



Standard Test Method for Particle Size Distribution of Catalytic Material by Electronic Counting¹

This standard is issued under the fixed designation D 4438; 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.

^{ε1} NOTE—Editorial changes were made throughout in October 2001.

1. Scope

1.1 This test method covers the determination of particle size distribution of catalyst and catalyst carrier 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 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System⁴

3. Summary of Test Method

3.1 A carefully dispersed, dilute suspension of the 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. Apparatus⁵

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.

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- μm 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.⁶ 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

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 *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 14.04.

⁵ Apparatus used in this study are detailed in Research Report RR: D32-1011.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analytical Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

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 D 1193, 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- μm filter.

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

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

6.5 *Calibration Spheres*⁷, Near monosized, having a relative standard deviation from the mean of less than 5 %, or equivalent, as certified by the manufacturer.

7. Procedure

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

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

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

7.4 Place 150 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.

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

7.4.2 Place the sample and beaker (from 7.4.1) 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.

7.4.3 During stirring, using a 5-mL pipet, transfer 2 mL of the sample suspension (from 7.4.2) to another 250-mL round-bottom beaker containing 200 mL of clean electrolyte. Be sure all the contents in the pipet are transferred.

7.4.4 Remove the beaker (from 7.4.1) from the sampling stand. Flush the outside of the aperture tube, the stirrer, and outer electrode. Place the sample and beaker from 7.4.3 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.

7.4.5 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.

7.4.6 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, additional data may be obtained using a smaller aperture after large particles are scalped from the sample. Data from both apertures may then be combined, as described in the instrument manufacturer's instruction manual, although this step is beyond the scope of this test method.

8. Presentation of Data

8.1 The instrument measures the volume of each particle. Assuming all the particles in the sample have the same density, relative volume and weight can be considered to be interchangeable; therefore, cumulative and differential volume or weight percent can be printed or graphed directly from the accumulated data.

8.2 Results for samples containing mixed densities should be expressed as volume percent instead of weight percent and cannot be compared to a direct weight analysis method.

NOTE 4—Comparisons with other techniques have been found to be good for spherical particles within the sensing range covered by the aperture or apertures used. For non-spherical particles, and especially for elongated or plate-like particles, results may vary considerably.

8.3 Data are accumulated on a number basis, and may be processed in that form as well.

8.4 The median diameter is taken as the diameter corresponding to the 50th percentile of the cumulative volume distribution. This can be determined manually by drawing a line connecting the incremental segments corresponding to each data storage channel as shown in the sample curve in Fig. 1. A perpendicular is then dropped to the X-axis scale from the intersection of the drawn line and the 50th percentile grid line.

9. Precision and Bias ⁸

9.1 *Precision*—Based on the results of a multilaboratory study and using the procedures and definitions of Practice E 177 and Practice E 691 the single-laboratory, single-day repeatability is ± 2.3 % (2S %) of the mean of the measurements of the volume percent median equivalent spherical

⁷ Calibration spheres or equivalent calibration materials are generally available from respective instrument manufacturers.

⁸ Supporting data are available from ASTM International Headquarters. Request RR: D32-1011.

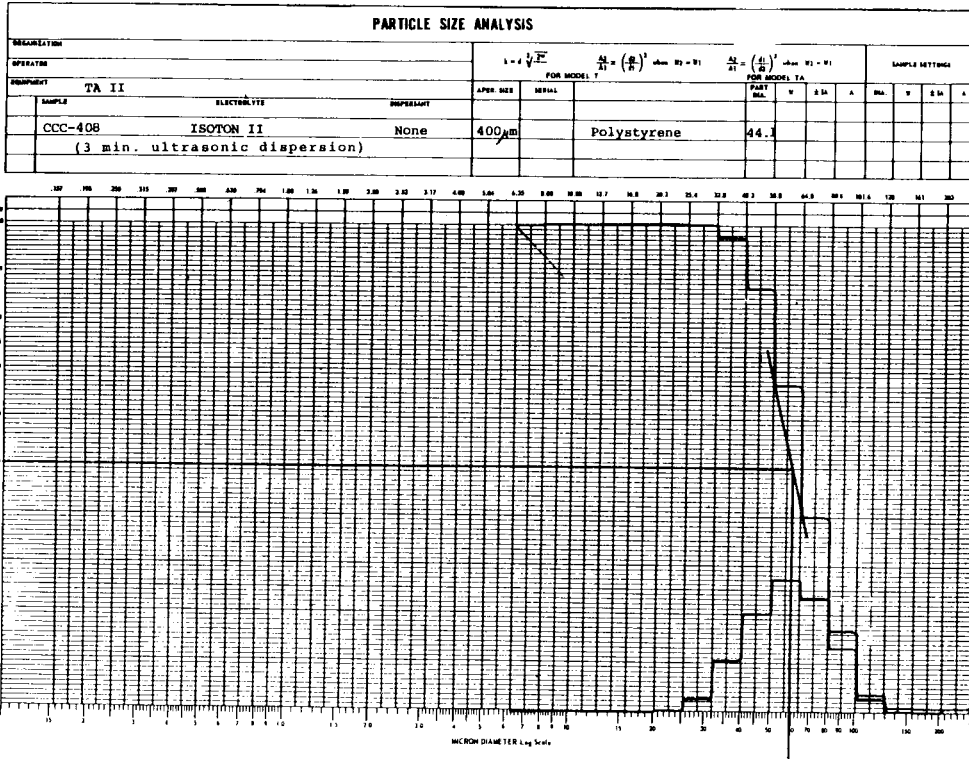


FIG. 1 Median Diameter-Sample Curve

diameter. The multilaboratory, multiday reproducibility is $\pm 4.6\%$ (2S %) of the mean of the measurements of the volume percent median equivalent spherical diameter, when a common calibration standard is used.

9.2 Bias—Reference standards have not yet been certified to

determine the bias of this test method.

10. Keywords

10.1 catalyst; electroconductive sensing; particle size

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