



# Standard Test Methods for Estimating Average Particle Size of Metal Powders and Related Compounds Using Air Permeability<sup>1</sup>

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 ( $\epsilon$ ) 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 75 $\mu\text{m}$ ) 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 ( $\text{g}/\text{cm}^3$ ) and gram (g) units is the longstanding industry practice; and the units for pressure, cm H<sub>2</sub>O - 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*:<sup>2</sup>

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

<sup>1</sup> 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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<sup>2</sup> 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.

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*:<sup>3</sup>

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 this test method are defined in Terminology B243.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *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 $\mu\text{m}$ .

3.2.2 *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  $\mu\text{m}$ .

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

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

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

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

3.2.7 *Fisher calibrator tube, n*—jewel with a precision orifice mounted in a tube similar to a sample tube. The calibrator tube value is directly traceable to the master tube maintained by ASTM International Subcommittee B09.03 on Refractory Metal Powders..

<sup>3</sup> 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

3.2.8 *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.

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

3.2.10 *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.

## 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  $\mu\text{m}$  (MIC SAS) or between 0.5 and 50  $\mu\text{m}$  (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 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 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)*<sup>4</sup>—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

<sup>4</sup> 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,<sup>1</sup> which you may attend.

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  $20.30 \pm 0.10$  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)*<sup>5</sup>—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*<sup>6</sup>—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 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  $19.30 \pm 0.10$  mm. Check all plug heights when new plugs are purchased and periodically thereafter to make sure all are equal in height.

<sup>5</sup> 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.

<sup>6</sup> 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.

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<sub>2</sub>O, using the metering valve; then checked and reset if necessary every few hours, or if the ambient temperature changes more than  $\pm 2^\circ\text{C}$ .

**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<sub>2</sub>O 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 hours of continued operation. Warm-up of the instrument is required if it has been off for more than 30 minutes.

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 micro-

scope 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 minutes. 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 minutes 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  $\pm 1\%$ .

6.2.3.6 If the Fisher Number value as indicated on the chart does not correspond to  $\pm 1\%$  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- $\mu\text{m}$ ) Fisher Number range while both flowmeters are used for the high (15.0- to 50.0- $\mu\text{m}$ ) 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 hours of continued operation. Warm-up of the machine is required if it has been off for more than 30 minutes.

## 7. Procedure

7.1 *Method 1 – MIC Sub-sieve AutoSizer (MIC SAS) – 0.2 to 75  $\mu\text{m}$ :*

7.1.1 *Temperature of Test*—Make average particle size determinations within  $\pm 2^{\circ}\text{C}$  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  $\pm 2^{\circ}\text{C}$ .

7.1.2 *Size of Test Sample*—The mass of sample used for tests shall be equal in grams (within  $\pm 5\%$ ) to the true (pore-free) density (in  $\text{g}/\text{cm}^3$ ) 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 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 5—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.

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

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

7.1.3.11 Press the "Next" button and the test will automatically run.

7.1.3.12 Monitor the test and remove the spacer (washer) after the first compression.

**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 6—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.

7.1.3.13 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 7—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 3.2.10, is preferred to describe the result from this instrument, rather than "particle size" or "equivalent spherical diameter."

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

## 7.2 Method 2 – Fisher Sub-Sieve Sizer (FSSS) – 0.5 to 50 $\mu\text{m}$ :

7.2.1 *Temperature of Test*—Make Fisher Number determinations within  $\pm 2^{\circ}\text{C}$  of the temperature at which standardization of the Fisher sub-sieve sizer was made. Restandardize if the temperature of the test varies more than  $\pm 2^{\circ}\text{C}$ .

7.2.2 *Size of Test Sample*—The mass of sample used for tests shall be equal in grams (within  $\pm 0.01$  g) to the true (pore-free) density of the powder (tungsten, 19.3 g; molybdenum, 10.22 g; tantalum, 16.6 g; nickel, 8.9 g; and so forth).

7.2.3 *Fisher Number Determination* —The Fisher Number determination shall be made by the same operator who makes the standardizations and is started after standardization or the determination of another sample. Proceed as follows:

7.2.3.1 With the sub-sieve sizer properly adjusted, set the range control to the range desired.

7.2.3.2 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.

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

7.2.3.4 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. Place the sample tube on the brass post beneath

the rack and pinion with the lower plug in contact with the upper end of the brass post.

7.2.3.5 Lower the rack, guiding it until the flat-bottom end comes in contact with the upper plug. Pack the sample firmly by turning down the pinion knob with the torque wrench or torque screwdriver until a compressive force of 222 N (50 lbf) is applied to the sample. After this force is applied, the sample tube should not be touching the block in which the brass post is mounted. In cases in which the tube tends to move down and rest on the block during compression, the tube can be held temporarily by hand or a spacer can be used until most of the compressive force has been applied. The spacer is then removed when the maximum force is actually applied. Apply and release maximum force a total of three times. After the final maximum compression force has been applied, check the rack to make sure it has not been removed upward with the final release of pressure. Check torque wrench or torque screwdriver for standardization at least once every month using sample pressure calibrator or an equivalent device.

7.2.3.6 Shift the calculator chart laterally until the extreme tip of the pointer just coincides with the sample-height curve on the chart. The pointer should be midway between the top and bottom of the line. The chart must not be moved after this setting until the determination is finished. Record the porosity value indicated at the bottom of the chart.

7.2.3.7 Without disturbing the sample in any way, mount the sample tube between the rubber-cushioned supports just to the right of the brass post. Clamp the upper cap down onto the sample tube so that an airtight seal is obtained at both ends.

NOTE 8—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.

7.2.3.8 Determine the Fisher Number, switching on the machine and allowing the liquid level in the manometer tube to rise until it reaches a maximum. Allow a minimum of 5 min for this to happen. The Fisher Number is indicated by the location of the tip of the pointer in relation to the curves on the calculator chart. Record this value along with the porosity for the sample and the ambient temperature at which the measurement was made.

NOTE 9—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 represented by the calculator chart of the Fisher Sub-Sieve Sizer 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 Fisher Sub-Sieve Sizer, 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 “Fisher Number” is preferred to describe the result from this instrument, rather than “particle size” or “equivalent spherical diameter.”

## 8. Report

8.1 Report the following information:

8.1.1 Reference to this standard.

8.1.2 Whether *Method 1 (MIC Sub-sieve AutoSizer)* or *Method 2 (Fisher Sub-Sieve Sizer)* 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 in accordance with Practice B859. If another laboratory method is used to deagglomerate or mill the powder, sufficient information to describe the procedure completely must 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 *Method 1 (MIC SAS)*, report the average particle size, rounded per Practice E29 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.

8.1.5 For *Method 2 (FSSS)*, report the Fisher Number, according to the limitations in Table 1.

8.1.6 For either method, report the measured porosity of the packed sample, to the nearest 0.001.

## 9. Precision and Bias

### 9.1 Precision

#### 9.1.1 Method 1 (MIC Sub-sieve AutoSizer):

9.1.1.1 *Repeatability*—The repeatability standard deviation, 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.

9.1.1.2 *Reproducibility*—The reproducibility of Method 1 is being determined and will be available on or before December 31, 2017.

#### 9.1.2 Method 2 (Fisher Sub-Sieve Sizer):

9.1.2.1 The results of an interlaboratory study to determine the precision of this test method are available in ASTM Research Report No. B09–1010<sup>7</sup>, a report on a study done in five laboratories on tungsten carbide powders in both the as-supplied and laboratory-milled conditions. Although this is not in conformance with the requirements of Practice E691 (six laboratories are required), the user of this test method may infer its precision from this interlaboratory study. The pertinent conclusions are presented in 9.1.2.2 and 9.1.2.3.

9.1.2.2 *Repeatability*—The within-laboratory repeatability limit, *r*, as defined by Terminology E456, was estimated to be 2 to 6 % of the measured Fisher Number. Duplicate results from the same laboratory should not be considered suspect unless they differ by more than *r*.

<sup>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1010.

**TABLE 1 Reporting Limitations**

Range (Fisher Number)	Porosity	Range Control	Chart Division (Fisher Number)	Read and Report to (Fisher Number)
0.5 to 1.0	0.55 to 0.80	read direct	0.1	0.02
1.0 to 4.0	0.45 to 0.80	read direct	0.1	0.02
4.0 to 8.0	0.4 to 0.80	read direct	0.2	0.05
8.0 to 15.0	0.40 to 0.65	read direct	0.5	0.2
15.0 to 20.0	0.40 to 0.75	read double	1.0	0.5
20.0 to 50.0	0.40 to 0.60	read double	1.0	0.5

9.1.2.3 *Reproducibility*—The between-laboratory reproducibility limit,  $R$ , as defined by Terminology E456, was found to be estimated by the following equation:

$$R = 0.173F - 0.042 \quad (1)$$

where:

$R$  = the reproducibility limit and  
 $F$  = the measured Fisher Number.

Results from two different laboratories should not be considered suspect unless they differ by more than  $R$ .

9.2 *Bias*—The average particle size (Fisher Number) is a calculated estimate of average particle diameter in a powder.

No absolute method of determining powder particle size exists, nor are there any universally recognized standard or reference powders for this measurement; therefore, it is not possible to discuss the bias of results by these test methods.

## 10. Keywords

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

## SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B330 – 12) that may impact the use of this standard.

**Rationale for Changes** - The Fisher Sub-Sieve Sizer (FSSS), previously the only instrument capable of performing this analysis, is no longer commercially available, nor supported with parts and service. A new instrument, the Sub-Sieve AutoSizer, (now in 2015) manufactured by the Micromeritics Instrument Corporation and known as the MIC SAS, is available to estimate average particle size using air permeability. This revision was therefore instituted to include the new instrument. The 2012 changes thus included:

- (1) The title was changed to indicate the method of analysis and the result of the measurement.
- (2) The title was also changed to indicate more than one method available, since the Fisher Sub-Sieve Sizer is still used in many laboratories.
- (3) A statement on units was added as Section 1.2, noting exceptions as recommended in the B09 Policy Guide.
- (4) The MIC (HEL) SAS was defined in Section 3.2.1 and added to the Apparatus Section 5.
- (5) In section 3.2.5, the words “commercially available” were deleted.
- (6) In Section 3.2.7, responsibility for the master calibrator tube was changed to ASTM Subcommittee B09.03.
- (7) The term average particle size was defined for the purposes of this standard only in Section 3.2.10.

(8) NOTE 1 in the former Section 5.1, regarding availability of replacement parts for the FSSS, was deleted.

(9) The sample weighing precision was changed to 0.1 g from 0.01 g (7.1.3.2 and 7.2.3.3), and the balance requirement changed accordingly in Section 5.4.

(10) Two alternative test methods were described in Sections 6, 7, and 8: Method 1 for the MIC (HEL) SAS, and Method 2 for the FSSS

(11) The calculation sections (the former 7.3.8.1, 7.3.8.2, and 7.3.8.3) were deleted.

(12) The former Section 7.3.8.4 was changed to NOTE 8, with the appropriate changes in wording.

(13) A precision statement for Method 1 (MIC (HEL) SAS) was added as Section 9.1.1.

(14) Several minor editorial changes were made, based on review of the former B330-07 and comments received on ballots of this version.

The 2015 changes included:

- (1) All references to “HEL” changed to “MIC.”
- (2) Revised Footnote 4 to indicate Micromeritics as the sole source of supply.

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