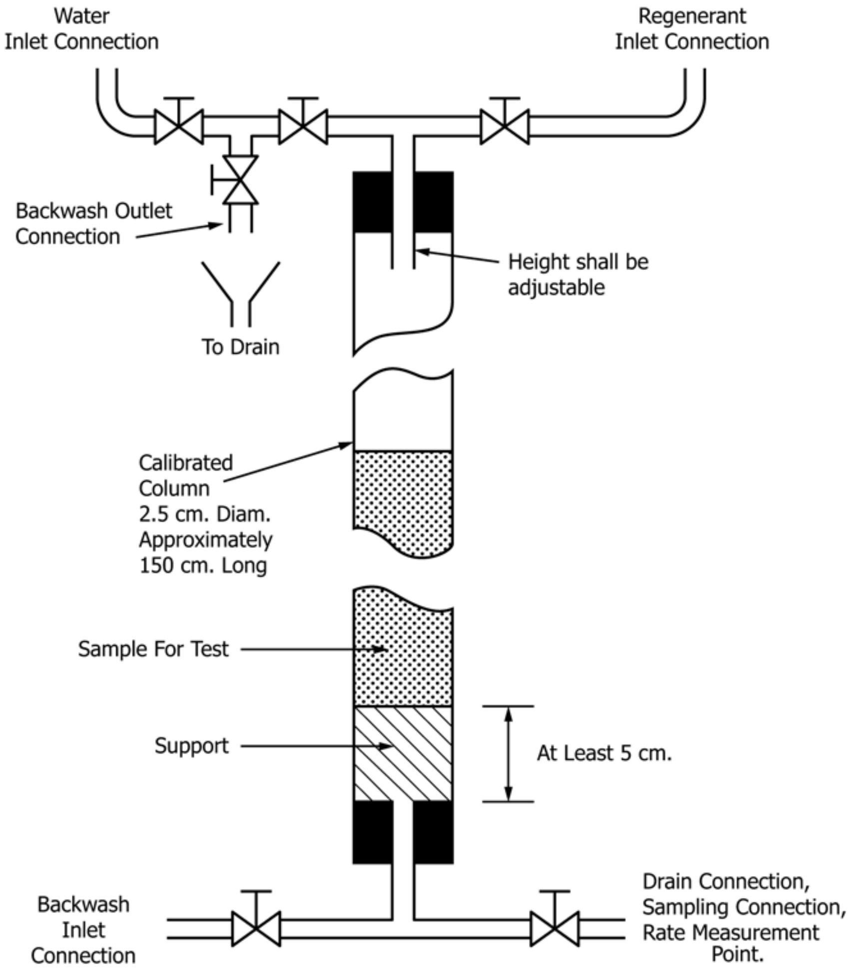

FIG. 1 Typical Arrangement of Apparatus for Performance Testing of Ion Exchange Materials

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.[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.

7.2 *Purity of Water*—All reference to water in this test method shall be understood to mean Reagent Water Type I or II conforming to Specification D1193, with the additional requirement that the silica concentration is less than 0.1 mg/L.

7.3 *Ammonium Hydroxide Solution* (1+19)—Carefully pour 50 mL of concentrated ammonium hydroxide (NH_4OH, sp gr 0.90) into 500 mL of water, stirring constantly. Cool to 25 ± 5°C and dilute to 1 L with water. Mix well.

7.4 *Ion Exchange Test Water D* (10 meq/L)—Prepare a test water containing, in each litre, 0.585 g of oven-dried (105°C) sodium chloride (NaCl). Approximately 25 L of this solution are required for a single test.

7.4.1 To standardize for chloride content, pipet three 100-mL portions. Add one drop of methyl orange indicator solution and one drop of phenolphthalein indicator solution to each and neutralize, if required, by dropwise addition of HNO_3 (1+9) until the color changes from yellow to orange followed by dropwise addition of NH_4OH (1+19) to restore the yellow color. Pipet in 1 mL of K_2CrO_4 solution (50 g/L) and titrate each with standard 0.1 *N* $AgNO_3$ solution until the color of the supernatant solution changes from yellow to red-orange and persists for 30 s with vigorous swirling. Record the average number of millilitres of $AgNO_3$ solution used to ±0.02 mL.

7.4.2 Calculate the strength of the solution in meq of chloride as follows:

$$\text{Chloride, meq/L} = V \times N \times 10 \tag{1}$$

where:

V = millilitres of $AgNO_3$ solution required for titration, and

N = normality of the $AgNO_3$ solution.

The test solution used should have a concentration of 10 ± 0.5 meq/L of chloride.

NOTE 1—Because of the large quantities of this reagent that are required, it is usually made up semi-quantitatively in large batches and then standardized per the above procedure. If desired, it is acceptable to prepare this reagent quantitatively (as for a primary standard) and then eliminate the above standardization.

7.5 *Methyl Orange Indicator Solution* (1.0 g/L)—Dissolve 0.10 g of methyl orange in water and dilute to 100 mL with water.

7.6 *Nitric Acid* (1+9)—Pour one volume of nitric acid (HNO_3, sp gr 1.42) into nine volumes of water and mix thoroughly.

7.7 *Phenolphthalein Indicator Solution* (10.0 g/L)—Dissolve 1.0 g of phenolphthalein in 100 mL of 95 % ethanol.

NOTE 2—In most cases certain denatured alcohols such as specially denatured Formula Nos. 3A, 30, or 2B may be substituted for ethanol.

7.8 *Potassium Chromate Solution* (50 g/L)—Dissolve 5.0 g of potassium chromate (K_2CrO_4) in 50 mL of water. Dilute to 100 mL with water.

7.9 *Silver Nitrate Solution, Standard* (0.10 *N*)—Dry crystalline silver nitrate ($AgNO_3$) at 105°C for 1 h and cool in a desiccator. Weigh out 17 ± 0.05 g. Transfer to a 1-L volumetric flask with water. Dissolve in 500 mL of water and mix thoroughly. Dilute to 1 L with water at 25 ± 5°C. Mix well. Store the solution in a tightly stoppered amber glass bottle.

7.9.1 To standardize, dry approximately 5 g of reagent sodium chloride (NaCl) in a glass container at 105°C for 2 h. Cool in a desiccator. Weigh accurately three 0.2500 ± 0.0100-g portions of the dried NaCl and transfer to separate 250-mL conical flasks. Add 100 mL of water and swirl to dissolve the NaCl. Pipet in 1 mL of K_2CrO_4 solution (50 g/L) and titrate with the 0.1 *N* $AgNO_3$ solution with vigorous swirling until the color of the solution changes from yellow to red-orange and persists for 30 s.

7.9.2 Calculate the normality of the $AgNO_3$ solution as follows:

$$N = D/0.05845 \times E \tag{2}$$

where:

N = normality of the $AgNO_3$ solution,

D = actual weight of NaCl used, and

E = millilitres of $AgNO_3$ solution used.

8. Sampling

8.1 For sampling refer to Practices D2687.

8.2 When sampling new mixed bed materials the drum-to-drum uniformity of the mixing is an important factor, particularly if the material is to be repackaged into small cartridges. Hence, if samples are taken from three or more separate drums or bags of a given lot a composite sample is not prepared but one column is run from each container sampled. Where the sequence of filling is known such samples should include the first and the last container filled.

9. Procedure

9.1 Weigh 270 ± 1 g of the moist, as received, mixed bed ion exchange material, and record weight to the nearest 0.1 g. Introduce through the backwash line about 25 mm of water to the column. Place about 25 mL of the sample in a large-stemmed plastic funnel inserted in the top of the column. Wash the portion of sample into the column with a minimum of water. Repeat until all the sample is transferred into the column. *Care must be exercised that no stratification takes place during the filling procedure.* Maintain the water level in the column about 10 mm over the bed level to minimize air trapping.

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[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 *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.

9.2 When all the mixed bed ion exchange material has been transferred slowly introduce test water D at the top of the column. Increase the flow rate to 100 mL/min, adjusting the rate as required with a flow regulator in the effluent line.

9.3 Measure and record the effluent conductivity according to Test Methods D1125 every 2 L. Continue until the 1.0 µS/cm value is passed, then record readings for each 500 mL of effluent until two successive readings greater than 50µ S/cm (<20 000 Ω·cm) are obtained.

9.4 When the effluent conductivity exceeds 1.0 µS/cm (1 MΩ·cm), measure the pH of the effluent according to Test Methods D1293. Continue the pH measurements at the same frequency as the conductivity measurements.

9.5 When the conductivity exceeds 50 µS/cm (20 000 Ω·cm), stop the flow of test water and record the total volume (*V*) of test water used in litres.

9.6 Open the backwash valves and backwash the bed with water for 10 min at a flow rate sufficient to maintain a 50 % expansion of the bed. Stop the flow of water and allow the bed to settle. Drain at the rate of 100 mL/min until the water level is 20 to 30 mm above the bed. *Do not jar*. Record the bed volume in millilitres. Repeat the backwash and drain procedure until two successive readings of the resettled bed volume agree within 5 mL. Record the average volume as *S* mL.

10. Calculation

10.1 The operating capacity may be calculated on either a volume or wet weight basis using a conductivity end point.

10.2 Calculate the operating capacity to the conductivity end point on a volume basis as follows:

$$\text{Capacity, meq/mL} = C \times V/S \qquad (3)$$

where:

C = total electrolytes in test water D expressed in milliequivalents per litre as determined in 7.4.2,
V = volume of test water used, litres, measured in 9.5, and
S = volume of sample measured in 9.6.

This capacity may be converted to kilograins per cubic foot as $CaCO_3$ by multiplying by 21.8.

10.3 Calculate the operating capacity to the conductivity end point on a wet weight basis as follows:

$$\text{Capacity, meq/g wet} = C \times V/270 \qquad (4)$$

where *C* and *V* are defined as in 10.2.

NOTE 3—Other end points may be agreed upon. When this is done the species and level selected should be specified with the results.

11. Report

11.1 The report of results of this test method should include not only the capacity as calculated above but the end point used. For certification purposes the averaging of results from three separate samples is customary. However, when samples from individual drums are analyzed the individual results as well as the average should be reported.

12. Precision and Bias

12.1 The collaborative study for this test method involved six laboratories, six operators, and three replicate determinations on one material, IRN-150 mixed bed resin. The analysis of a single material was judged to be sufficient to represent the various mixed resins in general use, since they would be expected to fall in a relatively narrow range of column capacity values.

12.2 The single-operator precision, in a given laboratory, for this test method is equal to ±2.3 % of the column capacity expressed as milliequivalents per millilitre.

12.3 The precision of this test method, within its designated range, may be expressed as follows:

$$S_T = 0.168 - 0.0208X \qquad (5)$$

where:

S_T = overall precision in meq/mL, and
X = mixed bed column capacity expressed as meq/mL for a conductivity end point.

12.4 Bias cannot be determined since known standards are not available.

13. Quality Control

13.1 In the analysis of ion exchange resins, it is not possible to prepare a known standard resin for comparison with the actual sample. Therefore it is impossible to test the accuracy of the results, and the method does not include a bias statement.

13.2 Analysts are expected to replicate samples to determine if the results are within the expected precision stated in Section 12.

13.3 Analysis of the resin column effluent is subject to the quality control requirements of the referenced analytical methods, that is, Test Methods D1125 for conductivity measurements and Specification D1193 for pH measurements.

14. Keywords

14.1 column capacity; conductivity; ion exchange; mixed bed

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