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# Standard Test Methods for Operating Performance of Particulate Cation-Exchange Materials<sup>1</sup>

This standard is issued under the fixed designation D1782; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 These test methods cover the determination of the operating capacity of particulate cation-exchange materials when used for the removal of calcium, magnesium, and sodium ions from water. It is intended for use in testing both new and used materials. The following two test methods are included:

Sections

Test Method A—Sodium Cycle Test Method B—Hydrogen Cycle	8 to 14 15 to 21 Sections
	Sections
Test Method A—Sodium Cycle	<del>-8 to 14</del>
Test Method B Hydrogen Cycle	<del>15 to 21</del>

- 1.2 The values givenstated in SI units are to be regarded as the standard. The inch-pound units values given in parentheses are for information only 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 safety, health and health environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

ASTM D1782-17

D1067 Test Methods for Acidity or Alkalinity of Water 70b0e5-cb40-42ee-b16e-b9d03 [a0e93/astm-d1782-17

D1126 Test Method for Hardness in Water

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2687 Practices for Sampling Particulate Ion-Exchange Materials

## 3. Terminology

- 3.1 Definitions—Definitions: For definitions of terms used in these test methods, refer to Terminology D1129
- 3.1.1 For definitions of terms used in these standards, refer to Terminology D1129.
- 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 Certain terms in these test methods standards that relate specifically to ion exchange are defined as follows:
- 3.2.2 free mineral acidity—the quantitative capacity of aqueous media to react with hydroxyl ions to pH 4.3.
- 3.2.3 *hydrogen cycle*—the operation of a cation-exchange cycle wherein the removal of specified cations from influent water is accomplished by exchange with an equivalent amount of hydrogen ion from the exchange material.

<sup>&</sup>lt;sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

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<sup>&</sup>lt;sup>2</sup> 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.



3.2.4 theoretical free mineral acidity—the free mineral acidity that would result from the conversion of the anions of strong acids in solution to their respective free acids.

#### 4. Summary of Test Methods

- 4.1 Test Method A consists of repeated cycles of backwash, brine regeneration, rinse, and exhaustion of the sample in the form of a bed in a transparent column. The exhausting medium used is an ion-exchange test water.
- 4.2 Test Method B consists of repeated cycles of backwash, acid regeneration, rinse, and exhaustion of the sample in the form of a bed in a transparent column. The exhausting medium used is an ion-exchange test water.

#### 5. Apparatus

- 5.1 Test Assemble (see Fig. 1), consisting of the following:
- 5.1.1 Column, transparent, vertically supported,  $25.4 \pm 2.5$  mm ( $1.0 \pm 0.1$  in.) in inside diameter and approximately 1500 mm (60 in.) long. The bottom of the column shall be closed and provided with an outlet of approximately 6-mm inside diameter. Connections shall be provided at top and bottom for admission and removal of solutions as described in Section 10. Adequate means for measuring and regulating flow shall be provided. Calibrate the column in such a manner that the volume readings required by the test method can be made. Make all measurements at  $25 \pm 5$ °C.
- 5.1.2 Support, for the sample, so designed that the distance from the sample to the column outlet is at least 50 mm. A suggested supporting bed utilizes quartz, 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. However, other supports such as fritted glass or polyester screens may be used at the discretion of the interested parties.

#### 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 Committee on Analytical Reagents of the American Chemical Society, where

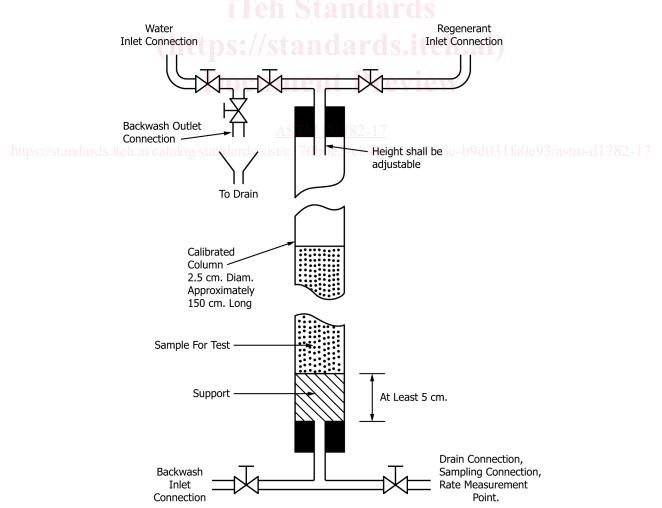


FIG. 1 Typical Arrangement of Apparatus for Performance Testing of Ion-Exchange Materials



such specifications are available.<sup>3</sup> 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 reagent water, Type IV, conforming to Specification D1193.

## 7. Sampling

- 7.1 To obtain a representative sample of particulate ion-exchange material, either from a shipment of new product in the manufacturer's original packages or from a bed of used material, refer to Practices D2687.
- 7.2 Transfer the laboratory sample to a 2-L beaker and add enough water to bring the water level up to that of the ion-exchange material and soak for 1 h (see Note 1). Mix the sample thoroughly and transfer a sufficient representative portion to fill a 400-mL beaker. Use this portion of sample in the procedure.

Note 1—Where new materials are shipped dry, follow the manufacturer's instructions for preconditioning.

#### TEST METHOD A—SODIUM CYCLE

## 8. Scope

8.1 This test method is designed to simulate operating conditions on a sodium cycle used for the removal of calcium and magnesium and other divalent ions from water.

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#### 9. Significance and Use

- 9.1 Cation exchange materials are frequently used in the sodium form to exchange divalent and trivalent ions in the influent water for sodium ions on the resin sites. This process is commonly referred to as *softening* water since it removes those ions that form a "hard" curd of insoluble salts with the fatty acids used in some soaps and that also precipitate when water is boiled. In such a process, sodium chloride is used as the regenerant to return the cation-exchanging groups to the sodium form.
- 9.2 This test method is intended to simulate the performance of such materials in actual usage. It may be used either to compare the performance of a material that has been used with its original performance.
- 9.3 Regenerant concentrations and dosages used herein are typical for the types of materials used in this application. If different concentrations or amounts of regenerant are agreed upon by parties using this test method, this fact should be stated when the results are reported. Similarly, the test water specified is the agreed upon standard. Where other test waters or the water to be treated are used in the test, the analysis of the water in terms of total solids, sodium, calcium, magnesium, other di- or trivalent metals as well as the major anions present should be reported with the test results.

## 10. Reagents and Materials

- 10.1 Brine Regenerants:
- 10.1.1 For synthetic organic ion-exchange materials:
- 10.1.1.1 <u>Sodium Chloride (100 g/L)—Sodium Chloride Dissolve (100 g/L)—Dissolve enough sodium chloride (NaCl) in water to make a solution containing in each litre 100.0 g of NaCl.</u>
  - 10.1.2 For all other ion-exchange materials:
- 10.1.2.1 <u>Sodium Chloride (50 g/L)—Sodium Chloride Dissolve (50 g/L)—Dissolve</u> enough NaCl in water to make a solution containing in each litre 50.0 g of NaCl.
- 10.2 Cation-Exchange Test Water A (10 meq/L)—Dissolve enough calcium chloride ( $CaCl_2 \cdot 2H_2O$ ) and magnesium sulfate (MgSO<sub>4</sub>·7H<sub>2</sub>O) in water to make a solution containing, in each litre, 0.49 g of  $CaCl_2 \cdot 2H_2O$  and 0.415 g of MgSO<sub>4</sub>·7H<sub>2</sub>O. Adjust the pH to 7.5 by the addition of Na<sub>2</sub>CO<sub>3</sub> (30 g/L) and determine the hardness of the solution in accordance with Test Method D1126. The hardness of the test water will be 10.0  $\pm$  0.5 meq/L. Use the determined hardness in calculating operating capacity as indicated in 12.1. This test water shall be used for all tests.
- 10.3 *Hardness Test Reagents*—For reagents used in determining hardness, refer to Test Method D1126. This reagent is used only in preparation of test water (see 10.2).
  - 10.4 Sodium Carbonate Solution (30 g/L)—Dissolve 30 g of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) in water and dilute to 1 L.

<sup>&</sup>lt;sup>3</sup> Reagent Chemicals, American Chemical Society Specifications, 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.



#### 11. Procedure

- 11.1 Adjust the temperature of the water and all solutions to be used in this procedure to  $25 \pm 5^{\circ}$ C and maintain this temperature throughout the test.
- 11.2 Fill the column approximately half full of water and add sufficient sample to give a bed height of  $750 \pm 75$  mm 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 deep above the top of the bed at all times during the procedure.
- 11.3 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 11.4. If the 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.
- 11.4 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 above the top of the bed. Do not jar. Record the volume, in millilitres, of ion-exchange material. Repeat the 10-min backwash until two successive readings of volume agree within 5 mL. The average of these two readings shall be the sample volume for new materials shipped in the sodium form.
- 11.5 Exhaust the ion exchanger with cation-exchange test water A at a flow rate of 0.33 mL/min/mL of exchanger, as measured in 11.4. Maintain a head of liquid not less than 50 mm above the top of the bed. Continue the run until the effluent shows 0.2 meq/L (or other agreed-upon hardness level) when tested in accordance with Test Method D1126. Record the volume of test water used.
- 11.6 Repeat the 10-min backwash and drain as described in 11.3 and 11.4. When testing new material shipped in the sodium form, only one backwash is necessary at this point because a determination of volume has already been made. However, used material other than in the sodium form must have a volume determination made here as described in 11.4. Use this sample volume determined on the exhausted material in calculating the capacity of used ion-exchange materials.
- 11.7 Determine the amount of brine regenerant and rate required, from Table 1. For use with Table 1, the volume sample for new material shall be that determined in accordance with 11.4 and for used material shall be that determined in accordance with 11.6.
- 11.8 Pass the specified volume of brine regenerant through the bed at the specified rate until only a 20 to 30-mm layer of liquid remains above the bed. Rinse the bed with water, using the same rate, until one bed-volume of liquid has been displaced. Increase the rinse rate to approximately 100 mL/min. Test for hardness at 3-min intervals by adding 0.5 mL of buffer solution to 50 mL of the effluent followed by three drops of hardness indicator and 0.5 mL of sodium ethylendiamine tetraacetate solution (1 mL = 1.0 mg CaCO<sub>3</sub>), with stirring. If a blue color develops, the effluent contains 0.2 meq/L or less hardness and the rinse is completed. If the color is red, the end point has not been reached. Continue the rinse until the effluent shows 0.2 meq/L or less hardness.
  - 11.9 Repeat the service run described in 11.5.
- 11.10 Repeat the cycle, beginning with a single backwash (see 11.6), omitting the determination of bed volume. Continue with a regeneration and rinse (see 11.8), and end with a service run (see 11.5). Repeat the cycle until each of three successive runs agrees within  $\pm 5$  % of their average capacity as calculated in accordance with Section 12.

#### 12. Calculation

12.1 Calculate the operating capacity, in milliequivalents per millilitre, of the ion exchange material as-as:

capacity, meq/mL =  $(A \times B)/S$ 

#### where:

 $A = \frac{\text{hardness of test water, meq/L}}{\text{.}}$ 

B = volume of test water used in service run, L, and

S = volume of sample in the bed, mL. For new materials, this refers to the average volume of the material in the sodium form as determined in 11.4. For used materials, it is the average volume of the material in the exhausted form as determined in 11.6.

capacity, meq/mL =  $(A \times B)/S$ 

TABLE 1 Amount of Brine Regenerant Required for Use in Test Method A

Type of Exchange	Brine Regenerant,	Rate of Flow, mL	Contact Time,	Regeneration Level	
Material	<del>g/L</del>	brine/min/mL of Exchanger	<del>min</del>		
	g/L	mL brine/min/mL of Exchanger	<u>min</u>	lb/ft <sup>3</sup>	g/L
Synthetic organic	100	0.032	30	6.00	96.1
Greensand	50	0.027	15	1.25	20.0
Synthetic siliceous	50	0.080	20	3.00	80.1
Carbonaceous	50	0.067	15	3.15	50.5