



Designation: D 4266 – 96 (Reapproved 2007)

## Standard Test Methods for Precoat Capacity of Powdered Ion-Exchange Resins<sup>1</sup>

This standard is issued under the fixed designation D 4266; 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 These test methods cover the determination of the operating ion-exchange capacity of both powdered cation-exchange resins (hydrogen form) and powdered anion-exchange resins (hydroxide form). These test methods are intended for use in testing new powdered ion-exchange resins when used for the treatment of water. The following two test methods are included:

	Sections
Test Method A—Operating Capacity, Anion-Exchange Resin, Hydroxide Form	7 to 15
Test Method B—Operating Capacity, Cation-Exchange Resin, Hydrogen Form	16 to 24

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.*

### 2. Referenced Documents

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

- D 1125 Test Methods for Electrical Conductivity and Resistivity of Water
- D 1129 Terminology Relating to Water
- D 1193 Specification for Reagent Water
- D 2687 Practices for Sampling Particulate Ion-Exchange Materials
- D 4456 Test Methods for Physical and Chemical Properties of Powdered Ion Exchange Resins
- E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

<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.

Current edition approved Dec. 1, 2007. Published January 2008. Originally approved in 1983. Last previous edition approved in 2001 as D 4266 – 96 (2001).

<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. Terminology

#### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *powdered ion-exchange material, n*—an ion-exchange resin that has undergone post-manufacturing size reduction to less than 300  $\mu\text{m}$ .

3.1.2 *resin dosage, n*—the weight of mixed resin applied per unit area of precoatable filter surface. This is expressed as dry pounds per square foot.

3.1.3 *resin floc, n*—that voluminous aggregate formed when powdered anion-exchange resin and powdered cation-exchange resin are slurried together in an aqueous suspension.

3.1.4 *resin ratio, n*—the ratio of the weights of powdered cation-exchange resin to powdered anion-exchange resin used to prepare a resin slurry. If not otherwise indicated, it is understood to be the ratio of the dry resin weights.

3.2 *Definitions*—For definitions of other terms used in these test methods, refer to Terminology D 1129.

### 4. Significance and Use

4.1 The salt removal capacity of a powdered resin precoat is limited by the capacity of either the anion-exchange resin or the cation-exchange resin contained in it. Applications include condensate polishing in fossil-fueled electric generating plants, as well as condensate polishing, spent fuel pool water treatment, reactor water treatment, and low-level radioactive liquid waste treatment in nuclear-powered electric generating plants.

4.2 By determining the ion-exchange capacity profile of either a cation exchange resin or an anion-exchange resin (capacity expended per unit of time under specific conditions), it is possible to estimate runlength and remaining capacity when treating a liquid of the same makeup. Although they cannot accurately predict performance during condenser leaks, these test methods are useful for determining operating capacities as measured under the test conditions used.

4.3 These test methods may be used to monitor the performance of either powdered anion-exchange resin or powdered cation-exchange resin. The total capacity of either resin depends primarily upon the number density of ion-exchange sites within the resin. The operating capacity is a function of the total capacity, degree of conversion to the desired ionic form when received, and properties of the resin and the system that affect ion exchange kinetics.

## 5. Purity of Reagents

5.1 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.<sup>3</sup>

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water, Specification **D 1193**.

## 6. Sampling

6.1 Obtain a representative sample of the powdered ion-exchange resin in accordance with Practices **D 2687** but substituting a 12.5-mm (½-in.) inside diameter tube.

### TEST METHOD A—OPERATING CAPACITY, ANION-EXCHANGE RESIN, HYDROXIDE FORM

## 7. Scope

7.1 This test method covers the determination of ion-exchange capacity, on a dry weight basis, of new powdered anion-exchange resins in the hydroxide form.

7.2 The ion-exchange capacity obtainable in commercial installations depends not only upon the initial state of the powdered resin, but also on how the resin floc is prepared and applied, on the condition of the equipment on which it is to be used, and the pH and general chemistry of the water system being treated. Thus, this test method has comparative rather than predictive value and provides an upper limit on exchange capacity that may be expected.

## 8. Summary of Test Method

8.1 The powdered anion-exchange resin to be tested is slurried with an appropriate amount of powdered cation-exchange resin in the hydrogen form, and the resulting floc is precoated onto a filter disk. Then a dilute standardized solution of a strong acid is fed to the precoat while monitoring the effluent stream conductometrically.

## 9. Apparatus

9.1 Test apparatus, as shown in **Fig. 1**, with the following components:

9.1.1 *Water Pump*—adjustable between 0 to 7.57 L/min (0 to 2 gal/min) at  $2.76 \times 10^5$  Pa (40 psig) pressure.<sup>4</sup>

9.1.2 *Pressure Gages* (two), 0 to  $4.137 \times 10^5$  Pa (0 to 60 psig) with appropriate snubbers.

<sup>3</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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>4</sup> The sole source of supply of the Millipore pump (ZPN100400) apparatus known to the committee at this time is the Millipore Corporation, 290 Concord Rd., Billerica, MA 01821. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

9.1.3 *Disk Filter Holder*, 142-mm diameter with sufficient clearance above the filter disk to allow for uniform application of resin precoat.<sup>5</sup>

9.1.4 *Filter-Disk*, 142-mm diameter, with nominal retention rating of 25 to 30 μm and absolute retention rating of 40 to 60 μm.<sup>6</sup>

9.1.5 *Flow Metre*, 0 to 1.89 L/min (0 to 30 gal/h) with regulating valve.

9.1.6 *Beaker*, stainless steel, 4 L to volume with bulkhead fittings installed at tubing penetrations.

9.1.7 *Chemical Pump*, with pumping rate between  $8.33 \times 10^{-6}$  and  $8.33 \times 10^{-5}$  L/s (30 to 300 mL/h) at  $3.45 \times 10^6$  Pa (500 psig) pressure. Suction tubing should be 3.2-mm (⅛-in.) outside diameter stainless steel and discharge tubing should be 1.6-mm (⅙-in.) outside diameter stainless steel.<sup>7</sup>

9.2 *Electrical Conductivity Measurement Apparatus*, conforming to the requirements given in Test Methods **D 1125**, Method B.

## 10. Reagents

10.1 *Hydrochloric Acid Solution, Standard (0.10 N)*—Prepare and standardize as described in Practice **E 200**.

10.2 *Polyacrylic Acid Solution, Standard (1+99)*—Pipet 1 mL of polyacrylic acid<sup>8</sup> (25 weight % solids, MW <50 000) into a 100 mL volumetric flask and dilute to 100 mL with water. Mix well. Prepare this solution fresh daily.

## 11. Sample Preparation

11.1 *Selection of Proper Sample Weight*—Use a resin dosage of 1 kg/m<sup>2</sup> (0.2 lb/ft<sup>2</sup>) and a resin ratio of 2+1.

11.1.1 If the purpose of the capacity test is to eliminate the resin as a consideration in a situation involving a performance problem in a commercial plant, then the capacity test may be performed using the same *wet* resin ratio and the same resin dosage as is used in the commercial equipment.

11.1.2 Using a resin dosage of 1 kg/m<sup>2</sup> (0.2 lb/ft<sup>2</sup>), the correct dry weight of resin to be used on a 142-mm diameter filter is 15.5 g. At a resin ratio of 2+1, the dry weights to use

<sup>5</sup> The sole source of supply of the Millipore filter holder (YY2214230) with acrylic cylinder (XX4214201) and accessories apparatus known to the committee at this time is the Millipore Corporation, 290 Concord Rd., Billerica, MA 01821. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>6</sup> The sole source of supply of the BG or DG filter apparatus known to the committee at this time is the Pall Corporation, 30 Sea Cliff Ave., Glen Cove, NY 11542. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>7</sup> The sole source of supply of the Milton Roy pump (1960066002) apparatus known to the committee at this time is Milton Roy USA, 201 Ivyland Rd., Ivyland, PA 18974. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>8</sup> The sole source of supply of the Accumer (1510) apparatus known to the committee at this time is the Rohm and Haas Company, 100 Independence Mall West, Philadelphia, PA 19106. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.