



Standard Test Method for Particle Size Distribution of Alumina or Quartz by Electric Sensing Zone Technique¹

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^{ε1} NOTE—Section 9 was added editorially in October 1997.

1. Scope

1.1 This test method, one of several found valuable for the measurement of particle size, covers the determination of the particle size distribution of alumina or quartz powders (0.6 to 56.0 μm) using electric sensing zone particle size analyzers.² These instruments use an electric current path of small dimensions which is modulated by individual particle passage through an aperture, and produces individual pulses of amplitude proportional to the particle volume.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety problems, 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. Summary of Test Method

2.1 A carefully dispersed, dilute suspension of the powder 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 particle passing is recorded on an electronic counter according to selected particle size levels.

2.2 The instrument response is essentially particle volume (liquid displacement), therefore equivalent spherical diameter is commonly used to express the particle size. (Comparisons with other techniques have been found to be good for spherical particles: for non-spherical particles results may differ.)

3. Significance and Use

3.1 This test method is useful to both sellers and purchasers of alumina and quartz for determining particle size distributions for materials specifications, manufacturing control, and development and research.

4. Apparatus

4.1 *Electric Sensing Zone Particle Counter*,² Coulter Counter Industrial Models B, ZB, ZM, T, TA, TA II. Particle Data Elzone Models EZ 211, 80XY, 111, 112, 180, 180t, 180XY. Equivalent instruments may be used.

4.2 *Aperture Tubes*, diameter ranging from approximately 30 to 140 μm . 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 (see Fig. A1.1).

NOTE 1—In certain cases, apertures up to 280 μm are usable.

4.3 *Sample Beaker*, capable of maintaining all particles uniformly in suspension (for example, round-bottom).

4.4 *Blender*,³ capacity 1-L glass container. Speed control with a variable transformer⁴ is required.

4.5 *Beakers*, 100, 500, and 1000-mL.

4.6 *Pipet*.

4.7 *Wash Bottles*.

4.8 *Membrane Filtering Device*, rated at 0.45- μm filters or finer.⁵

5. Reagents

5.1 *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 accuracy of the determination.

5.2 *Dispersing Media*—Ten percent solution of purified or

³ The Waring Blender model PB-5A, or equivalent has been found suitable for this test method.

⁴ The Variac transformer has been found suitable for this test method.

⁵ The membrane filters may be the Millipore, Gelman, General Electric, Selas Flotronics metal type, or their equivalent.

⁶ *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 Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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² Available from the Coulter Electronics Inc., Hialeah, FL 33010 or from Particle Data Inc., Box 265, Elmhurst, IL 60126.

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reagent grade sodium hexametaphosphate in distilled water twice filtered through the membrane filtering device.

NOTE 2—Deionized water may be substituted for distilled water.

NOTE 3—This liquid should not be retained longer than 1 month and should not be pH modified or heated.

5.3 *Electrolyte*—Dissolve 10.0 g of reagent grade sodium chloride (NaCl) in 1000 mL of distilled water and filter twice through the membrane filtering device.

5.4 *Wash Water*—Distilled water twice filtered through the membrane filtering device.

5.5 *Calibration Particles*—Monosized systems listed in Fig. A1.1.

6. Procedure

6.1 *Summary*—Disperse the test powder in the electrolyte with a blender. Transfer a representative portion to the sample beaker that contains filtered electrolyte. Place sample beaker in the counting position and obtain particle data at several selected particle size levels. Obtain relative weight fraction by assuming constant particle density.

6.2 Precalibrate the aperture and electrolyte combination following the manufacturer's instruction manual.

NOTE 4—Calibration should be performed in accordance with the instruction manual. Monosized calibration standards should be selected from Fig. A1.1. Mutual agreement on the source and size of calibration standards is necessary for interlaboratory comparisons.

6.3 Check background counts by filling the sample beaker with filtered electrolyte and taking counts without any sample added. Follow 6.6, 6.7, and 6.8.

6.4 Disperse approximately 0.7 g of sample in 200 mL of electrolyte containing 5 drops of dispersing media, by mixing at high speed on the blender or its equivalent for 5 min.

NOTE 5—The proper dispersion conditions for a given mixer or blender should be predetermined by obtaining a time-speed versus median diameter curve (see typical curve in Fig. A1.2) while ensuring that grinding does not occur. The position of the plateau will indicate the proper dispersion conditions for the sample. Experience has shown that full speed on the Waring Blender may cause size reduction. Slightly less than full speed should be used (Variat at 100 V). For some suspensions ultrasonic treatment from 1 to 5 min is effective.

6.5 With a pipet, transfer an appropriate aliquot of dispersed sample into the sample beaker containing electrolyte with dispersing media added in the ratio of 3 drops/200 mL of electrolyte. The aliquot size is dependent on the aperture size used. Wash down the pipet by rinsing with electrolyte several times (see 6.9.3).

NOTE 6—The blender or mixer should be stirring just rapidly enough to maintain a uniform particle suspension while withdrawing the sample. The pipet should deliver all of the withdrawn slurry to ensure a representative transfer of sample in the event of any size classification during the transfer.

6.6 Place the sample beaker in position on the sample stand.

6.7 Adjust the speed of the stirrer to furnish sufficient agitation to maintain a uniform particle suspension, but below air bubble generation speeds.

6.8 Take the particle count readings progressively at the prescheduled dial settings (see Fig. A1.3 for a typical single channel instrument schedule). Repeat the scheduled settings three times, beginning and ending at the smallest particle size level. Make additional count readings where values are below 100. Average the count readings for each size level. Multi-channel instruments record all the size levels simultaneously. Computer-connected instruments record, average, manipulate, and output the data automatically.

6.8.1 *Recommended Number of Count Readings:*

6 for below 10 particles per reading

4 for below 100 particles per reading

2 for above 100 particles per reading

6.9 *Precautions:*

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

6.9.2 Ensure that the calibration of the instrument is correct by checking the calibration factor at least once a week.

6.9.3 The number of particles per unit volume in the sample beaker should not exceed that which will give a 5 % coincidence correction for the aperture tube being used (see Fig. A1.1).

7. Presentation of Data

7.1 Where required (for example, single-channel manual model) convert data to cumulative weight percent greater than stated particle size according to instrument instruction manual. Coincidence is insignificant if total counts are limited to Fig. A1.1.

NOTE 7—For all electric sensing zone counters the conversion is actually to volume percent. If all particles in the sample have the same density the volume percent and weight percent are interchangeable.

7.2 Plot volume or weight percent curve on suitable graph paper. Log probability and semi-log graph papers are commonly used.

7.3 Quartiles may be interpolated from a carefully plotted curve.

7.4 Computer-connected instruments will be programmed to output graphs, tables, and statistics such as: weight percent, count percent, volume percent, mean, median, mode, quartiles, and standard deviation.

8. Precision and Bias

8.1 *Intralaboratory, Same Operator*—Experience of several laboratories indicates that the test method is capable of a precision of $\pm 1\%$ (95 % confidence level) for all size levels.

8.2 *Interlaboratory*—Experience of several laboratories indicates that the test method is capable of a precision of $\pm 3\%$ (95 % confidence level) for all size levels.

8.3 *Bias*—Instrument calibrations shall be performed using uniform spheres with relative standard deviation of 5 % or less, traceable to the U.S. Bureau of Standards SRM uniform spheres.

9. Keywords

9.1 alumina; particle size; quartz; sensing zone