Designation: E2980 – 15

Standard Test Methods for Estimating Average Particle Size of Powders Using Air Permeability[1]

This standard is issued under the fixed designation E2980; 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 test methods use air permeability to determine an envelope-specific surface area and its associated average equivalent spherical diameter (from 0.2 to 75 µm) of powders. Two test methods are described: One test method for inorganic materials (Test Method 1), and another test method for organic materials (Test Method 2). The values obtained are not intended to be absolute but are generally useful on a relative basis for control purposes.

1.2 *Units*—With the exception of the values for density and the mass used to determine density, for which the use of the gram per cubic centimetre (g/cm^3) and gram (g) units is the longstanding industry practice; and the units for pressure, cm H_2O—also long-standing practice; the values in SI units are to be regarded as standard.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B330 Test Methods for Estimating Average Particle Size of Metal Powders and Related Compounds Using Air Permeability

B859 Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E1638 Terminology Relating to Sieves, Sieving Methods, and Screening Media

E2589 Terminology Relating to Nonsieving Methods of Powder Characterization

2.2 *ISO Documents:*[3]

ISO 10070 Metallic Powders: Determinations of Envelope-Specific Surface Area from Measurements of the Permeability to Air of a Powder Bed Under Steady-State Flow Conditions

3. Terminology

3.1 *Definitions*—Many terms used in this test method are defined in Terminologies E1638 and E2589.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air permeability, n*—measurement of air pressure drop across a packed bed of powder.

3.2.2 *average particle size, n—for the purposes of these test methods only,* an estimate of the equivalent average spherical particle diameter, calculated from the measured envelope-specific surface area, assuming that all the powder particles are spherical and that all are exactly the same size.

3.2.3 *de-agglomeration, n*—process used to break up agglomerates of particles.

3.2.4 *envelope-specific surface area, n*—specific surface area of a powder as determined by gas permeametry in accordance with ISO 10070.

3.2.5 *MIC Sub-Sieve AutoSizer (MIC SAS), n*—a commercially available permeability instrument for measuring envelope-specific surface area and estimating average particle size from 0.2 to 75 µm.

3.2.6 *porosity of a bed of powder, n*—ratio of the volume of the void space in the powder bed to that of the overall volume of the powder bed.

4. Significance and Use

4.1 These test methods provide procedures for determining the envelope-specific surface area of powders, from which is calculated an "average" particle diameter, assuming the particles are monosize, smooth surface, nonporous, spherical

[1] These test methods are under the jurisdiction of ASTM Committee E29 on Particle and Spray Characterization and is the direct responsibility of Subcommittee E29.02 on Non-Sieving Methods.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

***A Summary of Changes section appears at the end of this standard**

particles. For this reason, values obtained by these test methods will be reported as an average particle size. The degree of correlation between the results of these test methods and the quality of powders in use will vary with each particular application and has not been fully determined.

4.2 These test methods are generally applicable to all powders, including metals (see Test Methods B330 for specific metal powder requirements), ceramics, and organic materials, for particles having diameters between 0.2 and 75 μm. They should not be used for powders composed of particles whose shape is too far from equiaxed - that is, flakes or fibers. In these cases, it is permissible to use the test methods described only by agreement between the parties concerned. These test methods shall not be used for mixtures of different powders, nor for powders containing binders or lubricants. When the powder contains agglomerates, the measured surface area may be affected by the degree of agglomeration. Methods of deagglomeration such as that specified in Practice B859 may be used if agreed upon between the parties concerned.

4.3 When an "average" particle size of powders is determined by these methods, it should be clearly kept in mind that this average size is derived from the determination of the specific surface area of the powder using a relationship that is true only for powders of uniform size and spherical shape. Thus, the results of these methods are only *estimates* of average particle size.

4.4 Reported particle size measurement is a function of both the actual dimension and shape factor as well as the particular physical or chemical properties of the particle being measured. Caution is required when comparing data from instruments operating on different physical or chemical parameters or with different particle size measurement ranges. Sample acquisition, handling, and preparation can also affect reported particle size results.

5. Apparatus

5.1 *MIC Sub-Sieve AutoSizer (MIC SAS),*[4] consisting of an air pump, a calibrated gas mass flow controller, a precision-bore sample tube, a sample tube retaining collar, a spacer tool, a gas flow metering valve, two precision pressure transducers (inlet and outlet), a stepper motor controlled ballscrew-mounted piston, and computer hardware and software for instrument control and calculation and reporting of results. Included is accessory equipment consisting of a plug manipulator (extraction rod), two porous plugs, and a supply of paper disks.

NOTE 1—When homing the piston, adjust the sample packing assembly (*1*) as described in the manufacturer's directions, with the plugs and paper disks stacked together and placed on the fixed anvil spigot, or (*2*) using a specially designed baseline (homing) gauge instead of the plugs and paper disks. This baseline gauge shall have a height of 20.30 ± 0.10 mm. Check all plug heights when new plugs are purchased and periodically thereafter to make sure all are equal in height.

5.2 *Powder funnel,* stainless steel, with spout outside diameter slightly smaller than the sample tube inside diameter.

5.3 The manufacturer provides instructions which should be followed. Particular attention should be given to proper maintenance of the instrument with special reference to the instructions on (*1*) "homing" the piston when turning on from an unpowered state, (*2*) setting the pressure and periodic checking of the pressure, (*3*) condition of O-rings on the piston and sample spigot, and (*4*) the sample packing assembly (plugs and paper disks).

5.4 *Balance,* having a capacity of at least 50 g and a sensitivity of 0.001 g.

6. Standardization of Apparatus

6.1 Before proceeding with standardization of the MIC SAS instrument, the following items shall be checked:

6.1.1 The sample tube and plugs shall not be worn to the point where results are affected.

6.1.2 Inspect the O-ring seals for tears and abrasion marks. The O-ring seals shall not be worn to the point where the sample tube moves easily by hand or the pressure reading varies as the sample tube is moved.

6.1.3 The drying agent shall be in proper condition.

6.2 Whenever the instrument is turned on from an unpowered state, the piston shall be "homed" according to the manufacturer's instructions. See Note 1 above.

6.3 Before running the initial sample, the pressure shall be set to 50.0 (+0.1, –0.5) cm H_2O, using the metering valve; then checked and reset if necessary every few hours, or if the ambient temperature changes more than ±2°C.

NOTE 2—The metering valve position should not be adjusted for repeat runs of the same sample as this will likely lead to a loss of precision even if the inlet pressure reading has drifted a little outside the 50.0 (+0.1, –0.5) cm H_2O range. Further adjustment is not necessary as the pressure is controlled precisely during the particle size measurement.

6.4 Standardization is recommended before and after any series of determinations or at least every 4 hours of continued operation. Warm-up of the instrument is required if it has been off for more than 30 minutes.

6.5 Calibration of the pressure transducers is recommended every 3–6 months, using a traceable external pressure gauge per the manufacturer's instructions.

7. Procedure

7.1 *Temperature of Test*—Make average particle size determinations within ±2°C of the temperature at which standardization of the MIC Sub-Sieve AutoSizer was made. Reset the pressure if the temperature of the test varies more than ±2°C.

7.2 *Size of Test Sample*—The mass of sample used for tests shall be equal in grams (within ±5 %) to the true (pore-free) density (in g/cm^3) of the powder (for example, iron, 7.8 g; tungsten, 19.3 g; molybdenum, 10.2 g; tantalum, 16.6 g; nickel, 8.9 g; and so forth).

[4] The sole source of supply of the MIC Sub-Sieve AutoSizer (MIC SAS) known to the committee is Micromeritics Instrument Corporation, Particulate Systems, 4356 Communications Drive, Norcross, GA 30093-2901, USA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

7.3 *Average Particle Size Determination*—The average particle size determination shall be made by the same operator who makes the standardizations and is started after standardization or the determination of another sample. Proceed according to the MIC SAS manufacturer's instructions as follows:

7.3.1 *Test Method 1 – Inorganic Materials (For Example: Metals, Ceramics, Metal Oxides, Metal Carbides):*

7.3.1.1 Press the **"Inorganics"** button.

7.3.1.2 Determine the mass of the sample to the nearest 0.01 g.

7.3.1.3 Select the test parameters: **3** compressions; **slow** decompression; **slow** termination.

7.3.1.4 Proceed to 7.3.3.

7.3.2 *Test Method 2 – Organic Materials (For Example: Lactose):*

7.3.2.1 Press the **"Organics"** button.

7.3.2.2 Determine the mass of the sample to the nearest 0.01 g.

7.3.2.3 Select the test parameters—Starting Porosity, Step Size, Final Porosity—previously determined for the particular material being tested.

7.3.2.4 Proceed to 7.3.3.

7.3.3 Press the **"Run Test"** button and enter the Sample Details, including the true density of the material and the actual mass of the sample used.

7.3.4 Lay a paper disk over one end of the sample tube using one of the porous plugs with the perforated surface of the plug against the surface of the paper disk. This crimps the paper around the edges and the paper precedes the plug into the sample tube. Push the plug into the tube until it is even with the end of the sample tube. Place the sample tube in a vertical position in a support with the paper side of the plug up.

7.3.5 With the aid of the powder funnel, completely transfer the sample into the sample tube by tapping the side of the tube and funnel. Lay a second paper disk over the top of the sample tube. Place the perforated surface of a porous brass plug on top of the paper disk and force the plug and paper disk down into the sample tube until the plug is just inside the sample tube.

7.3.6 Push the sample tube retaining collar onto the sample tube.

7.3.7 Push the sample tube onto the fixed anvil spigot with the retaining collar below the sample tube holder, centered in the sample tube holder and leaving enough of a gap at the bottom of the sample tube to fit the SAS spacer tool below the sample tube.

NOTE 3—The sample tube may eventually wear and cause faulty values. When this condition is suspected, replace the tube. Sample tubes with obvious wear or scratches, or both, should be discarded.

7.3.8 Insert the SAS spacer tool into the gap below the sample tube.

7.3.9 Using an Allen key or cam lock device, lock the sample tube retaining collar into position just below the sample tube holder arms.

7.3.10 Press the **"Next"** button and the test will automatically run.

7.3.11 Monitor the test and remove the spacer after the first compression (Test Method 1, Inorganic Materials) or after the piston has engaged (Test Method 2, Organic Materials). (**Warning—**The piston moves slowly but with considerable force. Keep all body parts clear of the mechanism while in motion. Do not operate with any guards removed.)

NOTE 4—The sample tube must be held off the spigot to ensure that the full force is applied to the sample and not dissipated through the spigot.

7.3.12 When the test is finished, the results will be displayed on the instrument's screen. Record the Porosity, (Average) Particle Size, and Specific Surface Area (SSA). The data will automatically be saved with the file name indicated during entry of the sample details.

NOTE 5—A calculation of an equivalent spherical diameter ("average particle diameter," "average particle size"), based on the relationship between envelope-specific surface area and particle diameter, is automatically performed by the MIC Sub-Sieve AutoSizer from the values related to the porosity and to the permeability of the powder bed measured by the instrument. In other words, what is determined with the instrument is the specific surface area of the powder. When an equivalent spherical diameter is determined using the MIC Sub-Sieve AutoSizer, it should be clearly kept in mind that this equivalent spherical diameter is derived from the determination of the specific surface area of the powder using a relationship that is true only for powders of uniform size and spherical shape. Hence, the term "average particle size," as defined in 3.2.2, is preferred to describe the result from this instrument, rather than "particle size" or "equivalent spherical diameter."

7.3.13 For later data extraction, refer to the manufacturer's instructions.

8. Report

8.1 Report the following information:

8.1.1 Reference to this standard.

8.1.2 Whether Test Method 1 (Inorganic Materials) or Test Method 2 (Organic Materials) was used.

8.1.3 All details necessary for identification of the test specimen, including whether the powder was de-agglomerated or milled in the laboratory before analysis, using a procedure such as that in Practice B859. If a laboratory method other than Practice B859 is used to de-agglomerate or mill the powder, sufficient information to describe the procedure completely shall also be included with the results. In any case of de-agglomeration by laboratory milling, identify the powder as "lab milled." Otherwise, identify the powder as "as-supplied."

8.1.4 For Test Method 1 (Inorganic Materials), report:

8.1.4.1 The average particle size (to two decimal places for average particle sizes less than 10 µm, or to one decimal place for average particle sizes greater than 10 µm).

8.1.4.2 The measured porosity of the packed sample (to the nearest 0.01).

8.1.5 For Test Method 2 (Organic Materials), report:

8.1.5.1 The final ("finished") data table of particle size and surface area versus porosity.

8.1.5.2 The particle size (to one decimal place for average particle sizes greater than or equal to 10 µm, or to two decimal places for average particle sizes less than 10 µm).

8.1.5.3 The surface area (to two decimal places) at the final porosity.

8.1.5.4 The final porosity (to the nearest 0.01).

8.1.5.5 All reported numerical values shall be rounded in accordance with Practice E29.

9. Precision and Bias

9.1 *Precision:*

9.1.1 *Repeatability*—The repeatability standard deviation for Test Method 1 (Inorganic Materials), based on repetitive testing of a single sample in the same laboratory, has been determined to be: 0.013 µm at an average particle size of 1.08 µm; 0.021 µm at an average particle size of 2.75 µm; and 0.042 µm at an average particle size of 4.02 µm.

9.1.2 *Reproducibility*—The reproducibility is being determined and will be available on or before December 31, 2019.

9.2 *Bias*—No information can be presented on the bias of the procedures in these test methods for estimating average particle size because no material having an accepted reference value is available.

10. Keywords

10.1 air permeability; average particle size; envelope-specific surface area; particle size; permeability; porosity; powder; specific surface

SUMMARY OF CHANGES

Committee E29 has identified the location of selected changes to this standard since the last issue (Dec. 1, 2015) that may impact the use of this standard. (Approved Dec. 1, 2015.)

(1) Deleted all references to "HEL," changed to "MIC."
(2) Revised Footnote 4 to indicate Micromeritics as the sole source.
(3) Added "Summary of Changes" section.