Designation: B330 – 20

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

This standard is issued under the fixed designation B330; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

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 75μm) of metal powders and related compounds. The powders may be analyzed in their "as-supplied" (shipped, received, or processed) condition or after they have been de-agglomerated or milled by a laboratory procedure ("lab milled") such as that specified in Practice B859. 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B243 Terminology of Powder Metallurgy

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

E456 Terminology Relating to Quality and Statistics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO/DIS Document:*[3]

ISO/DIS 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 these test methods are defined in Terminology B243.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *agglomerate, n*—several particles adhering together.

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

3.2.3 *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.

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

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

3.2.6 *Fisher calibrator tube, n*—jewel with a precision orifice mounted in a tube similar to a sample tube.

3.2.6.1 *Discussion*—The calibrator tube value is directly traceable to the master tube maintained by ASTM International Subcommittee B09.03 on Refractory Metal Powders.

[1] These test methods are under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and are the direct responsibility of Subcommittee B09.03 on Refractory Metal Powders.

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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

3.2.7 *Fisher Number, n*—calculated value equated to an average particle diameter, assuming all the particles are spherical and of uniform size.

3.2.8 *Fisher Sub-Sieve Sizer (FSSS), n*—a commercially available permeability instrument for measuring envelope-specific surface area and estimating average particle size (Fisher Number) from 0.5 to 50 µm.

3.2.9 *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.

3.2.10 *porosity of a bed of powder, n*—ratio of the volume of the void space in the powder bed to the 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 particles. For this reason, values obtained by these test methods will be reported as an average particle size or Fisher Number. 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 metal powders and related compounds, including carbides, nitrides, and oxides, for particles having diameters between 0.2 and 75 µm (MIC SAS) or between 0.5 and 50 µm (FSSS). 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 de-agglomeration 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 either the MIC SAS or the FSSS, 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.

5. Apparatus

5.1 *MIC Sub-sieve AutoSizer (MIC SAS)*[4]—Method 1, 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. See schematic diagram in Fig. 1.

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.

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

5.1.2 The manufacturer provides instructions which should be followed, using the *"Inorganics Test"* procedure when testing metal powders and related compounds. 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).

5.2 *Fisher Sub-Sieve Sizer (FSSS)*[5]— Method 2, consisting of an air pump, an air-pressure regulating device, a precision-bore sample tube, a standardized double-range air flowmeter, and a calculator chart. Included is accessory equipment consisting of a plug manipulator, powder funnel, two porous plugs, a supply of paper disks, and a rubber tube support stand.

NOTE 2—Necessary replacement parts should be obtained from the manufacturer, especially in the case of the precision manometer which is a part of the air flowmeter.

5.2.1 The manufacturer has also furnished instructions which should be followed, except as amended as follows. Particular attention should be given to proper maintenance of the instrument with special reference to the instructions on (*1*) periodic checking of the water level in the pressure regulator standpipe, (*2*) manometer level before the sample tube is inserted, and (*3*) the sample packing assembly.

5.2.2 *Jewel Calibrator Tube*[6]—A tube to be used as a standard for average particle size measurement. It allows operators to relate their data to that of other analysts. Each calibrator has been factory tested three times with the resulting readings and associated porosity recorded on the tube.

NOTE 3—Adjust the sample packing assembly (*1*) as described in the manufacturer's instructions with the exception that the plugs and paper disks are not inserted in the sample tube, but are merely stacked together

[4] The sole source of supply of the MIC Sub-sieve AutoSizer (latest version called MIC SAS II) 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.

[5] The Fisher Sub-Sieve Sizer (FSSS) is no longer commercially available, nor is it supported with parts and service. It is included here as apparatus for Method 2 because of several instruments still operating in the field. In-house repair or parts replacement is discouraged, as these are likely to detrimentally affect results and precision.

[6] The Jewel Calibrator Tube is no longer commercially available. A "Master" Jewel Calibrator Tube is maintained by ASTM International Subcommittee B09.03 for calibration and traceability of currently existing in-house calibrator tubes.

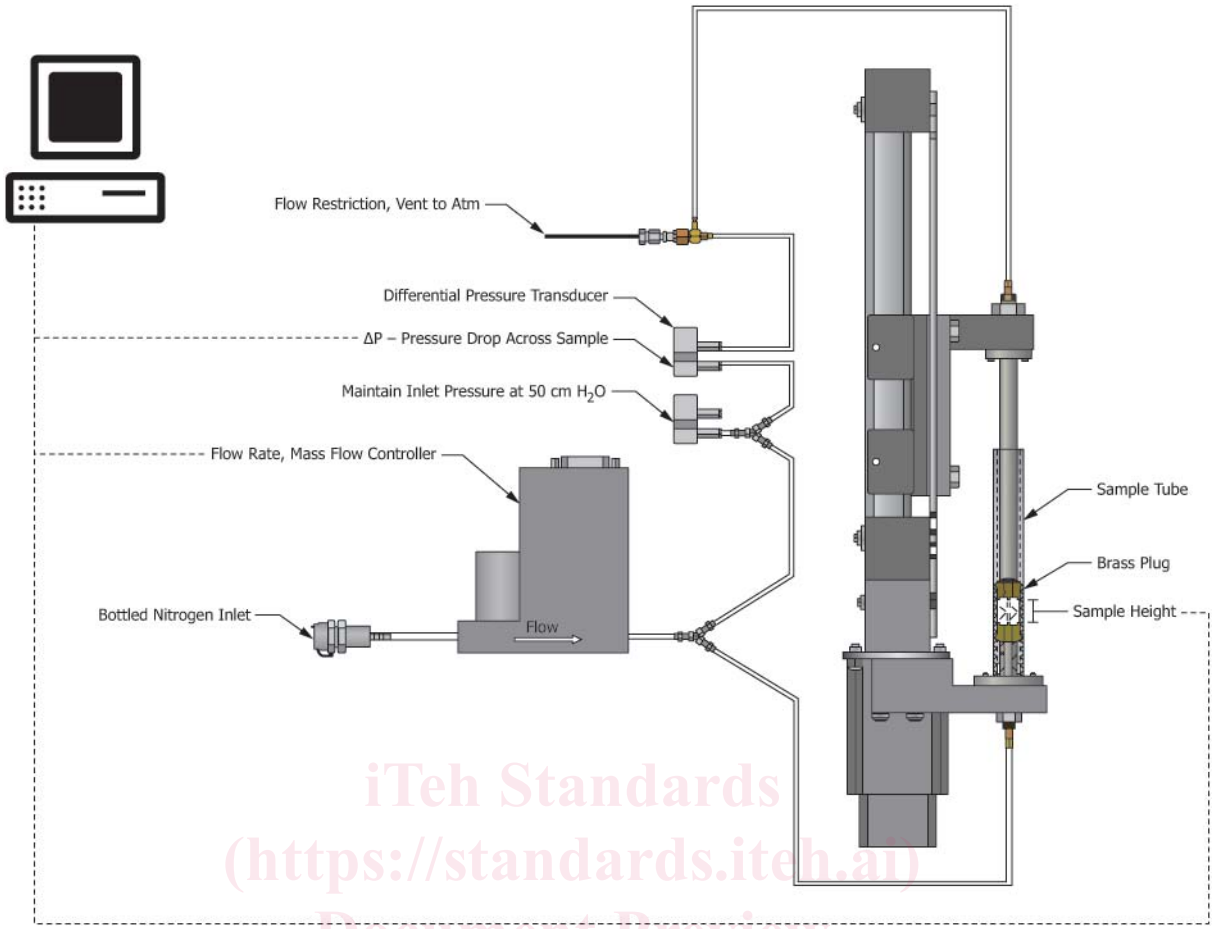

FIG. 1 Schematic Diagram of the MIC Sub-sieve AutoSizer (MIC SAS)

and placed between the brass support and the "flat" of the bottom of the rack; and (*2*) as previously described, except that a specially made baseline gauge is used instead of the plugs and paper disks. This baseline gauge shall have a height of 19.30 ± 0.10 mm. Check all plug heights when new plugs are purchased and periodically thereafter to make sure all are equal in height.

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

6. Standardization of Apparatus

6.1 *Method 1 – MIC Sub-sieve AutoSizer (MIC SAS):*

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

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

6.1.1.2 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.

6.1.1.3 The drying agent shall be in proper condition.

6.1.2 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.

6.1.3 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 4—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.

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

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

6.2 *Method 2 – Fisher Sub-Sieve Sizer (FSSS):*

6.2.1 Before proceeding with standardization of the FSSS, the following items shall be checked:

6.2.1.1 The chart shall be properly aligned horizontally with the indicator pointer.

6.2.1.2 The rack and pinion shall be properly aligned vertically with the chart.

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

6.2.1.4 The manometer and air resistors shall be free of visible contamination.

6.2.1.5 The rubber sample tube seals shall not be worn to the point where leakage occurs.

6.2.1.6 The sample packing post shall be properly adjusted.

6.2.1.7 The drying agent shall be in proper condition.

6.2.1.8 The manometer and standpipe levels shall be checked.

6.2.1.9 Adjust the manometer only when the machine is not operating and with the pressure released for minimum of 5 min to allow the manometer tube to drain completely.

6.2.2 The standardization of the Fisher Sub-Sieve Sizer shall be made using the Fisher jewel calibrator tube (jewel orifice tube) as the primary standard. Specification shall be made at both ranges of the machine. The Fisher jewel calibrator tube used for standardization shall be checked under a microscope at least once a month to determine the condition and cleanliness of the orifice. If the orifice is not clean, clean as described in the Fisher Sub-Sieve Sizer instruction manual.

6.2.3 With the sub-sieve sizer properly adjusted and set to the proper range, proceed as follows:

6.2.3.1 Mount the Fisher jewel calibrator tube between the rubber seal supports just to the right of the brass post. Clamp the upper cap down onto the tube so that an airtight seal is obtained at both ends.

6.2.3.2 Adjust the calculator chart so that the porosity reading corresponds to the value indicated on the jewel calibrator tube.

6.2.3.3 Switch on the instrument and allow it to warm up for a minimum of 20 min. Adjust the pressure-control knob, located near the bubble observation window at the lower left of the panel, until the bubbles rise in the standpipe at the rate of two to three bubbles per second. This will cause the water line to rise above the calibration mark on the upper end of the standpipe. This is normal and does not mean the calibration is in error.

6.2.3.4 The liquid level in the manometer tube will rise slowly until it reaches a maximum. Allow at least 5 min for this to happen. At the end of this period, using care not to disturb the chart, turn the rack up until the upper edge of the crossbar coincides with the bottom of the liquid meniscus in the manometer. The Fisher Number is indicated by the location of the pointer tip in relation to the curves on the calculator chart. Record the ambient temperature to the nearest 1 °C. Release the clamp on the upper end of the tube slowly so the manometer returns to its zero position slowly with very little overshoot. This limits the formation of liquid droplets on the inside of the manometer tube.

6.2.3.5 The value obtained in this manner must correspond to the Fisher Number indicated on the jewel calibrator tube within ±1 %.

6.2.3.6 If the Fisher Number value as indicated on the chart does not correspond to ±1 % of the value indicated on the jewel calibrator tube, calibrate the sub-sieve as follows: Adjust either the high needle valve or the low needle valve as required to bring the Fisher number indicated on the chart to the value indicated on the jewel calibrator tube. After adjustment is made, repeat 6.2.3.3.

6.2.3.7 Because only one flowmeter is used for the low (0.5 to 15.0 µm) Fisher Number range while both flowmeters are used for the high (15.0 to 50.0 µm) Fisher Number range, the low range should be standardized first. After the low range is standardized, the high range is then standardized, making adjustments only to the one flowmeter opened up by the range-control knob.

6.2.3.8 Standardization with the jewel calibrator tube is recommended before and after any series of determinations or at least every 4 h of continued operation. Warm-up of the machine is required if it has been off for more than 30 min.

7. Procedure

7.1 *Method 1 – MIC Sub-sieve AutoSizer (MIC SAS) – 0.2 to 75 µm:*

7.1.1 *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.

7.1.2 *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, tungsten, 19.3 g; molybdenum, 10.2 g; tantalum, 16.6 g; nickel, 8.9 g; and so forth).

7.1.3 *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:

7.1.3.1 Press the **"Inorganics"** button.

7.1.3.2 Determine the mass of the sample to the nearest 0.1 g.

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

7.1.3.4 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.

7.1.3.5 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.

7.1.3.6 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.

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

7.1.3.8 Push the sample tube onto the fixed anvil spigot with the retaining collar below the sample tube holder, centered in