



Standard Test Method for Determining the Particle Size Distribution of Alumina by Centrifugal Photosedimentation¹

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1. Scope

1.1 This test method covers the determination of the particle size distribution of alumina in the range from 0.1 to 20 μm having a median particle diameter from 0.5 to 5.0 μm .

1.2 The procedure described in this test method may be successfully applied to other ceramic powders in this general size range. It is the responsibility of the user to determine the applicability of this test method to other material.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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:

C 242 Terminology of Ceramic Whitewares and Related Products²

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

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

3. Terminology

3.1 Definitions:

3.1.1 Refer to Terminology C 242 for definitions of terms used in this test method.

4. Summary of Test Method

4.1 A homogeneous aqueous dispersion of the powder is prepared. While kept in a thoroughly mixed condition, a small aliquot is transferred to the analyzer sample cell, which is placed in the instrument and subjected to a controlled centrifugal acceleration at a known or controlled temperature. At

predetermined times related to the sedimentation of specific Stokes' diameters (Note 1), the optical absorbance is recorded and ratioed to the initial value to determine the fraction of the total sample that has sedimented a specific distance. A volume based size distribution is calculated from the absorbance-time data. Since alumina particles are not truly spherical, the results are reported as *equivalent diameters (spherical)* (Note 2).

NOTE 1—This diameter in micrometres is referred to as D in the equation:

$$D^2 = \frac{18 n (H/t)}{(\rho_s - \rho_f) u^2} \times 10^8 \quad (1)$$

where:

- n = viscosity of the fluid, P,
- H = height of the settling particles, cm,
- t = time for particle to settle, s,
- ρ_s = particle density, g/cm^3 ,
- ρ_f = fluid density, g/cm^3 , and
- u = the rotational velocity, cm/s .

NOTE 2—Refer to Terminology C 242 for the ASTM definition of this term. Most equipment manufacturers refer to this as the equivalent spherical diameter.

4.2 The instruments that have been found suitable for this test method incorporate microcomputers that control instrument operation and perform all required data acquisition and computation functions.

5. Significance and Use

5.1 Manufacturers and users of alumina powders will find this test method useful to determine the particle size distribution of these materials for product specification, quality control, and research and development testing.

6. Apparatus

6.1 *Centrifugal Particle Size Distribution Analyzer*⁴—The analyzer shall incorporate a centrifuge capable of subjecting a homogeneous dispersion of the sample to centrifugal acceleration in specially designed sample cells. A collimated beam of visible light (either monochromatic or broad-band) shall traverse the sample cell at a defined distance from the top of the

¹ This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.07 on Nonplastics.

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² *Annual Book of ASTM Standards*, Vol 15.02.

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

⁴ Instruments from Horiba Instruments, Inc., 17671 Armstrong Ave., Irvine, CA 92714, have been found satisfactory. Other instruments may also be found suitable.

cell. The change in photo extinction resulting from sedimentation of the sample shall be measured by a photo detector and appropriate electronic circuits, and used to calculate the volume-based sized distribution of the sample.

6.2 *Ultrasonic Probe*, consisting of power unit, ultrasonic transducer, and 13-mm (1/2-in.) diameter probe, 200 to 250 W.

6.3 *Ultrasonic Water Bath*, power density approximately 0.3 W/cm² (2 W/in.²).

6.4 *Balance*, top-loading, accurate to ±0.1 g.

6.5 *Stirrer*, magnetic, with 25-mm (1-in.) and 19-mm (3/4-in.) stirring bars.

6.6 *Thermometer*, mercury or alcohol, 0 to 50°C, accurate to 0.5°C.

6.7 *Sample Cells*, as supplied by the instrument manufacturer.

7. Reagents

7.1 *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.⁵ 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 precision of the determination.

7.2 *Sodium Hexametaphosphate Solution*, 0.1 %—Tare a 50-mL beaker on the balance. Weigh 1.0 ± 0.1 g of sodium hexametaphosphate into the beaker. Add 30 to 40 mL of distilled or deionized water and dissolve the salt. Pour the solution into the volumetric flask, rinse the beaker twice with water, and pour into the flask. Make up the volume to 1000 mL and mix thoroughly. Store in a stoppered glass or polyethylene reagent bottle labeled with the contents and date of preparation. Discard any solution after six weeks of storage.

NOTE 3—These reagents are adequate to cover the range of samples up to a maximum diameter of 20 µm.

8. Procedure

8.1 *Sample Preparation and Dispersion:*

8.1.1 Withdraw approximately 1 g of a thoroughly mixed powdered sample (or an equivalent amount of a slurried sample) and place into a 250-mL beaker. If the sample is received in a plastic or glass vial containing about 1 g (±0.25 g), transfer the entire contents to the 250-mL beaker. Add 200 mL of 0.1 % sodium hexametaphosphate solution and mix well by stirring. Disperse by either of the following methods:

8.1.1.1 *Ultrasonic Bath*—Place the beaker in the bath with the bottom of the beaker suspended above the bottom of the bath. In the ideal position, the top of the fluid in the beaker is even with the liquid level in the bath. Apply ultrasonic energy for 15 min with frequent stirring. Remove beaker from bath.

8.1.1.2 *Ultrasonic Probe*—Insert the probe into the beaker containing the sample and apply power for 30 s. Make sure the

sample is well suspended during this step. Remove probe from the beaker.

8.1.2 Add a 25-mm (1-in.) stirring bar to the beaker and place on a magnetic stirrer. Stir for approximately 3 min in a cold water bath to bring the sample to ambient temperature. Continue stirring at constant temperature.

NOTE 4—The concentration of the sample may require dilution with 0.1 % sodium hexametaphosphate solution to meet the optical absorbance tolerance specified in the instrument operating manual. Thorough mixing must accompany any dilution of the sample.

8.2 *Analyzer Preparation:*

8.2.1 To warm up the analyzer, apply power a minimum of 10 min prior to testing. Conduct the warm-up with the sample compartment closed. Make certain that ventilation airflow is not restricted by adjacent equipment, papers, or other materials. Check the printer to ensure a sufficient supply of paper. Clean a pair of sample cells and caps, rinse with the 0.1 % sodium hexametaphosphate solution and store inverted on absorbent paper.

8.2.2 If required by the manufacturer's operating manual, check and adjust the zero and full-scale settings.

8.2.3 Input the test parameters (sample and fluid density, fluid viscosity, test range, sedimentation distance, (if required), and centrifuge speed) according to the manufacturer's operating manual. Set the fluid viscosity at the value corresponding to the actual room temperature (see Table 1). Set the range such that the volume percent greater than the maximum diameter is certainly zero and the volume percent finer than the minimum diameter is less than 10 % (cumulative percent oversize greater than 90 %). Preliminary test runs may be required to establish the range and the size of the divisions needed to accomplish test limits. The test range and division settings shall provide a minimum of ten divisions within the test range.

8.2.4 If required by the manufacturer's operating manual, conduct a blank test with clean dispersant fluid in the sample and reference cells.

8.3 *Test Performance:*

8.3.1 Adjust the rate of the magnetic stirrer to produce a slight vortex in the sample dispersion. With a disposable plastic pipet, withdraw an appropriate volume of sample from the center of the dilute dispersion. Make certain that the dispersion is thoroughly mixed by vigorous pumping with the pipet (avoid sufficient agitation to create bubbles). Completely transfer the withdrawn sample portion to the drained sample cell. Insert the cell cap, carefully wipe all outer surfaces of the cell, place in the centrifuge, and start the test. At the beginning of the test, monitor the absorbance display on the instrument to ensure that the initial absorbance is within the specified tolerance. If not, refer to Note 4. When the test is completed, remove the sample cell, clean immediately, and rinse with dispersant fluid.

9. Presentation of Data

9.1 *Analyzer Printout*—The analyzers that have been found

TABLE 1 Viscosity Coefficients of Water^A

Temperature, °C	20	21	22	23	24	25	26	27	28	29
Viscosity coefficient, mPa·s or cP	1.01	0.98	0.96	0.94	0.92	0.89	0.87	0.86	0.84	0.82

^A Viscosity coefficient values to two decimal places have been found satisfactory for this test method.

⁵ *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.

suitable for this test method render data printouts that provide a permanent record of test conditions and graphic records of the size distribution. For many purposes, these printouts may provide sufficient information.

10. Fluid Densities and Viscosity Coefficients

10.1 Table 1 lists the viscosity coefficients for water over the temperature range from 20 to 29°C. Use these values for the sodium hexametaphosphate solution. Use a density of 1.00 g/mL for these solutions.

11. Additional Data Presentations

11.1 The analyzers that have been found suitable for use with this test method produce a printed report that may include a cumulative size distribution table showing the volume percent oversize, starting with the programmed maximum diameter. The incremental divisions are, in some operating modes, selectable by the operator, and, in others, determined

by the microcomputer in the instrument.

12. Precision and Bias

12.1 *Precision*—Based on the results of a multilaboratory study using the procedures and definitions of Practices E 691 – 79 and E 177 – 80, and over the sample range included in the study the precision is as follows:

12.1.1 *Repeatability*—The within-laboratory repeatability is 3.56 % ($2s$ %) of the measured value.

12.1.2 *Reproducibility*—Between-laboratory reproducibility is 9.92 % ($2s$ %) of the measured value.

12.2 *Bias*—Since no absolute method of particle size distribution is recognized, it is not possible to discuss the bias of the results obtained using this test method.

13. Keywords

13.1 alumina; particle size distribution; photosedimentation

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