Designation: D4456 – 17

Standard Test Methods for Physical and Chemical Properties of Powdered Ion Exchange Resins[1]

This standard is issued under the fixed designation D4456; 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 physical and chemical properties of powdered ion exchange resins and are intended for use in testing new materials. The following test methods are included:

	Sections
Test Method A—Particle Size Distribution	5 to 15
Test Method B—Solids Content	16 to 23

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are 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, health and 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:*[2]

D1129 Terminology Relating to Water

D1193 Specification for Reagent 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

F322 Test Method for Determining the Quality of Calibration Particles for Automatic Particle Counters (Withdrawn 1990)[3]

F651 Method for Particle Counter Single-Point Calibration by the Median Method (Withdrawn 1987)[3]

F658 Practice for Calibration of a Liquid-Borne Particle Counter Using an Optical System Based Upon Light Extinction (Withdrawn 2007)[3]

3. Terminology

3.1 *Definitions:*

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 that relate specifically to these standards are described as follows:

3.2.2 *powdered ion exchange resin*—an ion exchange resin that has undergone post-manufacturing size reduction to less than 30 µm.

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

4. Purity of Reagents

4.1 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,[4] where such specifications are available. 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.

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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.

Current edition approved Aug. 1, 2017. Published August 2017. Originally approved in 1985. Last previous edition approved in 2009 as D4456 – 99 (2009)$^{\varepsilon 1}$. DOI: 10.1520/D4456-17.

[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] The last approved version of this historical standard is referenced on www.astm.org.

[4] *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 *Annual 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.

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

TEST METHOD A—PARTICLE SIZE DISTRIBUTION

5. Scope

5.1 This test method covers the instrumental determination of the particle-size distribution of powdered ion exchange resins.

6. Summary of Test Method

6.1 A sample of powdered ion exchange resin is dispersed uniformly in a suitable aqueous liquid. The resulting suspension is passed through an instrument for measuring particle size distribution by weight, number, or volume. Determinations are made of mean particle size, percent below a specified smaller size limit, and percent above a specified larger size limit.

6.2 The analyst should be aware that adequate collaborative data for precision and bias statements as required by Practice D2777 are not provided. See Section 15 for details.

7. Significance and Use

7.1 The particle size distribution of powdered ion exchange resins and, more importantly, the derived parameters of mean particle size and percent above and below specified size limits are useful for determining batch to batch variations and, in some cases, can be related to certain aspects of product performance.

7.2 Although automatic multichannel particle size analyzers, of the type described in Section 9, yield information on the entire distribution of sizes present in a given sample, it has been found that, for this application, the numerical value of three derived parameters may adequately describe the particle size characteristics of the samples: the mean particle diameter (in micrometres), the percent of the sample that falls below some size limit, and the percent of the sample that falls above some size limit.

8. Interferences

8.1 Instruments requiring the use of an aqueous electrolyte for sample suspension, such as described in 9.1.2, may give results different than those instruments not requiring an electrolyte.

9. Apparatus

9.1 *Instruments for Measuring Particle Size Distribution,* from 2 to 300 µm in size, although it may not be necessary to cover this entire range for any one sample. Satisfactory results have been obtained from instruments with three different principles of operation. Instruments from other manufacturers may be suitable but have not been evaluated.

9.1.1 *Optical Particle Counters,* that pass a collimated light beam through a flowing stream of particles to measure and count each individual particle according to the reduction in light intensity created as it passes through the sensing zone.

9.1.2 *Electrical Resistance Particle Counters,* that set up a voltage difference across an aperture through which a conductive liquid flows to measure and count each particle in the liquid by the change in conductivity or resistance as it passes through the aperture.

9.1.3 *Light-Scattering Instruments,* that view a flowing stream of particles with a laser beam and produce a Fraunhoffer diffraction pattern from which a thirteen-segment histogram of volume size distribution is obtained.

9.2 *Sample Handling System,* compatible with the particle-sizing instrument being used.

9.3 *Auxiliary Equipment,* as specified by the manufacturer of the instrument being used.

10. Material

10.1 *Nonionic Wetting Agent,* to assist in the dispersion of the particles in the liquid. The volume to be added will be small, but its cleanliness should be such that the total count in the suspending liquid (with wetting agent) does not exceed 1 % of the total count during analysis of the resin sample.

11. Calibration and Standardization

11.1 Calibrate the instrument over the size range to be measured following either the manufacturer's instructions or Method F651, Practice F658, or Test Method F322.

12. Procedure

12.1 Into a clean sample container, add measured quantities of clean liquid, clean wetting agent, and a sample of powdered ion exchange resin. The size of the sample container and the quantities of each material to be added will be determined from the operating instructions for the instrument being used for the size range to be measured.

12.2 Agitate the resulting suspension to break up aglomerates and create a uniform dispersion of particles using the equipment specified in the operating instructions for the instrument.

12.3 If necessary, dilute this suspension to provide a concentration of particles not exceeding the maximum specified for proper instrument performance.

12.4 If the sample is diluted, the diluted sample must be agitated in accordance with the instrument operating instructions to create a uniform dispersion of particles.

12.5 Pass the sample through the instrument for analysis.

12.6 Record the results.

13. Calculation

13.1 From the information provided, calculate the mean value and the percent above and below the specified sizes on the basis of weight, number, or volume.

14. Report

14.1 Report the mean particle size in micrometres, and the percent of the sample tested that is below and above certain specified values.

15. Precision and Bias

15.1 Because of the improbability of a single laboratory having all three types of particle size analyzers described in this test method, round-robin testing is not feasible. Also, since the three instruments measure different properties, complete agreement is not expected. However, for aliquots of a single sample of powdered ion exchange resin sent to owners of each type of instrument, the mean particle size values did not differ by more than 20 %.

15.2 A statement of bias may not be obtainable for this test method, since different types of instruments are involved. More fundamentally, however, powdered resin particle size distribution measurements are statistical measurements on irregularly shaped particles, and an absolute, or true, value is not possible.

TEST METHOD B—SOLIDS CONTENT

16. Scope

16.1 This test method covers the determination of the percent solids in new powdered ion exchange resins. Both anion resin and cation resin may be tested by this test method. Resins are tested in the ionic form in which they are received.

16.2 It is important to note that ion exchange resins, particularly anion exchange resins in the hydroxide form, are subject to thermal degradation under the conditions of this test. Therefore, volatile degradation products are released along with moisture, resulting in a negative error. However, the value of this test method is that it is simple and reproducible, and it is applicable to as-received samples.

17. Summary of Test Method

17.1 This test method consists of determining the loss of mass on drying at 104 ± 2°C for 18 h.

18. Significance and Use

18.1 Powdered ion exchange resins are manufactured and shipped in a moist form. However, they are sold and used on a dry weight basis. Thus, it is important that the actual solids content of the resin be determined.

19. Sampling

19.1 Obtain a representative sample of the powdered ion exchange resin in accordance with Practices D2687, Practice A, but substitute a 12.5-mm inside diameter tube.

20. Procedure

20.1 Weigh three approximately 15-g representative samples of material to the nearest 1 mg into previously tared weighing vessels.

20.2 Heat the samples for 18 h at 104 ± 2°C.

20.3 Remove the samples from the oven, cool at least 30 min in a desiccator to room temperature and reweigh.

21. Calculation

21.1 Calculate the solids content, in percent, as follows:

$$\text{Solids content, \%} = (B/A) \times 100$$

where:

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

22. Report

22.1 Reject and repeat any sample analysis in which the result differs by more than 2 % from either of the other two samples.

22.2 Report the percent solids content as the average of the three values obtained.

23. Precision and Bias[5]

23.1 The overall precision of this test method may be expressed as follows:

$$S_A = 1.43$$
$$S_C = 0.391$$

where:

S_A = overall precision for powdered anion exchange resins in hydroxide form expressed as weight percent solids, and
S_C = overall precision for powdered cation exchange resins in hydrogen or ammonium forms expressed as weight percent solids.

NOTE 1—Using this test method with resins in different ionic forms other than those indicated will, in general, increase the precision.

23.2 For the collaborative study, completed in 1982, eight powdered IEX resin samples were sent to eight laboratories. Two samples each of hydrogen form, ammonium form, hydroxide form and hydrogen/hydroxide mixed beds with fibers, were evaluated. The mixed sample results were not included in the precision calculations. Results of this collaborative study may not be typical of results for matrices other than those studied.

23.3 *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 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.

24. Quality Control

24.1 In the analysis of ion exchange resins, it is not possible to prepare a known standard resin for comparison with the actual samples. Therefore, it is impossible to test the accuracy of the results, and these test methods do not include a bias statement.

24.2 Analysts are expected to replicate samples to determine if the results are within the expected precision stated in Section 23.

25. Keywords

25.1 floc; ion exchange; particle size; powdered resin; solids content

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1084. Contact ASTM Customer Service at service@astm.org.

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