Designation: D4266 − 17

Standard Test Methods for Precoat Capacity of Powdered Ion-Exchange Resins[1]

This standard is issued under the fixed designation D4266; 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 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.*

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:*[2]

D1125 Test Methods for Electrical Conductivity and Resistivity of Water
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2687 Practices for Sampling Particulate Ion-Exchange Materials
D4456 Test Methods for Physical and Chemical Properties of Powdered Ion Exchange Resins
D5391 Test Method for Electrical Conductivity and Resistivity of a Flowing High Purity Water Sample
E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of other terms used in this standard, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *powdered ion-exchange material, n*—an ion-exchange resin that has undergone post-manufacturing size reduction to less than 300 µm.

3.2.2 *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.

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

3.2.4 *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.

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,

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[1] 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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[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.

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.[3]

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water, Specification D1193.

6. Sampling

6.1 Obtain a representative sample of the powdered ion-exchange resin in accordance with Practices D2687 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 2.76×10^5 Pa (40 psig) pressure.[4]

9.1.2 *Pressure Gauges* (two), 0 to 4.137×10^5 Pa (0 to 60 psig) with appropriate snubbers.

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

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

9.1.5 *Flow Meter,* 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 8.33×10^{-6} and 8.33×10^{-5} L/s (30 to 300 mL/h) at 3.45×10 $_6$ 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 (1⁄16-in.) outside diameter stainless steel.[7]

9.2 *Electrical Conductivity Measurement Apparatus,* conforming to the requirements given in Test Methods D1125, Test Method B, and D5391.

10. Reagents

10.1 *Hydrochloric Acid Solution, Standard (0.10 N)*—Prepare and standardize as described in Practice E200.

10.2 *Polyacrylic Acid Solution, Standard* (1+99)—Pipet 1 mL of polyacrylic acid[8] (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.

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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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

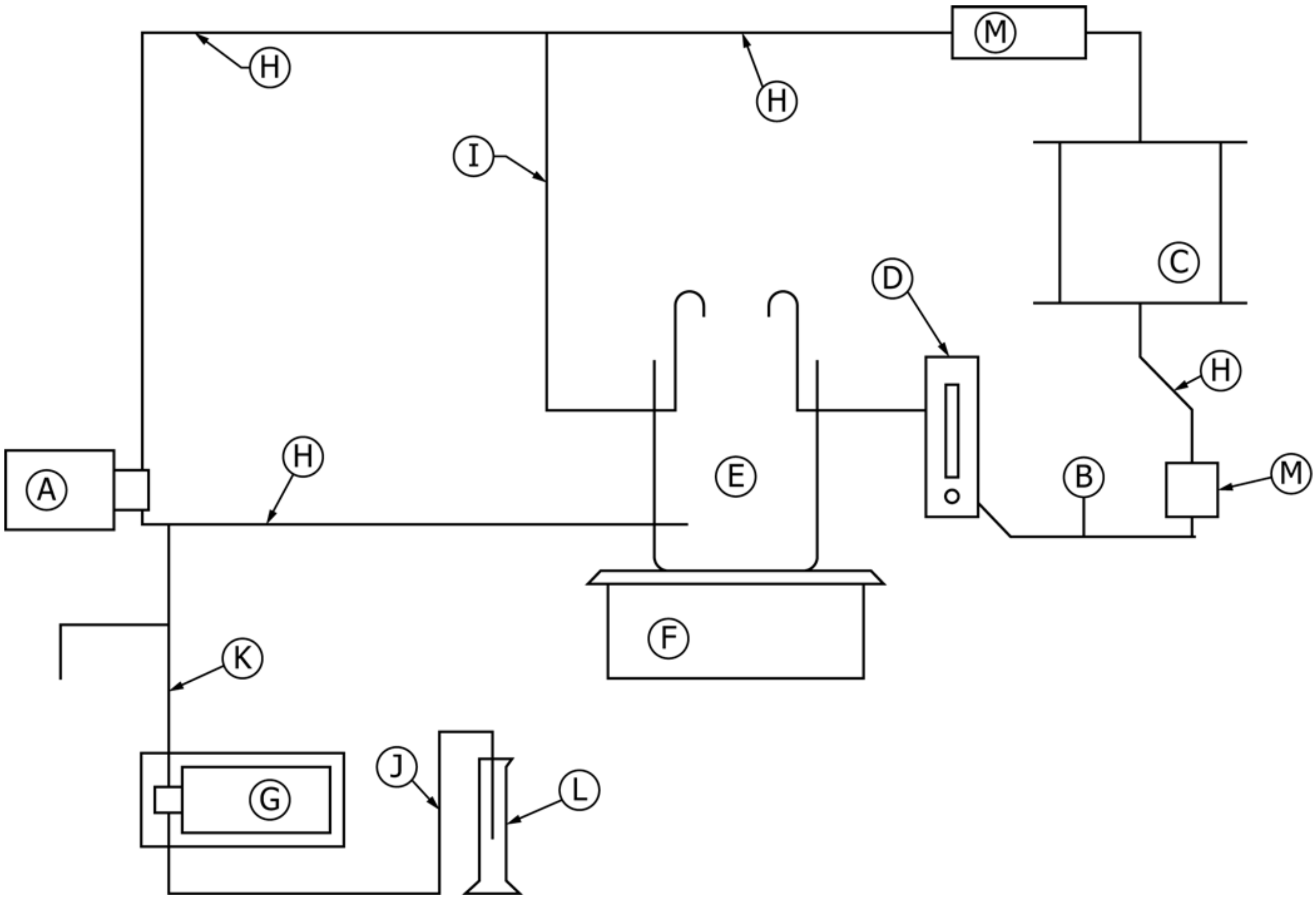
[4] The sole source of supply of the Millipore pump (ZPN100400) apparatus known to the committee at this time is the Millipore Corporation, 290 Concord Rd., Billerica, MA 01821. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments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

[5] 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,[1] which you may attend.

[6] 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,[1] which you may attend.

[7] 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,[1] which you may attend.

[8] 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,[1] which you may attend.



(A)	*Pump,* adjustable between 0 to 2 gal/min at 40 psig (0.27 MPa) pressure. Millipore pump (ZPN100400) has been found satisfactory for this use.
(B)	*Gauges,* 0 to 60 psig (0.41 Pa) pressure with appropriate snubbers.
(C)	*Disk filter holder,* 142-mm diameter with sufficient clearance above the filter disk to allow for a ½ in. (12.5 mm) powdered resin precoat. Millipore filter holder (YY2214230) with acrylic cylinder (XX4214201) and accessories has been found satisfactory for this use.
(D)	*Flow metre,* 0 to 30 gal/h with regulating valve.
(E)	*Stainless steel beaker,* 4-L volume with bulkhead fittings installed at the tubing penetrations.
(F)	*Magnetic stirrer and hotplate.*
(G)	*Instrument Pump,* with pumping rate between 30–300 mL/h at 100-psig pressure. Provide external pressure relief protection set for 100 psig. Milton Roy pump (1960066002) has been found satisfactory for this use.
(H)	*Tubing,* ⅜ in. (9.5 mm), precoat circuit.
(I)	*Tubing,* ¼ in. (6.4 mm), including regulating valve.
(J)	*Tubing,* ⅛ in. (3.2 mm).
(K)	*Tubing,* 1⁄16 in (1.6 mm), including three-way ball valve and flushing line.
(L)	*Graduated Cylinder,* 250 mL.

Disk Filters, 142 mm, either polypropylene or cellulose.
Nominal rating 25–30 μm.
Absolute rating 40–60 μm.
BG or DG filters have been found satisfactory for this use.

(M)	Electrical conductivity cell and measurement apparatus, including temperature measurement.

FIG. 1 Equipment Layout for Precoat Capacity Determination

11. Sample Preparation

11.1 *Selection of Proper Sample Weight*—Use a resin dosage of 1 kg/m^2 (0.2 lb/ft^2) 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^2 (0.2 lb/ft^2), 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 are 10.3 g of cation and 5.2 g of anion exchange resins. The solids contents should be determined by Test Method B of Test Methods D4456.

11.1.3 From the known solids content of the resins, and the dry weight of resin desired for the test, calculate the weight of wet resin to be taken for analysis as follows:

$$W = (B/S) \times 100$$

where:

W = weight of wet resin, g,
B = weight of dry resin, g, and
S = solids content of resin, %.

11.2 *Resin Slurry Preparation:*

11.2.1 Measure 300 mL of water into each of three 500-mL beakers and place each beaker on a magnetic stirrer. Adjust the stirring speed to as high as possible without appreciable vortex formation.

11.2.2 Accurately weigh three portions each of the cation resin and the anion resin, using the wet weights as determined in 11.1. Place the proper amount of cation resin and anion resin in each of the three beakers and stir gently for 5 min.

11.2.3 For each beaker, after mixing for five min, turn off the magnetic stirrer and allow the floc to settle for 5 min. The desired floc density is obtained if the top surface of the settled floc appears flat and uniform.

11.2.4 If the desired settled floc density is not obtained after the initial 5 min settling time, turn the magnetic stirrer on and add 1 mL of the polyacrylic acid solution. Mix for 5 min, allow the floc to settle an additional 5 min, and check the settled floc density again. Continue with 1 mL incremental additions of the polyacrylic acid solution until the desired floc density is obtained. Use the minimum required amount of polyacrylic acid solution. The function of the polyacrylic acid is to partially declump the floc to ensure optimum precoatability of the slurry. The addition of an insufficient amount of polyacrylic acid will result in a floc that is too voluminous and precoating difficulties and too porous a precoat will result. Conversely, an excess of polyacrylic acid solution will declump the floc excessively, and may result in the release of resin fines, observed as turbidity, that tend to foul the precoat filter.

12. Procedure

12.1 Install a 142-mm diameter disk filter (C) into the filter holder and arrange the apparatus as shown in Fig. 1.[6]

12.2 Add water to the beaker (E) as required to maintain a ¾-full level.

12.3 Open the filter inlet sample valve, the flowmeter regulating valve, and the filter holder vent valve.

12.4 Start the water pump (A) and fill and vent the filter disk holder and tubing from the beaker. Close the filter vent valve and the filter inlet valve when full. Further additions of water to the beaker may be required during the filling and venting procedures.

12.5 Add 0.10 *N* HCl to approximately 5 mL above the 250-mL mark in a graduated cylinder (L).

12.6 Adjust the chemical pump (G) to ½-full stroke, open the three-way valve to the vent position and operate the pump to vent the pump and tubing. Shut off the pump when vented and adjust the volume of 0.10 *N* HCl to exactly 250 mL in the graduated cylinder, if required. Adjust the three-way valve to feed into the filter circulation pump suction.

12.7 Maintain the filter circulation flow rate at 1.40 ± 0.05 L/min (22 ± 0.5 gal/h) and maintain the filter inlet pressure at 2.07 Pa (30 psig) by adjusting the water pump output and the flowmeter regulating valve (D).

12.8 Turn on the magnetic stirrer (F) under the stainless steel beaker and adjust the speed so that minimal vortex formation occurs.

12.9 Transfer the contents of one of the resin slurry beakers into the stainless steel beaker and allow the resin to precoat onto the disk filter. Continue circulation until the water above the filter appears clear.

12.10 Open and adjust the inlet valve. Start the chemical pump and adjust the pump rate to maintain a 25°C conductivity of 50 ± 2µ S/cm at the inlet sample point.

12.11 Monitor and record the filter inlet and outlet conductivities (M), the stream temperature, and volume of 0.10 *N* HCl consumed as a function of time. When the filter outlet conductivity rises to 0.2µ S/cm (25°C), or other desired conductivity endpoint, record the volume of 0.10 *N* HCl consumed, and shut down the chemical pump.

12.12 Turn off the water pump and vent, drain, and clean the filter holder and tubing.

12.13 Repeat 12.1 through 12.12 on each of the remaining two resin slurry samples.

13. Calculation and Interpretation of Results

13.1 For each of the three test runs, calculate the capacity as follows:

$$C = (N \times V)/B$$

where:

C = anion resin capacity, milliequivalents HCl/dry gram anion resin,
N = concentration of HCl used, eq/L,
V = volume of HCl consumed at endpoint, mL, and
B = dry weight of anion resin, g.

14. Report

14.1 Report the average of the capacities for the three tests and the effluent conductivity endpoint used.

15. Precision and Bias

15.1 *Precision*—No statement of precision for this test method can be made since the material is of unknown composition and is prepared, tested, and destroyed in one process. Preparation of this material in sufficient quantity to be subdivided is similarly not possible since the preparation and testing is one procedure.

15.2 *Bias*—Ion exchange resins are the products of a complex, multiple step synthesis involving a polymerization reaction followed by one or more additional reactions to put functional groups on the polymeric structure. Consequently, the true value for any property of the finished product is unknown and a bias statement cannot be given.

TEST METHOD B—OPERATING CAPACITY, CATION-EXCHANGE RESIN, HYDROGEN FORM

16. Scope

16.1 This test method covers the determination of ion-exchange capacity, on a dry weight basis, of new powdered cation-exchange resin in the hydrogen form.

16.2 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 used, and the pH and general chemistry of the water system being treated. Thus, this test method has comparative, rather than predictive, value and gives an upper limit on exchange capacity that may be expected.

17. Summary of Test Method

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

18. Apparatus

18.1 Test apparatus, as described in Section 9 and shown in Fig. 1.

19. Reagents

19.1 *Sodium Hydroxide Solution* (0.10 *N*)—Prepare and standardize as described in Practice E200.

20. Sample Preparation

20.1 *Selection of Proper Sample Weight,* as described in 11.1.

20.2 *Resin Slurry Preparation,* as described in 11.2.

21. Procedure

21.1 The procedure is the same as described in Section 12, except wherever HCl appears, it should be substituted by NaOH and a 750-mL graduated cylinder should be substituted for the 250-mL cylinder in 12.5 and 12.6.

22. Calculation and Interpretation of Results

22.1 For each of the three test runs, calculate the capacity:

$$C = (N \times V)/B$$

where:

C = cation resin capacity, milliequivalents NaOH/dry gram cation resin,
N = concentration of NaOH used, eq/L,
V = volume of NaOH consumed at endpoint, mL, and
B = dry weight of anion resin, g.

23. Report

23.1 Report the average of the capacities for the three tests and the effluent conductivity endpoint used.

24. Precision and Bias

24.1 *Precision*—See 15.1.

24.2 *Bias*—See 15.2.

25. Quality Control

25.1 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 test method does not include a bias statement.

25.2 Analysts are expected to replicate samples to determine if the results are within the expected precision stated in Section 15.

25.3 Analysis of the resin filter effluent is subject to the quality control requirements of the referenced analytical methods.

26. Keywords

26.1 floc; ion exchange; powdered resin