



Designation: D4438 – 24

Standard Test Method for Particle Size Distribution of Catalysts and Catalyst Carriers by Electronic Counting¹

This standard is issued under the fixed designation D4438; 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 This test method covers the determination of particle size distribution of catalyst and catalyst carrier (see Terminology [D3766](#)) particles using an electroconductive sensing method and is one of several valuable methods for the measurement of particle size.

1.2 The range of particle sizes investigated was 20 μm to 150 μm (see [IEEE/ASTM SI 10](#)) equivalent spherical diameter. The technique is capable of measuring particles above and below this range. The instrument used for this method is an electric current path of small dimensions that is modulated by individual particle passage through an aperture, and produces individual pulses of amplitude proportional to the particle volume.

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

- [B215 Practices for Sampling Metal Powders](#)
- [D1193 Specification for Reagent Water](#)
- [D3766 Terminology Relating to Catalysts and Catalysis](#)
- [E105 Guide for Probability Sampling of Materials](#)

¹ This test method is under the jurisdiction of ASTM Committee [D32](#) on Catalysts and is the direct responsibility of Subcommittee [D32.02](#) on Physical-Mechanical Properties.

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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.

- [E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process](#)
- [E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)
- [E456 Terminology Relating to Quality and Statistics](#)
- [E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)
- [E1272 Specification for Laboratory Glass Graduated Cylinders](#)
- [IEEE/ASTM SI 10 Standard for Use of the International System of Units \(SI\): The Modern Metric System](#)

3. Summary of Test Method

3.1 A carefully dispersed, dilute suspension of the representative sample in a beaker filled with an electrolyte is placed in the counting position on the instrument sample stand. The suspension is forced through a restricting aperture. Each passing particle is recorded on an electronic counter, and the data are accumulated according to selected particle size intervals for subsequent processing.

3.2 The instrument response is proportional to liquid displacement by the particle volume. Equivalent spherical diameter is commonly used to express the particle size.

4. Significance and Use

4.1 This test method can be used to determine particle size distributions for material specifications, manufacturing control, and research and development work in the particle size range usually encountered in fluidizable cracking catalysts.

5. Apparatus³

5.1 *Electronic Particle Counter*, with sample stand and stirring motor.

5.2 *Aperture Tubes*, with varying diameters. The diameter required is dependent upon the particle size distribution of the sample. Generally, any given tube will cover a particle size range from 2 % to 40 % of its aperture diameter.

³ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D32-1011.

- 5.3 *Ultrasonic Tank, 100 W.*
 5.4 *Beaker, 100-mL.*
 5.5 *Graduated Glass Pipet, 5-mL.*
 5.6 *Wash Bottles.*
 5.7 *Membrane Filtering Device with 0.22- μ m filters.*
 5.8 *Round-Bottom Sample Beakers, 250-mL.*
 5.9 *Micro-Riffler or Chute Riffler.*

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 such specifications are available.⁴ 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 conforming to Specification D1193, Type II.

6.3 *Electrolyte*—Dissolve 10.0 g of reagent grade sodium chloride (NaCl) in 1 L of distilled or deionized water and filter twice through a 0.22- μ m filter. (See Specification E1272)

NOTE 1—Commercially available Electrolyte solution of the same concentration can also be used, but should be filtered for apertures smaller than 100 μ m.

6.4 *Wash Water*—Distilled or deionized water, twice filtered through a 0.22- μ m filter. Electrolyte may also be used as wash water.

6.5 *Calibration Spheres*⁵, near monosized, having a relative standard deviation from the mean of less than 5 %, or equivalent, as certified by the manufacturer.

7. Sampling

7.1 Test sample(s) shall be obtained from larger composites by riffing or splitting in accordance with subsection 5.12 of STP 447A⁶ with the aim of obtaining a representative sample that mirrors the shape and size distribution of the larger composite. Guide E105 provides guidance on constructing a sampling plan and Practice E122 assists the analyst with determining the representative sample size. The analyst is also urged to consult Practice B215 which contains excellent sampling guidance.

⁴ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, 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.

⁵ Calibration spheres or equivalent calibration materials are generally available from respective instrument manufacturers.

⁶ Manual on Test Sieving Methods, ASTM STP 447A, ASTM International, 2005.

8. Procedure

8.1 Follow instrument manufacturer's instruction manual for instrument settings.

8.2 Follow the manufacturer's instructions for calibrating each aperture and electrolyte combination that will be used.

8.3 Before each analysis, using the wash bottle and filtered wash water, wash all surfaces coming in contact with the sample.

8.4 Place 150 mL to 200 mL of electrolyte in a round-bottom beaker on the sample stand with the stirring rod turning moderately fast. Position the stirring blades near the bottom of the beaker and increase the stirrer speed to a rate just below air bubble formation (Note 2). Follow the instruction manual and take several background counts. If they exceed the limit in the manual for the aperture in use, refilter the electrolyte and flush the glassware. If the background is due to electrical interference, the instrument manufacturer's recommendations for sample should not be used when it can disintegrate fragile primary particles.

NOTE 2—Proper adjustment of the position and speed of the stirrer will prevent the loss of large particles by settling and formation of air bubbles during counting.

8.4.1 Transfer the sample into a 250-mL round-bottom beaker containing about 200 mL of clean electrolyte. Be sure all the sample is transferred.

8.4.2 Place the sample and beaker (from 8.4.1) in the instrument sampling stand. Adjust the stirring blades close to the bottom of the round-bottom beaker so that they effectively sweep the bottom of the beaker to maintain all particles uniformly in suspension.

8.4.3 During stirring, using a 5-mL pipet, transfer 2 mL of the sample suspension (from 8.4.2) to another 250-mL round-bottom beaker containing 200 mL of clean electrolyte. Be sure all the contents in the pipet are transferred.

8.4.4 Remove the beaker (from 8.4.1) from the sampling stand. Flush the outside of the aperture tube, the stirrer, and outer electrode. Place the sample and beaker from 8.4.3 in the sampling stand. Adjust the stirring blades close to the bottom of the round-bottom beaker so that they effectively sweep the bottom of the beaker again. Increase the stirrer speed moderately. Check for particle settling by visually observing the stirring system with a flashlight and viewing the surface perpendicular to the bottom. If particles are observed settling, increase stirring rate without generating air bubbles or increase the electrolyte viscosity, or both.

NOTE 3—Electrolyte viscosity can be increased, for example, by adding filtered glycerol up to 30 % by volume.

8.4.5 Initiate a preliminary count and check that the total count is in the range suggested in the manufacturer's instructions to avoid excessive coincidence loss. If the count is too high, adjust by diluting with clean electrolyte.

8.4.6 Initiate an analysis count, and accumulate and process data in accordance with the manufacturer's instructions. For increased precision take a total of three sets of data and average them. If the resulting data in differential volume form show more than a few percent in the smallest active channel,