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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Particle Size Measurement of Dry Toners¹

This standard is issued under the fixed designation F 577; 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 a single aperture particle size analysis using an electronic sensing zone apparatus. Dry inks, toners, etc., are covered. Particles as small as 1.4 μm and as large as 80 μm can be analyzed.

1.2 *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. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *number size distribution*—the number size distribution is measured and is characteristically represented as a cumulative number percent curve (Fig. 1).

2.1.2 *volume size distribution*—the volume size distribution is mathematically calculated and is characteristically represented as a cumulative volume percent curve (Fig. 1).

2.1.3 *median particle size*—the median size (50 % oversize or undersize) is a convenient value for the central tendency of a size distribution curve. For a distribution derived by number of particles, it is called the number median size. If volume is used to derive the distribution, it is referred to as the volume median size.

2.1.4 *channel/size/class/interval*—the class interval is a subgroup that has been obtained by dividing the range of particle sizes into an arbitrary number of channels. Most instruments have divided the channels to give class intervals that are on a geometric progression using a factor of

3. Summary of Test Method

3.1 This technique (1)² determines the number and size of particles suspended in an electrolyte by causing them to flow through a small orifice on both sides of which are immersed electrodes. Voltage pulses, whose amplitudes are proportional

to the particle volumes, are generated by changes in resistance as the particles pass through the orifice. These pulses are amplified, sized, and counted, yielding size distribution data for the suspended phase.

3.2 This test method covers the size range from 2 % to 40 % of the aperture diameter chosen as being appropriate to the expected particle size range.

Aperture Diameter, μm	Particle Size Range, μm
70	1.4 to 28
100	2.0 to 40
140	2.8 to 56
200	4.0 to 80

Two aperture techniques for broad size ranges are not covered by this test method.

4. Significance and Use

4.1 This test is useful in determining particle size characteristics of dry toners used in copying machines. It is a practiced method for use in quality control of toner particle size.

5. Apparatus

5.1 *Electrical Sensing Zone Instrumentation (2)*,³ equipped with a minimum capability of twelve size channels and a nominal 70, 100, 140, or 200- μm aperture tube is suitable for most dry toner measurements.

5.2 *Ultrasonic Dispersing Probe*, or alternative equipment suitable for dispersing the dry toner in an aqueous electrolyte.

6. Reagents and Materials

6.1 *Electrolyte*⁴—4 weight % aqueous sodium pyrophosphate or 1 weight % sodium chloride. The electrolyte shall be adequately filtered to remove almost all particle contaminants greater than 1 μm . Some aqueous electrolytes are commercially available.

6.2 *Surfactant*⁵—a nonionic surface active agent suitable

³ The Counter from Coulter Electronics has been found satisfactory. This is available from Coulter Electronics, Hialeah, FL.

⁴ Aqueous filtered saline solutions are available from Curtin Matheson Scientific, Houston, TX, and J. T. Baker Co., Phillipsburg, NJ.

⁵ Triton X-100, available from Rohm and Haas, Philadelphia, PA, diluted to 0.05 weight % and filtered has been found acceptable. This and other nonionic surfactants can also be obtained from Curtin Matheson Scientific. These have already been diluted in the proper concentration and adequately filtered to remove fine particles.

¹ This test method is under the jurisdiction of ASTM Committee F-5 on Business Copy Products and is the direct responsibility of Subcommittee F05.04 on Electrostatic Copy Products.

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² The **boldface** numbers in parentheses refer to the list of references at the end of the method.

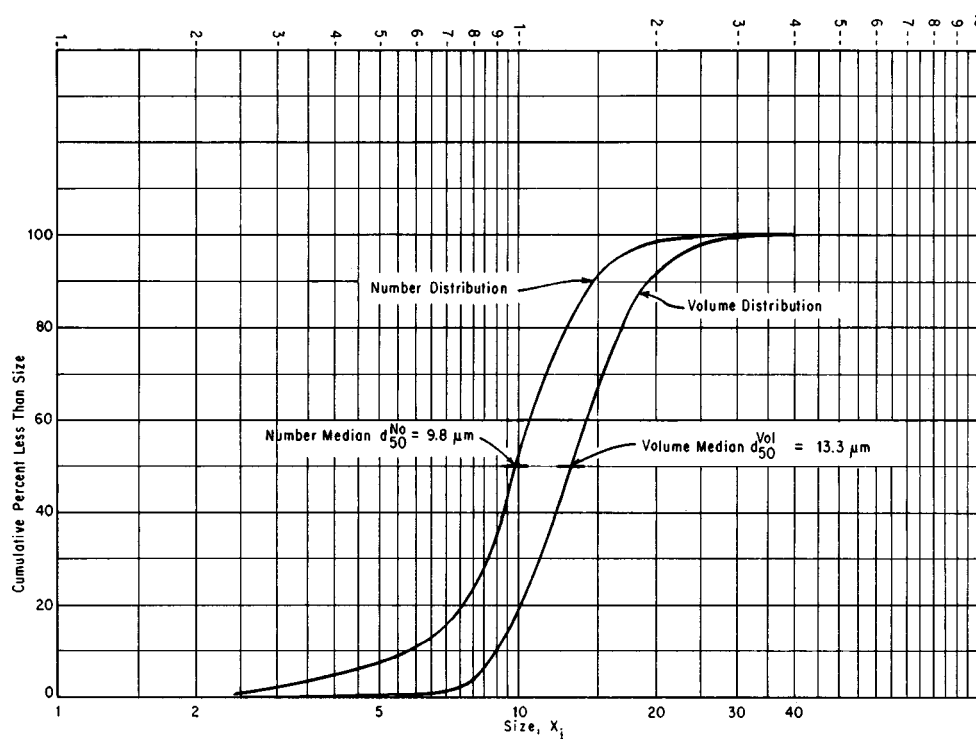


FIG. 1 Toner Particle Size Distribution-Electrical Sensing Zone Method

for keeping toner particles separated while in suspension.

6.3 Near monosized spherical particles for calibration standards.⁶

7. Sampling

7.1 Sample the powder when flowing (1).

7.2 Sample the entire powder flow over small intervals of time. This is preferable to a continuous withdrawal of a small fraction of the flow.

NOTE 1—Often the processes used to produce dry toner and the semicohesive, electrostatic nature of the fine material can make it prohibitively difficult to follow these important general rules for powder sampling.

NOTE 2—These rules are especially important for free-flowing powders of broad-size distribution, which tend to segregate.

NOTE 3—In the case of toner, because of its semicohesive nature and relatively narrow distribution, in contrast to many powders generated from mineral processing, the problems of segregation are frequently not as severe.

7.3 A further positive aspect is that electrostatic imaging requires material that produces uniform, stable, and acceptable copy quality, one copy after the other. In general, the usage rate is in the range from 10 to 100 mg per copy, depending on the original document and the electrostatic conditions. Each copy, consequently, contains a small sampling of the bulk toner.

7.4 The above considerations tend to permit a practical assessment of quality by the measurement of a number of small samples taken from various sections of nonmoving powder beds and containers. These samples may be obtained by

probes, also known as “thieves,” for which many designs exist. In fact, this method is often preferable to more elaborate techniques, like sample splitters, which have moving parts. Such devices are difficult to maintain, and may have places where the thermally sensitive powder is fused by shear to form large, undesirable aggregates.

8. Calibration and Standardization

8.1 The electrical sensing zone equipment should be calibrated with monosized latex polymer microspheres (3, 4) which have been accurately sized. Calibration standards should be checked to assure the accuracy of their reported sizes.

9. Procedure

9.1 Set up the electrical sensing zone apparatus in accordance with the manufacturer’s instruction manual.

9.2 Measure the total background count of filtered electrolyte for the chosen orifice. The 70- μm orifice with the lowest active channel at a 1.4- μm diameter should have less than 370 counts per 0.5 mL. The 100- μm orifice with the lowest active channel at 2.0- μm diameter should have less than 200 counts per 0.5 mL. The 140- μm orifice with the lowest active channel at 2.8- μm diameter should have less than 80 counts per 0.5 mL.

9.3 Measure approximately 5 to 10 mg of toner into a 50-mL borosilicate beaker filled with filtered electrolyte, and add 1 or 2 drops of filtered nonionic surfactant. The amount of toner used in an analysis is very important if the material has a high percentage of finer particles (5).

9.4 Fully disperse the toner in the electrolyte-surfactant mixture using an ultrasonic probe at approximately 6 W power. Care should be taken in sonication to avoid the fracturing of particles from the beaker. Thirty seconds maximum sonication at the wattage setting recommended has been found acceptable.

⁶ Available from Coulter Electronics, Hialeah, FL, Dow Chemical, Indianapolis, IN, Duke Scientific, Palo Alto, CA, Particle Information, Grants Pass, OR.