

Standard Test Method for Particle Size Distribution of Alumina or Quartz by X-Ray Monitoring of Gravity Sedimentation¹

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1. Scope

1.1 This test method covers the determination of the particle size distribution of alumina or quartz powders in the range from 0.5 to 50 μm and having a median particle diameter from 2.5 to 10 μm using a sedimentation method. This test method is one of several found valuable for the measurement of particle size. Instruments used for this test method employ a constant intensity X-ray beam that is passed through a sedimenting dispersion of particles.

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

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.* For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:

C 242 Terminology of Ceramic Whitewares and Related Products²

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

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology C 242.

4. Summary of Test Method

4.1 An aqueous homogeneous dispersion of the specimen is permitted to settle in a cell. The decrease in particle concentration over a programmed settling distance is monitored by an X-ray beam passing through the sedimenting dispersion to a detector. The specimen concentration at any given sedimenta-

tion distance is inversely proportional to the X-ray flux and the equivalent diameter (spherical) is calculated from Stokes' law.

NOTE 1—Refer to Terminology C 242. Most equipment manufacturers refer to this as the equivalent spherical diameter.

5. Apparatus

5.1 X-Ray Sedimentation Apparatus.⁴

5.2 *Ultrasonic Probe or Bath*—An ultrasonic probe approximately 13-mm ($\frac{1}{2}$ -in.) in diameter and approximately 50 to 100 W or an ultrasonic bath of approximately 0.3 W/cm² (2 W/in.²).

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.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 accuracy of the determination.

6.2 Distilled or Deionized Water.

6.3 *Dispersing Liquid*—Dissolve 1.0 g/L of reagent grade sodium hexametaphosphate in distilled or deionized water. Discard any remaining solution after six weeks from date of preparation.

7. Hazards

7.1 Precautions applying to the use of low-intensity X-rays should be observed.

7.2 Users should be aware that sample concentrations used in this test method may be higher than those considered ideal by some authorities and that the range of this test method extends into the region where Brownian motion could be a factor in conventional gravity sedimentation. Within the range

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 available from Quantachrome Corp., 5 Aerial Way, Syosset, NY 11791, and Micromeritics Instrument Corp., One Micromeritics Drive, Norcross, GA 30093, have been found satisfactory for this purpose.

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.

of this test method neither the sample concentration nor Brownian motion are believed to be significant.

8. Procedure

8.1 Prepare the instrument for use according to the manufacturer's instructions.

8.2 If necessary, manually zero the instrument with the cell filled with the dispersing liquid.

8.3 Transfer 2.5 g of the dry specimen (or an equivalent amount of a slurried specimen) into a 50-mL beaker and add 30 mL of the dispersing liquid. Mix well using a rubber policeman to break up any clumping.

8.3.1 The amount of specimen required may vary between instruments. Adjust the specimen weight to reduce the X-ray intensity according to the manufacturer's recommended conditions.

8.4 Disperse the specimen using either of the instrument's given in 8.4.1 and 8.4.2:

8.4.1 *Ultrasonic Bath*—Place the beaker in the bath and disperse the specimen for 5 min with frequent stirring. It is beneficial to cause the liquid level in the bath and beaker to match.

8.4.2 *Ultrasonic Probe*—Insert the probe into the beaker and disperse the specimen for 1 min. Do not allow the temperature of the dispersion liquid to rise more than 2°C.

8.5 Add a 25-mm (1-in.) magnetic stirring bar to the beaker, and place the beaker in the instrument's cell compartment. Adjust the stirring rate to produce a slight vortex.

8.5.1 If an instrument provides a different method for stirring and circulating the dispersion, be sure to follow the appropriate procedure.

8.6 Fill the cell with the dispersed slurry according to the manufacturer's instructions, making sure that no air bubbles remain in the cell.

8.7 Run the particle size distribution in the "Percent Finer Than" mode using a starting diameter of 50 µm. Calculate the instrument rates using a density of 3.95 g/m³ for alumina and 2.65 g/m³ for silica.

8.7.1 It is recommended that 8.6 and 8.7 be repeated in order to increase confidence in the analysis and to ensure that

good dispersion has been achieved.

8.8 Upon completion of the analyses, rinse or clean the instrument for the following run as suggested by the manufacturer.

9. Precision and Bias ⁶

9.1 *Interlaboratory Test Program*—An interlaboratory study of the particle size distribution of alumina and quartz was run in 1989. Nine laboratories participated in the study, which included three different models of instruments, with each laboratory testing five specimens of each material. Except for the use of only two materials, Practice E 691 was followed for the design and analysis of the data.

9.2 *Test Result*—The precision information given in 9.3 in the units of equivalent spherical diameter (in micrometres) is for the comparison of two test results, each of which is the average of five test determinations.

9.3 Precision:

Test range	1 to 50 µm
95 % repeatability limit (within laboratory)	10 % (5 to 21 %) of the test result
95 % reproducibility limit (between laboratories)	15 % (8 to 29 %) of the test result

The above terms repeatability limit and reproducibility limit are used as specified in Practice E 177. The respective percent coefficients of variation among test results may be obtained by dividing the above numbers by 2.8.

9.4 *Bias*—No absolute method of particle size determination is recognized. Therefore, no justifiable statement can be made on the bias of the procedure in this test method.

10. Keywords

10.1 alumina; gravity sedimentation; particle size; particle size distribution; quartz; sedimentation

⁶ Supporting data are available from ASTM Headquarters. Request RR: C21-1004.

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