



Standard Test Method for Operating Performance of Anion-Exchange Materials for Strong Acid Removal¹

This standard is issued under the fixed designation D 3087; 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 operating capacity of anion-exchange materials when used for the removal of hydrochloric and sulfuric acid from water. It is designed to simulate operating conditions for strong acid removal and is intended for use in testing both new and used materials.

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

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.* Specific precautionary statements are given in Note 1.

2. Referenced Documents

2.1 ASTM Standards:

- D 1067 Test Methods for Acidity or Alkalinity of Water²
- D 1125 Test Methods for Electrical Conductivity and Resistivity of Water²
- D 1129 Terminology Relating to Water²
- D 1193 Specification for Reagent Water²
- D 2687 Practices for Sampling Particulate Ion-Exchange Materials³

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129.

4. Summary of Test Method

4.1 The test method consists of repeated cycles of backwash, base regeneration, rinse, and exhaustion of the sample in the form of a bed in a transparent column. The exhaustion medium used is an ion-exchange test water.

5. Significance and Use

5.1 This test method can be used for evaluating performance of commercially available anion-exchange materials regardless of the basic strength of the ion exchange groups. When previous operating history is known, a good interpretation of resin fouling or malfunction can be obtained by comparison against a reference sample of unused ion-exchange material evaluated in the same way.

5.2 While resistivity has been chosen as the preferred analytical method for defining the exhaustion end point, with titration as the alternative, it is understood that observation of pH during rinse and the service run can yield useful information. The variations in pH observed with an ion exchange material suspected of having degraded, can be helpful in interpretation of performance when compared with similar data for a reference sample of unused material exhausted in the same way.

6. Apparatus

6.1 *Test Assembly* (see Fig. 1), consisting of the following:
6.1.1 *Column*, transparent, vertically supported, 25.4 ± 2.5 -mm (1.0 ± 0.1 -in.) inside diameter and approximately 1.5 m (60 in.) long. The bottom of the column shall be closed and provided with an outlet of approximately 6-mm ($1/4$ -in.) inside diameter. Connections shall be provided at the top and bottom for the admission and removal of the solutions described in Section 7. Adequate means for measuring and regulating the flow shall also be provided. The column shall be calibrated in such a manner that the volume readings required by the method can be made (see Section 9). All measurements shall be made at $25 \pm 5^\circ\text{C}$.

6.1.2 Support the sample at least 50 mm (2 in.) above the bottom of the column outlet using quartz, gravel, glass beads or other material from 1.5 to 3.5 mm ($1/16$ to $1/8$ in.) in diameter, insoluble in the reagents used, and retained on a corrosion-resistant screen. However, other supports may be used at the discretion of the interested parties.

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,

¹ This test method is under the jurisdiction of ASTM Committee D-19 on Water, and is the direct responsibility of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 11.02.

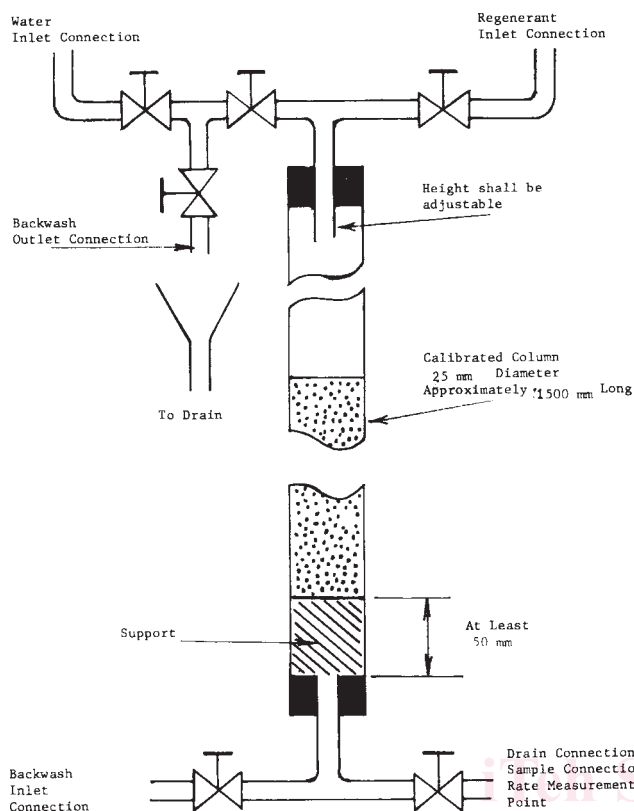


FIG. 1 Typical Arrangement of Apparatus for Performance Testing of Ion-Exchange Materials

where such specifications are available.⁴ 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— Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type III.

7.3 Acidity Test Reagents—For reagents used in determining acidity, refer to Test Methods D 1067.

7.4 Anion Exchange Test Water C (10 meq/L)—Carefully add 18.1 mL of sulfuric acid (H₂SO₄, sp gr 1.84) and 27.5 mL of hydrochloric acid (HCl, sp gr 1.19) to 500 mL of water and dilute to 1 L. Prepare the test water by adding 1 volume of the mixed acid solution to 99 volumes of water. Determine the acidity of the test water in accordance with Test Methods D 1067, titrating to the methyl purple end point. The acidity shall be 10.0 ± 0.5 meq/L (epm).

7.5 Base Regenerants:

7.5.1 For Weak Base Ion-Exchange Materials:

7.5.1.1 Ammonium Hydroxide (40 g NH₃/L)—Dilute 155 mL of ammonium hydroxide (NH₄OH sp gr 0.90) to 1 L with water. The solution should be freshly prepared to avoid absorption of carbon dioxide (CO₂) from the air.

7.5.2 For Weak, Intermediate, and Strong Base Ion-Exchange Materials:

7.5.2.1 Sodium Hydroxide Solution (40 g/L)—Dissolve 40 g of sodium hydroxide (NaOH) in 800 mL of water and dilute to 1 L. The solution should be freshly prepared to avoid absorption of carbon dioxide (CO₂) from the air.

8. Sampling

8.1 For sampling procedures refer to Practices D 2687.

9. Procedure

9.1 Adjust temperature of the water and all solutions to be used in this procedure to 25 ± 5°C and maintain this temperature throughout the test.

9.2 Fill the column approximately half full of water and add sufficient sample to give a bed height of 750 ± 75 mm (30 ± 3 in.) above the top of the support. To avoid drying out of the ion-exchange material, maintain a layer of liquid at least 20 to 30 mm (0.8 to 1.2 in.) deep above the top of the bed at all times during the procedure.

9.3 Backwash with water for 10 min using a flow rate that will maintain a 50 % expansion of the bed. If the supernatant liquid is clear at this point, proceed to 9.4. If the supernatant liquid is cloudy (indicating the presence of light, insoluble, extraneous material), adjust the backwash outlet tube to a height above the bed equal to 75 % of the bed height. Continue backwashing at the same rate until the effluent is clear.

9.4 Allow the bed to settle and then drain at a rate of approximately 100 mL/min until the water level is 20 to 30 mm (0.8 to 1.2 in.) above the top of the bed. Record the volume, in millilitres, of ion-exchange material for use in the following pretreatment. Regenerate the sample with the appropriate dilute sodium hydroxide solution (see Note 2) for 90 min at a flow rate of 0.11 mL/min for each millilitre of ion exchanger in the column. This corresponds to a regeneration level of 400 g/L (25 lb/ft³) of ion exchange material.

9.5 When only a 20 to 30-mm (0.8 to 1.2-in.) layer of liquid remains above the bed, rinse with water using the same flow rate, until a volume equal to the volume of resin has been displaced (one bed volume). Increase the rinse rate to approximately 100 mL/min and continue the rinse until a total of ten bed volumes liquid have been used.

9.6 Backwash with water for 10 min using a flow rate that will maintain a 50 % expansion of the bed. Allow the bed to settle and then drain at rate of approximately 100 mL/min until the water is 20 to 30 mm (0.8 to 1.2 in.) above the top of the bed (**Caution**—see Note 1). Record the volume in millilitres, of ion-exchange material. Repeat the foregoing procedure until two successive volume readings agree within 5 mL. Take the average of these two readings as the sample volume used in calculating the capacity of the ion-exchange material under test.

NOTE 1—**Caution:** The bed must be essentially free of vibration during this measurement; any shock will yield low and false bed volume data.

9.7 Exhaust the ion exchange material with the anion-exchange test water C at a flow rate of 0.33 mL/min/mL of ion exchange material as measured in 9.6. Maintain a head of liquid not less than 50 mm (2 in.) above the top of the bed

⁴ "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 "Analar Standards for Laboratory Chemicals," BDH Ltd., Poole, Dorset, U.K., and the "United States Pharmacopeia."