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An American National Standard

Standard Test Method for Operating Performance of Anion-Exchange Materials for Strong Acid Removal[1]

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 Water[2]
D 1125 Test Methods for Electrical Conductivity and Resistivity of Water[2]
D 1129 Terminology Relating to Water[2]
D 1193 Specification for Reagent Water[2]
D 2687 Practices for Sampling Particulate Ion-Exchange Materials[3]

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 (¼-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 ± 5°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 ⅛ 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,

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[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.

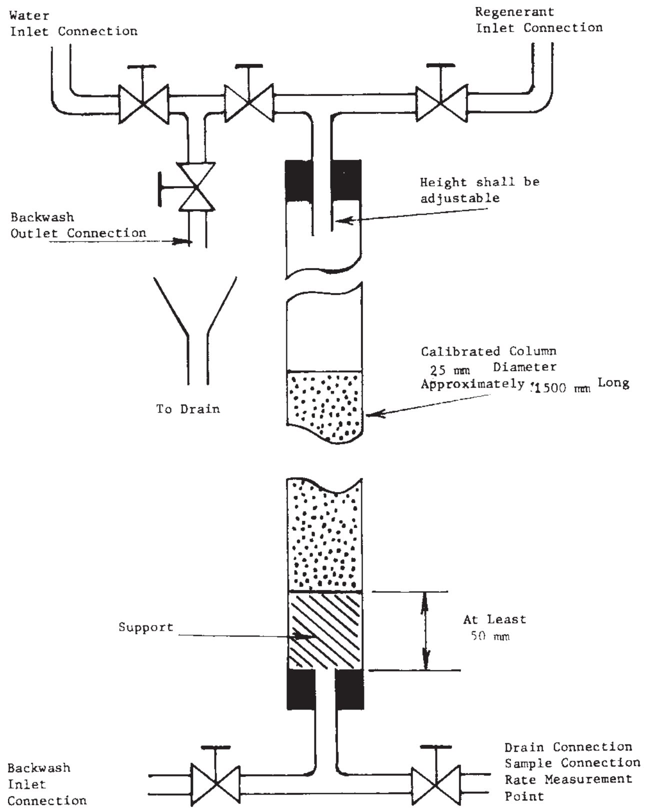

FIG. 1 Typical Arrangement of Apparatus for Performance Testing of Ion-Exchange Materials

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 (H_2SO_4, 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 NH_3/*L)*—Dilute 155 mL of ammonium hydroxide (NH_4OH sp gr 0.90) to 1 L with water. The solution should be freshly prepared to avoid absorption of carbon dioxide (CO_2) 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 (CO_2) 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/ft ^{3}) 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

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[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."

TABLE 1 Test Methods for Anion-Exchanger Effluent

Status	ASTM Designation	Rinse End Point	Service End Point
Preferred	D 1125	20 000 Ω-cm	20 000 Ω-cm
Alternative	D 1067	0.1 meq/L phenolphthalein	0.1 meq/L methyl purple

during the exhaustion. During the run, test the effluent periodically by one of the methods given in Table 1. Continue the run until the designated end point is reached, and record the volume of test water used.

9.8 Repeat the backwash and drain in accordance with 9.6, omitting the determination of bed volume.

9.9 Regenerate the sample with the appropriate NaOH solution (see Note 2), maintaining a flow rate through the bed of 0.054 mL/min/mL of ion-exchange material based on the sample volume determined in 9.6. Continue the flow for 30 min, leaving a 20 to 30-mm (0.8 to 1.2-in.) layer of liquid above the bed. This corresponds to a regeneration level of 64.1 g/L (4 lb/ft^3) of ion-exchange material.

NOTE 2—Alternative regenerant solutions, ammonia and soda ash, may be used in place of caustic for evaluation of weak base ion-exchange material; however, the operating capacity results based on these regenerants may be different than the operating capacity results based on caustic regeneration.

9.10 Rinse the bed with anion-exchange test water C (see Note 3) at the same rate until one bed volume of liquid has been displaced, then increase the rate to 0.33 mL/min/mL of ion-exchange material as measured in 9.6. The liquid level above the top of the bed should be maintained at 50 mm (2 in.). During the rinse, determine the effluent resistivity at least every 15 min, until the resistivity is greater than 20 000 Ω-cm and record the volume of rinse water.

NOTE 3—Test water C is used for the rinse step to simulate the use of cation bed effluent for anion bed rinse which is typical in many industrial demineralizer systems. Alternative rinse solutions, such as water, may be used in the evaluation of other types of systems; however, the operating capacity results based on these rinse solutions may be different than those using test water C as required in 9.10.

9.11 Exhaust the ion-exchange material by maintaining the flow of test water until the effluent resistivity is less than 20 000 Ω-cm. Record the total volume of test water.

9.12 Repeat the cycle, beginning with the backwash (see 9.8) followed by the regeneration (see 9.9). Continue with the rinse (see 9.10) and end with the service run (see 9.11). Repeat until each of three successive runs agrees within ± 5 % of the average capacity as calculated in accordance with Section 10.

10. Calculation

10.1 Calculate the operating capacity, C', in milliequivalents per millilitre of ion-exchange material as follows:

$$C' = [(B - A) \times F] / S$$

where:

A = litres of test water used in rinse (see 9.10),
B = total litres of test water used (see 9.11),
F = acidity of test water, meq/L, (see 7.4) and
S = millilitres of ion-exchange material used (see 9.6).

10.2 Calculate the operating capacity, C'', in kilograins of calcium carbonate per cubic foot, as follows:

$$C'' = C' \times 21.8$$

10.3 Calculate the rinse value, R, in litres of rinse water per litre of bed volume, as follows:

$$R = (A \times 1000) / S$$

where:

A = litres of rinse water (see 9.10), and
S = millilitres of ion-exchange material used (see 9.6).

10.4 Calculate the rinse value, R', in gallons of rinse water per cubic foot of bed volume, as follows:

$$R' = R \times 7.48$$

11. Report

11.1 Report the capacity of test material as the average of three successive service runs that agree within ± 5 % of the average.

12. Precision and Bias [5]

12.1 *Precision*—The Referenced Documents show that seven laboratories participated in a round robin test of the operating performance of anion-exchange materials of four resins using two different regenerants, sodium hydroxide, and ammonium hydroxide. Each of the four resin samples were evaluated by at least three different labs and in some cases four different labs. Each lab evaluated the assigned resin samples at least once, with three labs doing the assigned samples in duplicate, and one lab doing the assigned samples in triplicate. Separate evaluations of two resin samples were made by two people in one lab.

12.1.1 The precision of the values obtained by this test method is dependent upon the magnitude of the observed values and can be estimated from the following relationships:

12.1.1.1 For rinse values in litre of rinse per litre of resin bed (see 10.3):

$$\log S_R = 0.0267R - 0.523$$

where:

S_R = the absolute value of the standard deviation, and
R = rinse value in L of rinse/L of resin bed.

12.1.1.2 For capacity values in kilograins per cubic foot as $CaCO_3$(see 10.2):

$$S_c = 0.0481\, C'' - 0.1641$$

where:

S_c = the absolute value of the standard deviation, and
C'' = operating capacity in kilograins/ft^3.

12.2 *Bias*—Because no materials with known operating capacity can be prepared, calculation of bias is impossible.

13. Keywords

13.1 anion resin; ion exchange; operating performance

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[5] Supporting data have been filed at ASTM Headquarters. Request RR:D19-1011.

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