Designation: ~~D2187 – 94 (Reapproved 2009)$^{\varepsilon 1}$~~ D2187 – 17

Standard Test Methods and Practices for Evaluating Physical and Chemical Properties of Particulate Ion-Exchange Resins[1]

This standard is issued under the fixed designation D2187; 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.

~~ε^1 NOTE—A typo was editorially corrected in Section 47.7 in March 2010.~~

1. Scope

1.1 These test methods cover the determination of the physical and chemical properties of ion-exchange resins when used for the treatment of water. They are intended for use in testing both new and used materials. The following thirteen test methods are included:

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[1] These test methods and practices 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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1.2 The values stated 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.* Specific precautionary statements are given in Section 10.8.

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]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D1293 Test Methods for pH of Water
D2687 Practices for Sampling Particulate Ion-Exchange Materials
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

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 *anion-exchange material*—an ion-exchange material capable of the reversible exchange of negatively charged ions.

3.2.2 *cation-exchange material*—an ion-exchange material capable of the reversible exchange of positively charged ions.

3.2.3 *ion-exchange resin*—a synthetic organic ion-exchange material.

3.2.4 *mixed bed*—a physical mixture of anion-exchange material and cation-exchange material.

4. Reagents

4.1 *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 such specifications are available.[3] 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type IV reagent water described in Specification D1193.

5. Sampling

5.1 Obtain a representative sample of the ion-exchange resin in accordance with Practices D2687.

5.2 A minimum sample size of 1 L is recommended for a complete testing program.

TEST ~~METHOD~~PRACTICE A—PRETREATMENT

~~6. Scope~~

~~6.1 This test method covers the conversion of ion-exchange resins to a known ionic form and is intended for application to both new and used material.~~

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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.

[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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

6. Scope

6.1 This test practice covers the conversion of ion-exchange resins to a known ionic form and is intended for application to both new and used material.

7. Significance and Use

7.1 The ionic form of an ion-exchange material affects both its equivalent mass and its equilibrium water content. These in turn influence the numerical values obtained in exchange capacity determinations, in density measurements, and in the size of the particles. To provide a uniform basis for comparison, therefore, the sample should be converted to a known ionic form before analysis. This procedure provides for the conversion of cation-exchange materials to the sodium form and anion-exchange materials to the chloride form prior to analysis. These forms are chosen since they permit samples to be weighed and dried without concern for air contamination or decomposition. If other ionic forms are used this fact should be noted in reporting the results.

8. Apparatus

8.1 *Pretreatment Apparatus* ~~(See~~(see Fig. 1):

8.1.1 *Column,* transparent, vertically-supported, 25 ± 2.5 mm (1.0 ± 0.1 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 ~~method~~ test practice can be made. Make all measurements at 25 ± 5°C.

8.1.2 *Support,* for the sample, so designed that the distance from the sample to the column outlet is at least 50 mm. Suggested supports are corrosion-resistant screen or porous plate.

8.2 *Draining Apparatus* (Fig. 2):

8.2.1 *Buchner-Type Funnel,* containing a 125-mm filter paper and supported in a 1-L suction flask.

8.2.2 *Open-Arm Mercury Manometer,* connected by a T-tube to a vacuum train.

8.2.3 *Gas-Humidifying Tower,* of at least 500 mL capacity, two thirds filled with glass beads or similar material.

8.2.4 *Vacuum Pump,* capable of creating a pressure differential 40 mm Hg below atmospheric pressure.

9. Reagents

9.1 *Hydrochloric Acid* (1 + 9)—Carefully pour 100 mL of hydrochloric acid (HCl, sp gr 1.19) into 900 mL of water, stirring constantly. Cool to 25 ± 5°C.

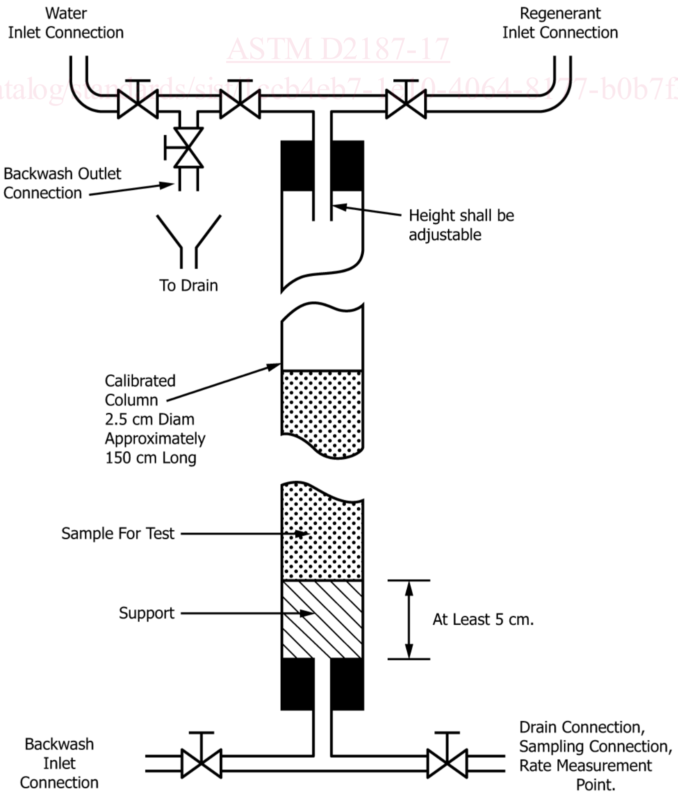

FIG. 1 Typical Arrangement of Apparatus for Pretreatment of Ion-Exchange Materials

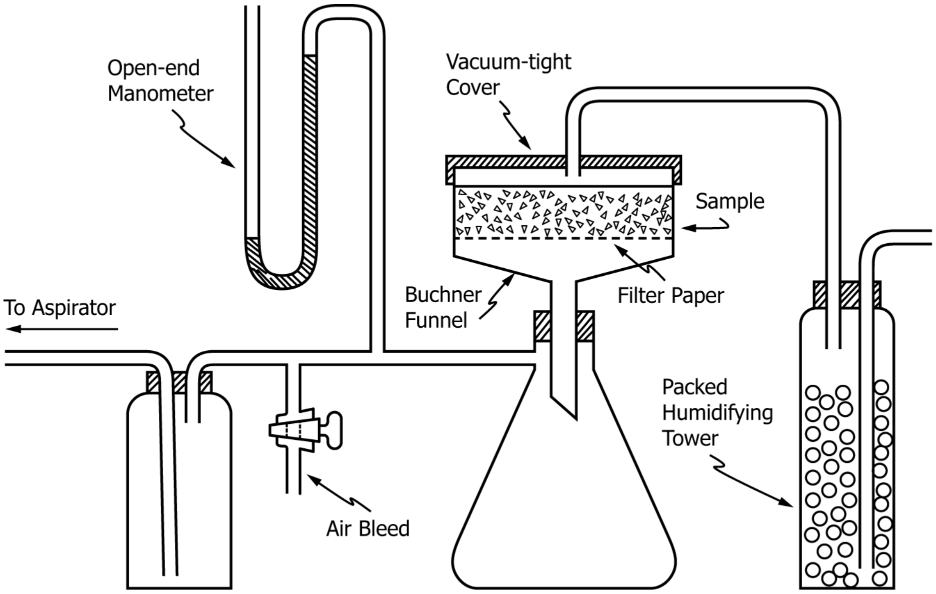

FIG. 2 Typical Arrangement of Water-Draining Apparatus

9.2 *Sodium Chloride Solution* (100 g/L)—Dissolve 100.0 g of sodium chloride (NaCl) in 800 mL of water and dilute to 1 L.

9.3 *Sodium Chloride Solution* (240 g/L)—Dissolve 240 g of sodium chloride (NaCl) in 800 mL of water and dilute to 1 L.

9.4 *Sodium Hydroxide Solution* (40 g/L)—Dissolve 40.0 g of sodium hydroxide (NaOH) in 800 mL of water. Cool and dilute to 1 L.

9.5 *Thymol Blue Indicator Solution*—Dissolve 0.1 g of thymol blue (thymol sulfonphthalein) in 10.75 mL of 0.02 N NaOH solution. Dilute to 250 mL with water.

9.6 *Tropaeolin O Indicator Solution*—Dissolve 0.10 g of tropaeolin O (p-benzene-sulfonic acid-azoresorcinol) in 50 mL of water and dilute to 100 mL in a volumetric flask.

10. Procedure

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

10.2 Transfer the entire sample as received to a 2-L beaker using water to rinse out the container. Adjust the water level to the sample level. Let stand a minimum of 1 h. Mix thoroughly and transfer a representative sample to fill a 400-mL beaker.

10.3 Fill the pretreatment column one half full of water. Transfer the entire contents of the 400-mL beaker to the column using additional water if necessary.

10.4 Backwash with water using a flow rate that will maintain a 50 % expansion of the bed. Adjust the backwash outlet tube to a height above the bed equal to 75 % of the bed height. Continue backwashing for a minimum of 10 min or until the effluent is clear. For mixed bed samples proceed in accordance with 10.5. For single component samples, proceed in accordance with 10.6.

10.5 If the sample is a mixed bed, displace the backwash water from the bed by slowly introducing NaCl solution (100 g/L) at the bottom of the column and allowing it to flow upward through the sample. When the water has been displaced, increase the flow rate until the anion-exchange resin is separated from and suspended above the cation-exchange resin. Lower the backwash outlet tube as required to siphon off the anion-exchange resin, collecting it in a separate pretreatment apparatus. Exercise care to prevent the removal of cation-exchange resin in this operation. When the transfer of the anion-exchange resin is complete, discontinue the flow of NaCl solution. If the separation of anion and cation-exchange resins has not been complete and a mixed band is left in the center, repeat the siphoning procedure to remove this band from the cation-portion of the sample. This mixed material that should not constitute more than 5 % of the original sample volume, is not included in subsequent tests. If more than 5 % of the sample remains unseparated, the separation should be repeated using NaCl solution (240 g/L). In either case proceed with the separated anion and cation components as separate samples as described in 10.6.

10.6 Allow the resin to settle until the liquid level is 20 to 30 mm above the top of the bed, and estimate its volume. Pass NaCl solution (100 g/L) downflow through the single component sample or the separated components of the mixed bed resin at the approximate rate of 0.133 mL/min/mL of sample for 1 h. Discontinue the flow of NaCl solution. Backwash with water for 10 min at a flow rate sufficient to maintain a 50 % expansion of the bed. Discontinue the flow of water.

10.7 Allow the bed to settle and then drain off the water at a rate of approximately 100 mL/min until the water level is 20 to 30 mm above the top of the bed. Estimate the volume of ion-exchange resin in millilitres.

10.8 Determine the amount of reagent and the flow rate required for the initial pretreatment from Table 1 using the sample volume determined in 10.7. (**Warning—**Swelling of the resin in the column may occur in subsequent steps.)

10.9 Pass the specified volume of reagent through the bed at the specified rate until only a 20 or 30 mm layer of liquid remains above the bed. Rinse the bed with two sample volumes of water at the same rate.

10.10 Determine the amount of reagent and the flow rate required for the second pretreatment from Table 2 using the sample volume determined in 10.7. Note that this second pretreatment is not used for some methods.

10.11 Pass the specified volume of reagent through a bed at the specified rate until only a 20 to 30-mm layer of liquid remains above the bed. Rinse the bed with one sample volume of water at the same rate. Increase the rinse rate to 100 mL/min. Rinse for 15 min. Thereafter test successive 100-mL portions of the effluent from anion-exchange resins by adding two drops of thymol blue indicator solution. Continue rinsing until a 100 mL portion of the effluent remains yellow (pH > 2.5) on the addition of the indicator. Test the effluent from the cation-exchange resins in the same manner with two drops of tropaeolin-O indicator solution. Continue rinsing until a 100-mL portion of the effluent remains yellow (pH < 11.0)[3] on the addition of the indicator.

10.12 Remove the ion-exchange resin from the pretreatment column, discarding any extraneous material that may have accumulated at the bottom of the bed. Transfer the resin to the Buchner funnel of the draining apparatus that has been fitted with a medium porosity filter paper. Drain the water to the top of the sample using suction if required. Cover the funnel with a suitable vacuum-tight cover, which is fitted with an inlet for air from the water-filled humidifying tower. Apply sufficient suction to maintain a pressure differential of 40 ± 5 mm Hg below atmospheric pressure. Continue passing humidified air through the sample for 10 min.

10.13 Transfer the entire drained sample to a clean, dry, 1-L (1-qt.), wide-mouthed bottle with a screw top or other vapor-tight closure.

TEST METHOD B—WATER RETENTION CAPACITY

~~11. Scope~~

~~11.1 This test method covers the determination of the amount of water retained by ion-exchange resins and is intended for testing both new and used materials.~~

11. Scope

11.1 This test method covers the determination of the amount of water retained by ion-exchange resins and is intended for testing both new and used materials.

12. Summary of Test Method

12.1 This test method consists of the determination of the loss of mass on drying at 104 ± 2°C.

13. Significance and Use

13.1 The water retention capacity of an ion-exchange material is proportional to its pore volume. For new materials of the same functionality and polymer type, higher values indicate lower effective crosslinking. Increases in water retention capacity of used materials as compared with the values for new material serve as an indicator of polymer decrosslinking: decreases may indicate either loss of functionality or fouling of the ion-exchange material. Since the numerical value is directly dependent on the ionic form of the material, careful preconditioning of both original and used samples to known ionic forms as outlined in Section 7 is essential when such comparisons are made.

TABLE 1 Requirements for Initial Pretreatment

	Anion-Exchange Resins	Cation-Exchange Resins
Reagent	NaOH	HCl
Concentration	40 g/L	1 + 9
Volume required	8 sample volumes	8 sample volumes
Contact time	1 h	1 h
Flow rate, mL/min-mL sample	0.133	0.133
Regeneration level:		
lb/ft ^{3}	20.0	21.2
g/L	320	340

TABLE 2 Requirements for Second Pretreatment

	Anion-Exchange Resins	Cation-Exchange Resins
Reagent	HCl	NaOH
Concentration	1 + 9	40 g/L
Volume required	8 sample volumes	4 sample volumes
Contact time	1 h	0.5 h
Flow rate, mL/min-mL sample	0.133	0.133
Regeneration level:		
lb/ft ^{3}	21.2	10.0
g/L	340	160

14. Procedure

14.1 Weigh three approximately 5-g representative samples of material pretreated in accordance with Section 10 to the nearest 1 mg into previously tared weighing vessels.

14.2 Dry the samples for 18 ± 2 h at 104 ± 2°C.

14.3 Remove the samples from the oven. Cool 30 min in a desiccator, and reweigh.

15. Calculation

15.1 Calculate the water retention capacity, in percent, as follows:
~~Calculate the water retention capacity, in percent, as follows:~~

$$\text{water retained, } \% = \left[(A - B)/A \right] \times 100 \tag{1}$$

where:

A = amount of wet sample used, g, and
B = amount of dry sample obtained, g.

16. Report

16.1 Report the percent water retained as the average of the three values obtained.

17. Precision and Bias[4]

17.1 *Precision*—The precision of this test method of determining water retention capacity of ion exchange resins may be expressed as follows:
~~The precision of this test method of determining water retention capacity of ion exchange resins may be expressed as follows:~~

$$S_T = 0.017x$$

$$S_o = 0.004x$$

where:

S_T = overall precision,
S_o = single-operator precision, and
x = water retention capacity determined in percent.

17.1.1 Information given for the precision statement is derived from round robin testing in which eight laboratories, including ten operators, participated. Four samples were included in the testing. The range of water retention capacity in the samples tested was 40 to 60 %.

17.2 *Bias*—Ion exchange resins are the product of a complex, multiple step synthesis involving a polymerization reaction followed by one or more additional reactions to place 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.

18. Quality Control

18.1 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 this test method does not include a bias statement.

18.2 Analysts are expected to use replicate samples to determine if the results are within the expected precision stated in Section 17.

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports ~~RR:D19-139~~RR:D19-0139 and RR:D19-1007. Contact ASTM Customer Service at service@astm.org.

TEST METHOD C—BACKWASHED AND SETTLED DENSITY

~~18. Scope~~

~~18.1 This test method covers the determination of the backwashed and settled density of ion-exchange resin and is intended for testing both new and used material.~~

19. Scope

19.1 This test method covers the determination of the backwashed and settled density of ion-exchange resin and is intended for testing both new and used material.

20. Summary of Test Method

20.1 The test method consists of the determination of the backwashed and settled volume of a known number of grams of chemically pretreated resin.

21. Significance and Use

21.1 This test method for the determination of backwashed and settled density of a hydraulically classified and settled bed was developed to correlate with the density of ion-exchange materials in operating units. Results obtained by this test method in a 25-mm (1-in.) column may be expected to agree with those obtained in larger diameter units within the over-all precision limits of the test, but the bias of these results, as compared with measurements in larger diameters, is toward lower values.

22. Procedure

22.1 Weigh a 200-g sample of resin, pretreated in accordance with Section 10, to the nearest 0.1 g. Transfer it quantitatively to a column that has been calibrated every 5 mL above the 200-mL volume.

22.2 Backwash with water for 10 min using a slow rate that will maintain a 50 % expansion of the bed.

22.3 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 resin. Repeat the 10-min backwash until two successive readings of volume agree within 5 mL.

23. Calculation

23.1 Calculate the backwashed and settled density, in grams per millilitre as follows:
~~Calculate the backwashed and settled density, in grams per millilitre as follows:~~

$$\text{density, g/mL} = A/B \tag{2}$$

where:

A = amount of sample used, g, and
~~B = volume of sample from 21.3, mL.~~
B = volume of sample from 22.3, mL.

23.2 Calculate the backwashed and settled density in pounds (grams) per cubic foot, as follows:
~~Calculate the backwashed and settled density in pounds (grams) per cubic foot, as follows:~~

$$\text{density, lb/ft}^3\ (\text{g/ft}^3) = C \times 62.4 \tag{3}$$

where:

C = density, g/mL.

24. Report

24.1 Report the density of the tested material as the average of that calculated from two volumes that agree within 5 mL.

25. Precision and Bias[4]

25.1 *Precision*—The precision of this test method of determining backwashed and settled density of ion exchange resins may be expressed as follows:
~~The precision of this test method of determining backwashed and settled density of ion exchange resins may be expressed as follows:~~

$$S_T = 0.035x$$
$$S_o = 0.005x$$

where:

S_T = overall precision,

S_o = single-operator precision, and
x = density determined in g/mL.

25.1.1 Information given for the precision statement is derived from round robin testing in which eight laboratories, including ten operators, participated. Four samples were included in the testing. Six of the operators ran each sample in duplicate. The remainder were single observations.

25.2 *Bias*—Ion exchange resins are the product of a complex, multiple step synthesis involving a polymerization reaction followed by one or more additional reactions to place 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.

26. Quality Control

26.1 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 this test method does not include a bias statement.

26.2 Analysts are expected to use replicate samples to determine if the results are within the expected precision stated in Section 25.

TEST METHOD D—PARTICLE SIZE DISTRIBUTION

~~25. Scope~~

~~25.1 This test method covers the wet sieve analysis of ion-exchange materials.~~

27. Scope

27.1 This test method covers the wet sieve analysis of ion-exchange materials.

28. Summary of Test Method

28.1 This test method consists of hand-sieving the chemically pretreated resin in water through a series of standard sieves of progressively decreasing size of opening. The volume retained on each of the sieves is measured.

29. Significance and Use

29.1 The particle size distribution of ion-exchange materials is determined in the fully-hydrated state and in known ionic form to provide a reproducible base for comparison of changes in size due to particle breakage in use.

30. Apparatus

30.1 *Sieves,* 203 mm (8 in.) in diameter, conforming to Specification E11. A suitable series of such sieves consists of U.S. Standard Sieves Numbers 8 (2.36-mm), 12 (1.70-mm), 16 (1.18-mm), 20 (850-µm), 30 (600-µm), 40 (425-µm), 50 (300-µm), 70 (212-µm), and 100 (150-µm).

30.2 *Water Bath,* minimum diameter 305 mm (12 in.); minimum depth, 152 mm (6 in.).

31. Procedure

31.1 Add sufficient water to the water bath to fill it to the level of the top rim of a sieve placed on the bottom of it.

31.2 Fill a 100-mL beaker with a representative portion of the sample pretreated in accordance with Section 10.

31.3 Transfer the entire sample onto the sieve with the largest mesh opening using water as required.

31.4 Gently raise and lower the sieve through the water interface in the bath so as to alternately lift the particles on the sieve and float them off again. Exercise care that none of the material on the sieve is floated over the edge. Repeat the operation until no further material passes through the screen.

31.5 Remove the sieve from the water bath. Transfer the particles in the bath quantitatively to a suitably-sized beaker.

31.6 Invert the sieve containing the ion-exchange material in the bath and wash the material from the openings with water. Remove the sieve and transfer the particles quantitatively to a suitable-sized graduated cylinder. Tap the material collected in the graduated cylinder until a constant volume is obtained. Record this volume in millilitres.

31.7 Place the sieve of next smaller mesh opening in the bath. Pour the particles that passed the first sieve onto it and adjust the bath level as described in ~~29.1~~31.1. Repeat the operation described in ~~29.4~~31.4 to ~~29.6~~31.6 using this smaller mesh sieve.

31.8 Repeat the sieving operation with sieves of progressively smaller mesh size until all the sieves in the series have been used. After the final sieving, collect and record the volume of any material remaining in the bath.

32. Calculation

32.1 Calculate the percentage of ion-exchange material retained on each sieve as follows:
~~Calculate the percentage of ion-exchange material retained on each sieve as follows:~~

$$\text{volume retained, \%} = 100X/\sum \quad (4)$$

where:

X = amount of material retained on a particular sieve, mL, and
$\sum$ = summation of all volumes retained by the sieves used, plus the volume passing the smallest sieve, mL.

32.2 Calculate the cumulative percent retained on each sieve by adding to the percentage retained on it the percentages retained on all of the sieves used having larger mesh openings. For example: in a series where U.S. Standard Sieves Nos. 8, 12, 16, 20, 30, 40, 50, 70, and 100 have been used, the cumulative percent retained on No. 16 equals:

~~percent retained on No. 8 + percent retained on No. 12 + percent retained on No. 16~~

percent retained on No. 8 + percent retained on No. 12 + percent retained on No. 16

32.3 Using normal probability paper, plot the cumulative percent retained on each sieve on the probability axis as a function of the sieve opening in millimetres on the linear axis. Draw the best straight line through the points giving greater weight to the points representing the largest resin fractions.

32.4 On the line drawn as described in ~~30.3~~32.3, determine the sieve openings that will retain 40 and 90 % of the sample. The sieve opening in millimetres that will retain 90 % of the sample is the effective size of that sample.

32.5 Calculate the uniformity coefficient of the sample as follows:
~~Calculate the uniformity coefficient of the sample as follows:~~

$$\text{uniformity coefficient} = \frac{\text{mesh size (mm) retaining 40\% of the sample}}{\text{mesh size (mm) retaining 90\% of the sample}} \quad (5)$$

33. Report

33.1 Report the numbers of the sieves used, and the cumulative percent retained on each. Report also the effective size and the uniformity coefficient.

34. Precision and Bias[4]

34.1 *Precision*—The precision for this test method of determining particle size distribution and uniformity coefficient of ion exchange resins may be expressed as follows:
34.1.1 *Spheroidal Materials:*

~~$S_T = 0.032$ (for effective size)~~

$$S_T = 0.061 \text{ (for uniformity coefficient)}$$

and
34.1.2 *Granular Materials:*

$$S_T = 0.05 \text{ (for effective size)}$$

$$S_T = 0.157 \text{ (for uniformity coefficient)}$$

where:

~~S_T = overall precision in millimetres for effective size, and a dimensionless unit for uniformity coefficient~~

S_T = overall precision in millimetres for effective size, and a dimensionless unit for uniformity coefficient.

34.1.3 Information given for the precision statement is derived from round robin testing in which eight laboratories, including ten operators, participated. Four samples were included in the testing, and of these, three were spherically shaped and one was granular. All tests were single observations.

34.2 *Bias*—Ion exchange resins are the product of a complex, multiple step synthesis involving a polymerization reaction followed by one or more additional reactions to place 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.

35. Quality Control

35.1 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 this test method does not include a bias statement.

35.2 Analysts are expected to use replicate samples to determine if the results are within the expected precision stated in Section 34.

TEST METHOD E—SALT-SPLITTING CAPACITY OF CATION EXCHANGE RESINS

~~33. Scope~~

~~33.1 This test method covers the determination of the number of milliequivalents of exchangeable hydrogen in a cation-exchange resin sufficiently acidic to split neutral salts.~~

36. Scope

36.1 This test method covers the determination of the number of milliequivalents of exchangeable hydrogen in a cation-exchange resin sufficiently acidic to split neutral salts.

37. Summary of Test Method

37.1 This test method consists of conversion of the sample to the hydrogen form, elution with sodium chloride solution, followed by titration of the hydrogen ion exchanged in this process.

38. Significance and Use

38.1 This test method is generally assumed to measure only the sulfonic acid groups in ion-exchange materials. It should be pointed out, however, that some phosphonic acid and carboxylic acid groups will also exhibit salt-splitting when tested by this procedure.

39. Apparatus

39.1 *Test Apparatus,* as shown in Fig. 3 shall consist of a filter tube of at least 30-mL capacity having a diameter of at least 20 mm containing a sintered glass plate of coarse (*A*) porosity, a 1-L-separatory funnel and a 1-L volumetric flask.

39.2 *Electrometric pH Measurement Apparatus,* conforming to the requirements given in Section 4 of Test ~~Method~~Methods D1293.

40. Reagents

40.1 *Carbon Dioxide-Free Water*—Prepare carbon dioxide-free water by heating Type II reagent water (see Specification D1193) to boiling in a conical flask. Boil vigorously for 10 min. Stopper with a one-hole rubber stopper fitted with a soda-lime drying tube and cool to 25 ± 5°C.

40.2 *Hydrochloric Acid* (1 + 9)—Carefully pour 100 mL of hydrochloric acid (HCl, sp gr 1.19) into 500 mL of water, stirring constantly. Cool to 25 ± 5°C and dilute to 1 L.

40.3 *Methyl Orange Indicator Solution* (0.5 g/L)—Dissolve 0.05 g of methyl orange in water and dilute to 100 mL with water.

40.4 *Phenolphthalein Indicator Solution* (5.0 g/L)—Dissolve 0.5 g of phenolphthalein in 50 mL of 95 % ethanol (see Note 1). Transfer to a volumetric flask and dilute to 100 mL with water.

NOTE 1—Specifically denatured ethyl alcohol conforming to Formula 3A or 30 of the U.S. Bureau of Internal Revenue may be substituted for 95 % ethyl alcohol.

40.5 *Sodium Chloride Solution* (50 g/L)—Dissolve 50 g of sodium chloride (NaCl) in 800 mL of water and dilute to 1 L.

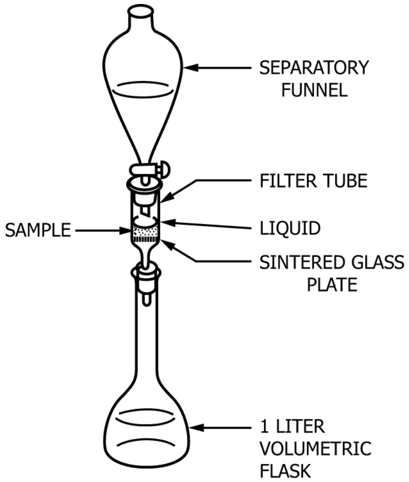

FIG. 3 Typical Arrangement of Apparatus for Salt-Splitting Capacity

40.6 *Sodium Hydroxide Solution*, 50 %—Prepare a saturated solution by dissolving 162 g of sodium hydroxide (NaOH) pellets in 150 mL of carbon dioxide-free water. Cool to 25 ± 5°C and decant the free liquid. Store in a plastic bottle.

40.7 *Sodium Hydroxide Solution Standard* (0.10 *N*)—Measure 5.45 mL or 8.0 g of 50 % sodium hydroxide (NaOH) solution into a 10 mL graduated cylinder. Rinse it into a 1 L volumetric flask with carbon dioxide-free water at 25 ± 5°C, dilute to 1 L with like water and mix well. Standardize monthly.

40.7.1 To standardize, dry approximately 10 g of primary standard grade potassium hydrogen phthalate ($KHC_5H_4O_4$) in a glass container at 120°C for 2 h. Cool in a desiccator. Weigh accurately three 1.00-g samples of the dried potassium hydrogen phthalate and transfer to separate 250-mL conical flasks. Add 100 mL of carbon dioxide-free water and stir gently to dissolve the sample. Titrate with the 0.10 *N* NaOH solution electrometrically to a pH of 8.2 or add two drops of phenolphthalein indicator solution and titrate to the first pink that persists for 15 s with swirling.

40.7.2 Calculate the normality of the NaOH solution as follows:
~~Calculate the normality of the NaOH solution as follows:~~

$$N = B/(0.20423 \times C) \tag{6}$$

where:

N = normality of the NaOH solution,
B = actual amount of $KHC_5H_4O_4$ used, g, and
C = amount of NaOH solution used, mL.

41. Procedure

41.1 Weigh accurately into separate 100-mL beakers, three 10-g representative samples of material pretreated in accordance with Section 10.

41.2 Rinse the weighed samples with water quantitatively into the filter tubes. Fill the separatory funnel with 1 L of HCl (1 + 9). Fill the sample tube with acid and tap to remove air bubbles. Attach the stem of the funnel to the filter tube with a suitable-size rubber stopper. Pass the acid through the sample at a rate of 20 to 25 mL/min, keeping the sample covered with acid at all times. Drain the liquid to the resin level. Discard the effluent.

41.3 Rinse the separatory funnel thoroughly with water. Run water through the acid-treated samples at the rate of 20 to 25 mL/min until the effluent is yellow to methyl orange or has a pH above 3.9. Drain to the resin level and discard the effluent water.

41.4 Position a clean 1-L volumetric flask under the tip of the filter tube. Fill the separatory funnel with 1 L of NaCl solution (50 g/L). Pass the NaCl solution through the sample at a rate of 20 to 25 mL/min keeping the sample covered with solution at all times. Collect the effluent in the volumetric flask. Discontinue the flow of the liquid when 1.0 L has been collected.

41.5 Stopper and mix the NaCl effluent thoroughly. Pipet out three 100-mL portions of each sample of effluent. Add 2 drops of phenolphthalein indicator solution to each and titrate with 0.1 *N* NaOH solution to the first pink color that will persist on 15-s swirling, or titrate electrometrically to a pH of 8.2. Record the volume of NaOH solution used in each titration to the nearest 0.01 mL. Use the average of the three titrations for each sample as *E*.

42. Calculation

42.1 Calculate the salt-splitting capacity in milliequivalents per wet gram as follows:
~~Calculate the salt-splitting capacity in milliequivalents per wet gram as follows:~~

$$\frac{\text{milliequivalents cationic salt} - \text{splitting capacity}}{\text{wet gram}} = (E \times N \times 10)/W \tag{7}$$

$$\frac{\text{milliequivalents cationic salt} - \text{splitting capacity}}{\text{wet gram}} = (E \times N \times 10)/W \tag{7}$$

where:

~~E = average millilitres of NaOH solution required for the titration in 38.5,~~
E = average millilitres of NaOH solution required for the titration in 41.5,
W = wet grams of the sample, and
N = normality of NaOH solution used.

42.2 Calculate the cationic salt-splitting capacity in milliequivalents per dry gram as follows:
~~Calculate the cationic salt-splitting capacity in milliequivalents per dry gram as follows:~~

$$\frac{\text{milliequivalents cationic salt} - \text{splitting capacity}}{\text{dry gram}} = H/(1-(M/100)) \tag{8}$$

$$\frac{\text{milliequivalents cationic salt} - \text{splitting capacity}}{\text{dry gram}} \tag{8}$$

$$= H/(1-(M/100))$$