AMERICAN SOCIETY FOR TESTING AND MATERIALS 100 Barr Harbor Dr., West Conshohocken, PA 19428 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Practices for Testing Polymeric Powders and Powder Coatings¹

This standard is issued under the fixed designation D 3451; 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 These practices cover the selection and use of procedures for testing polymeric powders and powder coatings. The test methods included are listed in Table 1. Where more than one test method is listed for the same characteristic, no attempt is made to indicate superiority of one method over another. Selection of the methods to be followed must be governed by experience and the requirements in each individual case, together with agreement between the purchaser and the seller.
- 1.2 These practices describe the testing of polymeric powders as applied by electrostatic spray, fluidized bed, or any other applicable method.
- 1.3 These practices apply to proper and safe packaging, shipping and receiving, and storage and handling during use and application of polymeric powders.
- 1.4 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. Referenced Documents

- 2.1 ASTM Standards:
- B 117 Practice for Operating of Salt Spray (Fog) Testing Apparatus²/standards.iteh.ai/catalog/standards/sigt/bff
- D 153 Test Methods for Specific Gravity of Pigments³
- D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings⁴
- D 523 Test Method for Specular Gloss⁴
- D 609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products⁴
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces⁵
- D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive⁴
- ¹ These practices are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.51 on Powder Coatings.
- Current edition approved May 15, 1992. Published July 1992. Originally published as D 3451-75. Last previous edition D 3451-76 (1987) $^{\epsilon 1}$.
 - ² Annual Book of ASTM Standards, Vol 03.02.
 - $^{\rm 3}$ Annual Book of ASTM Standards, Vol 06.03.
 - ⁴ Annual Book of ASTM Standards, Vol 06.01.
 - ⁵ Annual Book of ASTM Standards, Vol 06.02.

- D 659 Test Method for Evaluating Degree of Chalking of Exterior Paints⁴
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints⁴
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints⁴
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints⁴
- D 714 Test Method for Evaluating Degree of Blistering of Paints⁴
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints⁴
- D 792 Test Methods for Specific Gravity (Relative Density) and Density of Plastics by Displacement⁶
- D 870 Practice for Testing Water Resistance of Coatings
 Using Water Immersion⁴
- D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive⁴
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers⁴
- D 1044 Test Method for Resistance of Transparent Plastics to Surface Abrasion⁶
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁴
- D 1193 Specification for Reagent Water⁷
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes⁵
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base⁴
- D 1474 Test Methods for Indentation Hardness of Organic Coatings⁴
- D 1535 Test Method for Specifying Color by the Munsell System⁴
- D 1644 Test Methods for Nonvolatile Content of Varnishes⁴
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁴
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁸
- D 1731 Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting⁸

⁶ Annual Book of ASTM Standards, Vol 08.01.

⁷ Annual Book of ASTM Standards, Vol 11.01.

⁸ Annual Book of ASTM Standards, Vol 02.05.

TABLE 1 List of Test Methods

	Section	ASTM method		Section	ASTM method
Polymeric Powder Properties:			Gravelometer	24	D 3170
Particle size and distribution	8		Color, pigmented coatings	25	
Multiple sieve and analysis	8.1	D 1921	Visual	25.2	D 1535
Vacuum sieve analysis	8.2		Instrumental	25.3	E 308
Sonic sifter analysis	8.3		Color difference		26
Electronic counting analysis	8.4		Visual	26.2	D 1729
Package stability	9		Instrumental	26.2	D 2244
Glass vial method	9.1		Cracking resistance		
Open dish method	9.2		Humidity, thermal cycle27	D 2246	
Pourability10	D 1895		Elongation28		
Fluidity11			Conical mandrel	28.2	D 522
Nonvolatile content12	D 1644		Cylindrical mandrel	28.2	
Volatile content at fusion temperature	13.1		Filiform corrosion	29	D 2803
Gel time (stroke cure)	14		Film thickness		
Melting point	15		On nonmagnetic metal	20.5	D 1400
Viscosity (Weissenberg)	16		On magnetic base	20.5	D 1186
Flow test (incline method)	17		Destructive method	20.5	D 1005
Density and specific gravity	18		Gloss	30	D 523
Apparent density	18.1	D 1895	Hardness	31	
Bulk factor	18.2	D 1895	Knoop indentation	31.1	D 1474
Specific gravity	18.3	D 153	Pfund indentation	31.1	D 1474
Application Properties:			Impact resistance	32	D 2794
Efficiency of powder coating process	19		Outdoor exposure	33	
Powder Coating Properties:			Blistering	33.2.1	D 714
Abrasion resistance	21		Chalking	33.2.2	D 659
Air blast abrasion tester		D 658	Checking	33.2.3	D 660
Falling sand method		D 968	Cracking	33.2.4	D 661
Taber abraser		D 1044	Rusting	33.2.5	D 610
Adhesion			Erosion	33.2.6	D 662
Scrape adhesion 22	D 2197		Flaking	33.2.7	D 772
Parallel groove	77116	D 2197	Print resistance	34	D 2091
Tape adhesion		D 3359	Salt spray resistance	35	B 117
Chemical resistance	23	3 0000	Water resistance	36	
Household chemical resistance	23.2	D 1308	High humidity	36.2	D 1735
Detergent resistance	23.3	D 2248	Water immersion	36.3	D 870
Chip resistance	20.0	2 22 10	Safety and Handling	37	2 010
omp redictance	- Hacı	mei			

- D 1732 Practices for Preparation of Magnesium Alloy Surfaces for Painting⁸
- D 1735 Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus⁴
- D 1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials⁶
- D 1898 Practice for Sampling of Plastics⁶
- D 1921 Test Method for Particle Size (Sieve Analysis) of Plastic Materials⁶
- D 2091 Test Method for Print Resistance of Lacquers⁵
- D 2092 Guide for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting⁵
- D 2197 Test Method for Adhesion of Organic Coatings by Scrape Adhesion⁴
- D 2201 Practice for Preparation of Zinc–Coated and Zinc–Alloy–Coated Steel Panels for Testing Paint and Related Coating Products⁴
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁴
- D 2246 Test Method for Finishes on Primed Metallic Substrates for Humidity-Thermal Cycle Cracking⁴
- D 2248 Practice for Detergent Resistance of Organic Finishes⁴
- D 2454 Practice for Determining the Effect of Overbaking on Organic Coatings⁴
- D 2794 Guide for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)⁴

- D 2803 Guide for Filiform Corrosion Resistance of Organic Coatings on Metal⁴
- D 3170 Test Method for Chipping Resistance of Coatings⁵ D 3359 Test Methods for Measuring Adhesion by Tape Test⁴
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁹
- E 308 Method for Computing the Colors of Objects By Using the CIE System⁴

3. Terminology

- 3.1 Definitions of Terms Specific to This Standard:
- 3.1.1 *bulk density*—mass per unit volume of a material in powder form including the air trapped between particles.
- 3.1.2 coating powder—finely divided particles of organic polymer, either thermoplastic or thermosetting, which generally contain pigments, fillers, and additives and which remain finely divided during storage under suitable conditions.
- 3.1.3 *film formation from a coating powder*—the forming of a continuous film by melting powder particles and coalescing them by the application of energy.
- 3.1.3.1 *Discussion*—For thermosetting materials, a chemical reaction, either condensation or addition, also takes place. For thermoplastic materials, no chemical reaction takes place.

⁹ Annual Book of ASTM Standards, Vol 14.02.

The material flows when heat is applied and develops performance properties when cooled. Flow will reoccur when reheated. Both films have the uniformity of color, toughness, and other properties associated with protective and decorative coatings. This fused film has the uniformity, color, toughness, and other properties associated with protective and decorative coatings.

- 3.1.4 *fluidity*—the ability of a powder to move freely, uniformly, and continuously (somewhat like a liquid) when subjected to certain conditions of pressure, temperature, and velocity of a carrier gas.
- 3.1.5 *gel time*—interval required at a given temperature for a powder to be transformed from a dry solid to a gel-like state.
- 3.1.6 *particle size*—average diameter of an object having irregular boundaries that can be described in an artificial way as having a diameter.
- 3.1.7 particle-size distribution—arrangement of particle size measurements on a powder in groups of specified diameters.
- 3.1.8 *pourability*—the ability of a powder to flow uniformly or to be continuously poured from a container at a steady rate.
- 3.1.9 *powder coatings*—coatings which are protective or decorative, or both, formed by the application of a coating powder (3.1.2) to a substrate and fused into a continuous film by the application of heat or radiant energy.
- 3.1.10 *storage stability*—the ability of a powder to maintain uniform physical and chemical properties after being subjected to specified storage conditions.
- 3.1.11 *volatile content*—the quantity expressed as weight percent of the powder which is lost under specified conditions of temperatures and time.
- 3.1.12 *cloud chamber technique*—method of moving a charged or uncharged object through a charged or uncharged cloud of powder in an enclosed chamber.
- 3.1.13 *electrostatic deposition*—technique of moving and charging powder so that it is deposited onto an oppositely charged substrate by one of the following methods.
- 3.1.14 *fluidized bed technique*—method of moving a grounded object over or through a charged fluidized powder.
- 3.1.15 *spray technique*—method of spraying and charging powder so that it is deposited onto a grounded charged substrate.
- 3.1.16 *nonelectrostatic deposition*—technique of moving powder onto a substrate which may be heated above the fusion point of the powdered material.
- 3.1.17 *spray technique*—method of spraying powder onto a substrate which may be heated above the fusion point of the powdered material.
- 3.1.18 *fluidized bed technique*—method of dipping a hot object into a container holding the powder suspended in a gas stream so that it resembles a fluid and allowing the powder to melt onto the object being coated.
- 3.1.19 *flame spread technique*—method of applying powder using a compressed gas and melting the powder in a flame before the powder impinges on the surface.

4. General Requirements

4.1 All tests shall be made in diffused light (not direct sunlight) and at $23 \pm 2^{\circ}$ C and 50 ± 5 % relative humidity,

after conditioning 16 h unless otherwise specified.

5. Sampling

- 5.1 Sample the powder in accordance with Practice D 1898.
- 5.2 Prepare specimens as required for the specific tests on the coating.

6. Equipment

6.1 Use the equipment as specified in each method of test.

7. Conditions Affecting Polymeric Powder or Powder Coatings, or Both

- 7.1 Practical requirements and performance of powder and powder coating may vary with:
- 7.1.1 Substrate Type—Ferrous, nonferrous types, plastic, or elastomeric.
- 7.1.2 *Substrate Weathering*—Weathering of the substrate will probably adversely affect the performance.
- 7.1.3 The type, quality, and suitability of the metal treatment or primer used under the powder coating and the time before coating application.
 - 7.1.4 Application conditions.
 - 7.1.5 Contaminants on the surface of the substrate.
- 7.1.6 Damage to container, size of container, storage time, excessive temperature, and temperature fluctuations which may cause settling, caking, or chemical change.

POLYMERIC POWDER PROPERTIES

8. Particle Size and Distribution

Note 1—Technology in particle size and distribution measurement, such as laser diffraction, has been developed. Instruments are commercially available from several suppliers.

8.1 Multiple Sieve Analysis:

8.1.1 This method employs multiple sieves to determine particle sizes and their distribution. Standard 200-mm (8-in.) diameter sieves and a mechanical shaker are used. A representative quantity of the sample is sifted through a series of sieves and the amount retained on each sieve is weighed and calculated as percent of the total specimen.

8.1.2 Apparatus:

- 8.1.2.1 *Sieves*—Half-height sieves, 203.2 mm (8.0 in.) in diameter, conforming to the requirements of Specification E 11. A selection of sieves encompassing the expected range of particle sizes together with a cover and a bottom pan are required.
- 8.1.2.2 Mechanical Sieve Shaker with Automatic Time Switch 10 —The mechanical sieve-shaking device shall be capable of imparting uniform rotary motion and a tapping action at the rate of 150 ± 10 taps/min.
- 8.1.2.3 *Balance*—A laboratory balance with a minimum capacity of 500 g sensitive to 0.1 g, for weighing the specimen and the residues retained on the sieves.
 - 8.1.3 Procedure:
- 8.1.3.1 If the residues are not to be transferred for weighing, weigh each of the selected sieves and the bottom pan to 0.1 g.

¹⁰ Ro-Tap Sieve Shaker has been found suitable for this purpose and is available from W. S. Tyler Co., Cleveland, Ohio 44114. An equivalent may be used.

Nest the desired sieves in order of diminishing openings with the coarsest sieve on top and the pan on the bottom.

8.1.3.2 Weigh out a 100 ± 0.1 -g specimen and transfer it to the top sieve of the stack.

Note 2—If necessary this test may be made on a specimen of any size from 50 to 200 g. The weight of specimen used shall be stated in the report.

8.1.3.3 Place the cover on the top sieve, and place the stack in the mechanical sieve shaker. Operate the shaker for 10 min \pm 15 s

8.1.3.4 After shaking, carefully separate the stack of sieves, beginning at the top, and weigh the quantity of material retained on each sieve and that contained in the pan to 0.1 g. This may be accomplished either by transferring the fractions to the balance or by weighing the sieve or pan and its contents, and subtracting the tare weight from the total. If the material is transferred to the balance, carefully brush the sieve on both sides to ensure that adhering particles are transferred.

8.1.3.5 Calculate the percent in each fraction as follows:

$$\% = (R/S) \times 100 \tag{1}$$

where:

R = residue weight, g, and

S = specimen weight, g.

Note 3—Ordinarily there is a small loss of dust as indicated by the cumulative total weight being less than 100 %. If this loss is not over 2 %, the amount reported through the finest sieve shall be increased until the total of all portions of the sample equals 100 %. If the cumulative total is less than 98 %, repeat the test.

8.1.4 *Report*—The report shall include the following:

8.1.4.1 Complete identification of the sample,

8.1.4.2 Specimen weight,

8.1.4.3 Percent of material retained on each sieve,

8.1.4.4 If required, the total cumulative percent of material retained on each sieve and in the pan,

8.1.4.5 Temperature, and

8.1.4.6 Relative humidity.

8.2 Vacuum Sieve Analysis:

8.2.1 Summary of Method:

8.2.1.1 In this method sieving is accomplished by aerodynamics so that there is no reduction in particle size and absolutely no wear on the sieves.

8.2.1.2 The housing holds a standard 203.2-mm (8.0-in.) sieve and a transparent sieve cover. A slit nozzle rotates slowly below the sieve. An air current, produced by a standard vacuum cleaner-type device, is blown upwards through a hollow shaft and the slit nozzle to the sieve, where it clears the screen. The particles thus suspended in air between the sieve and the sieve cover are separated as the air current circulates. The fine materials are blown through the sieve and into a filter bag, while the coarse materials remain on top of the sieve. A manometer indicates the vacuum inside the housing.

8.2.2 *Apparatus*:

8.2.2.1 *Sieves*—As described in 8.1.2.1 and transparent sieve cover.

8.2.2.2 Vacuum Sieve Apparatus-A vacuum sieving de-

vice¹¹ capable of maintaining inside the housing a vacuum of 5 ± 0.5 in. (127 ± 12.7 mm) of water as measured by a manometer. The slit nozzle which rotates below the sieve shall revolve at a speed of 25 ± 2 rpm.

8.2.2.3 Balance—See 8.1.2.3.

8.2.3 Procedure:

8.2.3.1 If the residues are not to be transferred for weighing, weigh each of the selected sieves to 0.1 g.

8.2.3.2 Weigh out a 100 ± 0.1 g specimen and transfer it to one of the sieves.

Note 4—If necessary, this test may be made on a specimen of any size from 10 to 50 g. The weight of specimen used shall be stated in the report. For small sieve openings, 1 to 10 g is recommended.

8.2.3.3 Place the transparent cover on the sieve and place the sieve in position on the vacuum sieving apparatus. Operate the sieving apparatus for 5 min \pm 15 s at a vacuum of 5 \pm 0.5 in. (127 \pm 12.7 mm) of water.

8.2.3.4 Remove the sieve and cover from the apparatus and weigh the quantity of material retained on the sieve and adhering to the cover to the nearest 0.1 g. This may be accomplished either by transferring the retained material to the balance or by weighing the sieve, cover, and contents, and subtracting the tare weight from the total. If the material is transferred to the balance carefully brush the sieve on both sides, and removed all powder from the cover to ensure that all adhering particles are transferred.

8.2.3.5 Repeat the procedure for each sieve used.

8.2.4 Calculate the percent retained on each sieve as follows:

$$(2)$$

where:

 R_{\perp} = residue weight, g, and

S = specimen weight, g.

8.2.5 *Report*—The report shall include the following:

8.2.5.1 Complete identification of the sample,

8.2.5.2 Specimen weight,

8.2.5.3 Percent of material retained on each sieve,

8.2.5.4 Temperature, and

8.2.5.5 Relative humidity.

8.3 Sonic Sifter Analysis:

8.3.1 Summary of Method:

8.3.1.1 In the sonic sifter, the sieves are stationary and agitation is imparted to the particles by an oscillating column of air. Sieve wear and particle attrition are minimal.

8.3.1.2 The sonic sifter consists of a sieving chamber, a diaphragm at the top vibrating at 60 Hz, and a motor with the necessary controls. The amplitude of vibration is adjustable to the nature of the specimen.

8.3.2 Apparatus:

8.3.2.1 *Sieves*—See 8.1.2.1.

8.3.2.2 Sonic Sifter—A sonic sieving device¹² utilizing a

¹¹ Alpine Air Jet Sieve, Model 200, has been found suitable for this purpose and is available from Alpine American Corp., Michigan Drive, Natick, Mass. 01760. An equivalent may be used.

¹² Model L3P Sonic Sifter has been found suitable for this purpose and is available from ATM Corp., P.O. Box 2405, Milwaukee, Wisc. 53214. An equivalent may be used.

diaphragm vibrating at 60 Hz with controls to adjust the sift amplitude and the pulse amplitude.

- 8.3.2.3 *Balance*—An analytical balance with a minimum capacity of 150 g sensitive to 1 mg for weighing the specimen and the residues retained on the sieves.
 - 8.3.3 Procedure:
- 8.3.3.1 Weigh and record the weight of each sieve, and of the collar, cover, and fines collector.
 - 8.3.3.2 Weigh out a 2.5 \pm 0.001-g specimen.
- 8.3.3.3 Stack the sieves in descending order, coarsest on top, finest on the bottom, and transfer the specimen to the top sieve.
- 8.3.3.4 Insert the sieve stack assembly in the sieve chamber and lock. Set the sift amplitude at 3 and the pulse amplitude at 10. Operate the sifter for 3 min \pm 15 s.
- 8.3.3.5 Weigh each sieve, the fines collector, cover, and the collar, subtract the tare weight for each, and calculate the percent retained on each sieve.

NOTE 5—Include any material adhering to the cover and the collar in the weights for the top sieve.

- 8.3.4 *Report*—The report shall include the following:
- 8.3.4.1 Complete identification of the sample,
- 8.3.4.2 Specimen weight,
- 8.3.4.3 Percent of material retained on each sieve,
- 8.3.4.4 Temperature, and
- 8.3.4.5 Relative humidity.
- 8.4 Electronic Counting Analysis:
- 8.4.1 Summary of Method:
- 8.4.1.1 This method, one of several found valuable for the measurement of particle size, covers the determination of the particle size distribution of powder coatings (0.6 to $80.0~\mu m$) using the electronic particle counter. This instrument uses 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.
- 8.4.1.2 The powder is carefully dispersed ultrasonically or by some other appropriate method and a portion is added to a filtered electrolyte. Particle counts are obtained at several selected particle size levels and converted to relative weight fractions assuming constant particle density.
- 8.4.1.3 The instrument response is essentially to particle volume (liquid displacement); therefore equivalent spherical diameter is commonly used to express the particle size.
 - 8.4.2 *Apparatus*:
- 8.4.2.1 *Electronic Particle Counter*, ¹³ detecting particles using electric current through a liquid resistor.
- 8.4.2.2 *Aperture Tube*, 200-µm diameter. 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.
- 8.4.2.3 *Beaker*, 250 to 500-mL, capable of maintaining all particles uniformly in suspension (for example, baffled or round bottom).

- 8.4.2.4 Ultrasonic Agitator.
- 8.4.2.5 Membrane Filtering Device, 0.45-µm filters. 14
- 8.4.3 Reagents:
- 8.4.3.1 *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. ¹⁵ 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.
- 8.4.3.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.
- 8.4.3.3 *Dispersing Medium*—5 % solution of octyl phenoxy polyethoxyethanol¹⁶ in reagent water.
- 8.4.3.4 *Electrolyte*—Dissolve 10.0 g of reagent grade sodium chloride (NaCl) in 1000 mL of reagent water, add some bactericide such as 70 mg of sodium trichlorophenate then filter twice through the membrane (8.1.2.1).
 - 8.4.3.5 Wash Water—Reagent water filtered twice.
 - 8.4.4 Procedure:
- 8.4.4.1 Precalibrate the aperture and electrolyte combination following the manufacturer's instruction manual.
- Note 6—Calibration should be carried out using a lycopodium of microspheres suspension of 18.04- μm diameter with the half-count procedure as given in the instruction manual. It is necessary to have mutual agreement on monosized system diameters for interlaboratory comparisons.
- 8.4.4.2 Cone and quarter the sample until a specimen of convenient size is obtained.
- 8.4.4.3 Disperse the specimen by adding dispersing medium from the eye-dropper. Use gentle spatulation until a pasty consistency is obtained. Pick up a small portion of this mixture (about 0.1 mL more or less depending on the particle size of the powder) on a small spatula and rinse with wash water into a 25-mL beaker containing 5 mL of dispersing medium. Place the beaker in the ultrasonic agitator for 30 s and then rinse into 200 mL of electrolyte in the 250 to 500-mL beaker. Fill the beaker to capacity and place in the counting position of the counter stand.
- 8.4.4.4 Fill the aperture tube with electrolyte. Check the aperture for blockage and clear with a brush if necessary. Set the instrument controls for the smallest particle appropriate for the aperture in use. Typical instrument settings are shown in Fig. 1, Fig. 2, or Fig. 3.
- 8.4.4.5 Take three cumulative counts with 500- μL volume manometer and calculate the mean, which should be between 2700 and 5000. If it is not, adjust the particle concentration

¹³ Models ZB with M2 accessory, Model B with Model M accessory, Model T, Model TA, and Model TAII have been suitable for this purpose and are available from Coulter Electronics, Inc., 590 W. 20 St., Hialeah, FL 33010. An equivalent may be used.

¹⁴ The membrane filters may be Millipore, Gelman, General Electric, Nuclepore, Selas Flotronics metal type, or their equivalent.

¹⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

¹⁶ Triton X-100 has been found suitable for this purpose and is available from Rohm and Haas Co., Independence Square, Philadelphia, PA. An equivalent may be used.



INDUSTRIAL Model "B" or "Z" DATA AND WEIGHT CONVERSION MODEL "M" or "M2" Converter

ample							Source	_ Source				Electrolyte				Date		
Aperture Diameter 200		μ	Man Volu	ometer 500 µl			ļ	Source Calib. Matching 10K (Model B Calibrat Switch 128L Model B Data			Dispersant Triton X-100				Oner			
ertu	re		000	Ω	Gain Control	95	Matchii Switch	ng 128L M	odel B	Calibrat Data	ion t _L =14 1	/3, I=1,	A=1/2	for 18.04	4m (3074.0	Mm		
		<u> </u>		CONVERTER		ER	RELATIVE VOLUME VALUES		ES	V, =	d,=	d	RELATIVE An V		Wt			
t _L t _U	I	A	Т	D	R		RAW		AVE.	$V_{L} = k \cdot t_{L} \cdot I \cdot A$	$1.241\sqrt[3]{\overline{V_L}}$	a	ΔnV	ΣΔnV	%			
20	40	1	32	128	4	0						80.6						
20	40	1	16	64	4	1						64.0						
20	40	1	8	32	4	2						50.8	_					
20	40	1	4	16	4	3						40.3						
20	40	1	2	8	4	4						32.0						
20	40	1	1	8	2	5						25.4				<u> </u>		
20	40	1	1/2	8	1	6						20.2			1	<u> </u>		
20	40	1/2	1/2	8	1/2	7						16.0				ļ		
20	40	1/2	1/4	8	1/4	8	.,					12.7						
20	40	1/4	1/4	8	1/8	9						10.08						
20	40	1/4	1/8	8	1/16	10		ļ				8.00	, ·· · ·			<u> </u>		
20	40	1/8	1/8		1/32	11						6.35						
10	20	1/8	1/8	8	1/64	12						5.04						
5	10	1/8	1/8	8	1/128	13		ļ				4.00*				ļ		
											ļ					<u> </u>		
												ļ				<u> </u>		
								ļ			<u> </u>					<u> </u>		
				ļ				277										
_												<u>S</u>			ļ	 		
															 	<u> </u>		
				ļ			1111	IS://	3191	ndai	ed s i	toh	ai'			 -		
	ļ					1		5.77	5 0 0 0 1	1 (120)	42001				ļ	 		
				L						 I		/NN=/	 			ļ		
			ļ					VUL				CAA			ļ	 		
											SAME	VALUES			<u>42210</u>	I		

Note 1—Typical setting for smallest particle (8.4.4.4) but upper threshold must be turned to full counterclockwise position for cumulative count in 8.4.4.5. https://standards.iteh.ai/catalog/standards/sist/bff97e62-a85f-4522-8ed0-53107d1427e1/astm-d3451-92

FIG. 1 Model "B" or "Z" Typical Worksheet.

until it is. If more powder is added, it must be treated the same as the original specimen, except it may be rinsed directly into the beaker after ultrasonic agitation. If the suspension needs dilution, drain some of the suspension through a clean glass tube while agitating and then refill the beaker with electrolyte.

8.4.4.6 Set the instrument controls for normal accumulation of differential data. Typical instrument settings are shown in Fig. 1, Fig. 2, and Fig. 3. Take three counts at each size level for averaging.

8.4.5 Precautions:

8.4.5.1 Check the aperture for blockage at the end of each count. If blocked clear the aperture with the brush and retake the count.

8.4.5.2 Adjust the sample stand stirrer motor speed to furnish sufficient agitation to maintain a uniform particle suspension but below air bubble generation speeds.

8.4.5.3 Before each analysis flush all the surfaces coming in contact with the specimen. Use clean wash water and wash bottle.

8.4.6 Treatment of Result:

8.4.6.1 The values obtained in 8.4.4.6 represent relative volume of the particles between two particle size levels. They

may be plotted directly on semilog graph paper as a histogram (see Fig. 4).

8.4.6.2 The values obtained in 8.4.4.6 may be added cumulatively. All of these cumulative values may then be normalized to 100 % and a cumulative volume percent curve drawn (see Fig. 4).

8.4.6.3 Relative volume and relative weight are interchangeable if a constant density of the particle system is assumed. Therefore, the curves in 8.4.6.1 and 8.4.6.2 can be interpreted as weight histogram and weight percent curves.

9. Package Stability

- 9.1 Glass Vial Method:
- 9.1.1 Apparatus:
- 9.1.1.1 Aluminum-Foil Disk.
- 9.1.1.2 Steel Shot.
- 9.1.1.3 Glass Vial, 4-oz (120-ml), approximately 40-mm inside diameter.
 - 9.1.1.4 Balance, sensitive to 0.1 mg.
- 9.1.1.5 *Oven*, maintained at 50 ± 0.5 °C unless otherwise agreed upon between the purchaser and the supplier.
 - 9.1.1.6 Particle Size Determination Apparatus—See 8.