

Standard Test Method for Precision Electroformed Wet Sieve Analysis of Nonplastic Ceramic Powders¹

This standard is issued under the fixed designation C 925; 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 the particle size distribution of pulverized alumina and quartz for particle sizes from 45 to 5 μm by wet sieving.

1.2 *This standard does not purport to address the safety concerns 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:*

E 161 Specification for Precision Electroformed Sieves (Square Opening Series)²

3. Summary of Test Method

3.1 A separate dispersed suspension of the powder is wet sieved through each sieve, using vacuum and vibration. The sieve and sample are dried and weighed.

4. Significance and Use

4.1 Both suppliers and users of pulverized ceramic powders will find this test method useful to determine particle size distributions for materials specifications, manufacturing control, development, and research.

4.2 The test method is simple, although tedious, uses inexpensive equipment, and will provide a continuous curve with data obtained with standardized woven sieves.

5. Apparatus

5.1 *Precision Electroformed Sieves*, 3-in., mounted in brass frames, having nominal apertures of 45, 30, 20, 10, and 5 μm and a support grid having 5.7 lines per centimetre.³ Intermediate sizes may also be used.

5.2 *Sieving Device* (Fig. 1):

5.2.1 *Filtering Flask* (suction flask), 1-L, with side arm,

5.2.2 *Büchner Funnel* (for example, Coors No. 2),

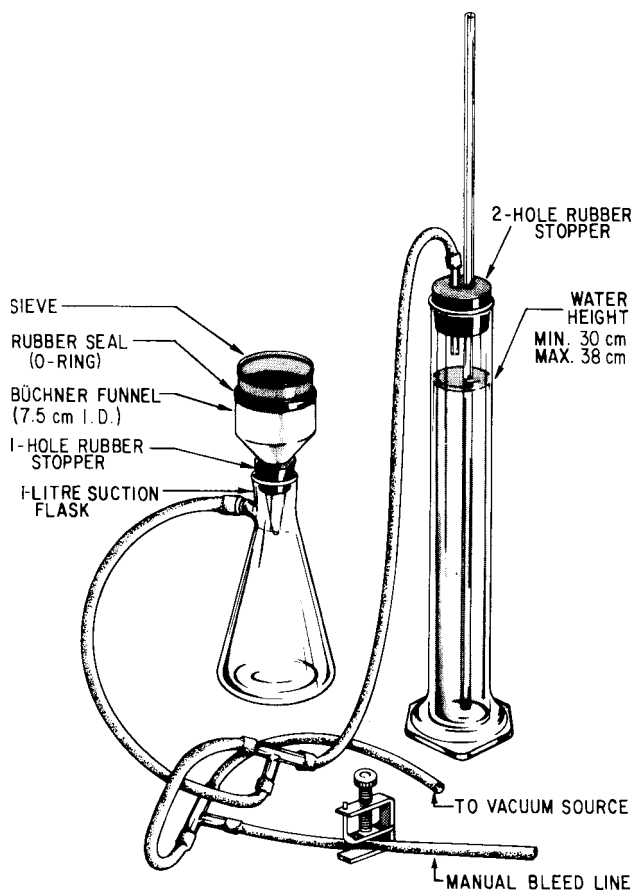


FIG. 1 Wet Sieving Apparatus

5.2.3 *O-Ring*, 7.5-cm, rubber,

5.2.4 *Graduate*, 1-L,

5.2.5 *Rubber Stoppers*, one-hole to fit the flask and the funnel, two-hole to fit the graduate,

5.2.6 Quantity of glass tubing and rubber tubing,

5.2.7 *Metal Rod*, 15 to 20-cm, about 5 mm in diameter, and,

5.2.8 *Vacuum Source*.

5.3 *Ultrasonic Cleaner*, required to clean all sieves below 20 μm . It should be low-powered (for example, 100 W).

5.4 *Analytical Balance*, capable of weighing up to 100 g and having at least three significant digits after the decimal.

5.5 *Drying Oven*, capable of maintaining $110 \pm 5^\circ\text{C}$.

5.6 *Desiccator*, containing magnesium perchlorate or other suitable desiccant.

¹ This test method is under the jurisdiction of ASTM Committee C-21 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 14.02.

³ A support grid, manufactured by Buckbee Mears Co., 245 E. Sixth St., St. Paul, MN 55101, has been found satisfactory for this purpose.

6. Reagents and Materials

6.1 *Water*, visually clear and particle free, not necessarily distilled, at room temperature or slightly above.

6.2 *Sieving Solution*, a dispersing media consisting of 0.1 weight % solution of sodium hexametaphosphate or sodium pyrophosphate in water.

6.3 *Drying Agents*, acetone or methyl alcohol, commercial grade.

7. Sampling

7.1 Since the amount of sample used in the determination is quite small, great care must be taken to avoid segregation. Gross samples must be cut down very carefully using splitters, riffles, or simple cone and quartering techniques. Adjusting the portion to an exact weight must be avoided. That is, a representative portion must be extracted from the analytical sample and all of it weighed and used.

8. Preparation of Apparatus

8.1 *Cleaning of Sieves*—Use sufficient sieving solution to cover the sieve when suspended near the bottom of the ultrasonic tank. (Some organic liquids are better but create a definite explosion hazard.) Turn on the generator for about 30 s. Fine sieves may need multiple treatments. (Exposure to ultrasonic energy for periods exceeding 30 s at one time may damage the sieves.) After each cleaning carefully examine the sieve against a light, using a magnifying glass, for blockages and breaks. (Breaks can be repaired with epoxy resin.) Sieves of 20 μm and finer should be cleaned after each analysis. Rinse in clear water and dry at 110°C.

8.2 *Collector and Vacuum Control*—Assemble apparatus as shown in Fig. 1.

9. Calibration and Standardization

9.1 *Calibration of Sieves*—This must be done for highly accurate analyses or to verify the apertures that may not be within the quoted 2- μm tolerance. The calibration procedure is given in Appendix X1 of Specification E 161.

10. Procedure

10.1 Wash the sieves with the sieving solution, rinse with water and then with the drying agent, dry at 110°C for 15 min, cool in the desiccator, and weigh to at least the nearest 1 mg.

10.2 *Sample Size* is somewhat dependent upon density, but the following schedule is satisfactory for alumina and quartz:

Nominal Sieve Apertures, μm	Sample Weight (approximate), g
45, 30, 20	1.0
15, 10	0.4
5	0.15

10.3 Weigh the required amount of sample into a 100-mL beaker, fill half with sieving solution (containing the dispersing agent), and disperse by placing beaker in ultrasonic bath for 1

min. Microscopical examination of the particles after dispersion may be helpful in determining if the dispersion is complete.

10.4 Wash the dispersed sample onto the sieve using a wash bottle containing the sieving solution, turn on vacuum (a maximum of 15 in. of water), and agitate. Continuously add sieving solution, at a rate sufficient to maintain a small amount in the sieve, until about 1 L has passed. Below 6 μm the amount of liquid required is less due to the time for sieving. Agitate by sharp tapping on the sieve frame with the metal rod. For sieves above 30 μm the liquid runs quickly enough without vacuum so that the air-bleed valve can be opened. The bleed-line should be pinched occasionally, however, to draw the larger particles through, but care must be taken to avoid damage to the sieve. For apertures of 10 μm or less, when the sieving appears complete (that is, the filtrate is clear) or the rate of throughput becomes very slow, draw the sieve down to near dryness with the vacuum. Remove the sieve from the sieving device and partially immerse it in the sieving solution in the ultrasonic tank for about 1 min. The particles that have blocked the apertures will be forced back into the sieve. Regular sieving is then continued. This procedure must be repeated until the wash solution in the ultrasonic tank remains clear (not visually cloudy) after washing. This may be as many as 4 to 5 times for the 5- μm sieve to assure that the sieving is complete.

10.5 Rinse with distilled water to remove any sieving solution and then rinse with drying agent, dry for 15 min at 110°C, cool in the desiccator, and weigh sieve and sample.

10.6 Triplicate replications are advised to assure repeatability.

11. Calculation and Report

11.1 Report the “weight percent greater than aperture” for each replicate and for each sieve along with the calibrated sieve openings. Calculate as follows:

$$W = \frac{B - C}{D} \times 100 \quad (1)$$

where:

W = weight percent greater than aperture,

B = weight of sieve with sample, g,

C = weight of empty sieve, g, and

D = original sample weight, g.

12. Precision and Bias

12.1 *Precision*—The precision of the procedure in Test Method C 925 is being determined.

12.2 *Bias*—The true value of the particle size can only be defined in terms of the test method. Within this limitation, this test method has no known bias.

13. Keywords

13.1 ceramic powders; particle size distribution; precision electroformed sieves; wet-sieves

 **C 925**

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