Designation: C721 – 15

Standard Test Methods for Estimating Average Particle Size of Alumina and Silica Powders by Air Permeability[1]

This standard is issued under the fixed designation C721; 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 cover the estimation of the average particle size in micrometres of alumina and silica powders using an air permeability method. The test methods are intended to apply to the testing of alumina and silica powders in the particle size range from 0.2 to 75 μm.

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 long-standing 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

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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

3.1.3 *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. The average particle size obtained by this procedure is a calculated average based on air permeability. It will have a value that is numerically equal to six times the total volume of the sample under test divided by the total envelope-specific surface area of all the particles contained in the sample, or:

$$d_{avg} = 6/\rho s \quad (1)$$

d_{avg} = the estimated average particle size obtained by this procedure, μm,
ρ = absolute density of the particles, g/cm^3, and
s = total envelope-specific surface area of the sample, m^2/g.

NOTE 1—The value of d_{avg} will probably not be numerically equal to the average particle size as obtained by particle size distribution analysis methods since it is independent of particle shape or size distribution. The test methods actually measure sample surface area by air permeability and converts that to an average particle diameter.

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

3.1.5 *envelope-specific surface area, n*—specific surface area of a powder as determined by gas permeametry.

3.1.6 *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.

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

[1] These test methods are under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.04 on Raw Materials.

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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.

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

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

3.1.9 *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.

3.1.10 *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 The estimation of average particle size has two chief functions: first, as a guide to the degree of fineness or coarseness of a powder as this, in turn, is related to the flow and packing properties; and, second, as a control test on the uniformity of a product.

4.2 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.

4.3 These test methods are generally applicable to alumina and silica powders, for particles having diameters between 0.2 and 75 μm (MIC SAS) or between 0.5 and 50 μm (FSSS). They may be used for other similar ceramic powders, with caution as to their applicability. 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 may be used if agreed upon between the parties concerned.

4.4 When an "average" particle size of powders is determined using 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)*[3]——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.

NOTE 2—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 ceramic powders. 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)*[4]——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.

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*[5]—a tube to be used as a standard for average particle size measurement. It allows

[3] 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.

[4] 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.

[5] 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.

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 ± 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 0.01 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 2.

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 four hours 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 to 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 a 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 6.2.3.4.

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, alumina, 3.95 g; silica, 2.65 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 0.01 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 3.1.3, 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 μm:

7.2.1 *Temperature of Test*—Make Fisher Number determinations within ±2°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 ±2°C.

7.2.2 *Size of Test Sample*—The mass of sample used for tests shall be equal in grams (within ±0.01 g) to the true (pore-free) density of the powder (for example, alumina, 3.95 g; silica, 2.65 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 0.01 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, 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 when the upper edge of the cross bar coincides with the bottom of the meniscus in the manometer. 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. If the powder was de-agglomerated or milled prior to analysis, 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, 2019.

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

9.1.2.1 An analysis of variance study was performed by the responsible subcommittee among six laboratories, using an alumina powder of average particle size 2.2 µm and two silica powders of average particle size 2.0 and 4.0 µm. This study, not strictly in accordance with Practice E691, returned the following results:

TABLE 1 Reporting Limitations

Range (Fisher Number)	Range Control	Chart Division (Fisher Number)	Read and Report to (Fisher Number)
0.2 to 4.0	read direct	0.1	0.02[A]
4.0 to 8.0	read direct	0.2	0.1
8.0 to 15.0	read direct	0.5	0.1[B]
15.0 to 20.0	read double	1.0	0.2
20.0 to 50.0	read double	5.0	1.0

[A] For porosities less than 0.55, the reading interval for 0.5 to 4-µm range is 0.05 µm.

[B] For porosities greater than 0.6, the reading interval for 11 to 15-µm range is 0.2 µm.

9.1.2.2 *Repeatability*—The within-laboratory repeatability limit, *r*, as defined by Terminology E456, was estimated to be 0.06 µm. Duplicate results from the same laboratory should not be considered suspect unless they differ by more than *r*.

9.1.2.3 *Reproducibility*—The between-laboratory reproducibility limit, *R*, as defined by Terminology E456, was estimated to be 0.39 µm. 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; alumina; average particle size; envelope-specific surface area; Fischer Number; particle size; permeability; porosity; powder; quartz; silica; specific surface

SUMMARY OF CHANGES

Committee C21 has identified the location of selected changes to this standard since the last issue (C721 – 14) that may impact the use of this standard. (Approved Dec. 1, 2015.)

(1) Deleted all references to "HEL" and changed them to "MIC."

(2) Changed Footnote 3 to indicate Micromeritics as the sole source.

Committee C21 has identified the location of selected changes to this standard since the last issue (C721 – 81 (1997)$^{\varepsilon 1}$) that may impact the use of this standard. (Approved July 1, 2014.)

Rationale for Change—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, manufactured by the Hazard Evaluation Laboratory and known as the HEL SAS, is now available to estimate average particle size using air permeability. This revision was therefore instituted to include the new instrument. The current changes thus included:

(1) The title was changed to indicate that the average particle size is only estimated by this test method.

(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) The statement on units in subsection 1.2 was changed to indicate SI units as standard, with noted exceptions.

(4) Definitions of significant terms were added to subsection 3.1.

(5) The Significance and Use section (now Section 4) was expanded to include other areas of applicability and cautions and assumptions made in estimating average particle size by these methods.

(6) In subsection 6.2.3.8, different recommendations for the frequency of standardization were indicated, compared to the former Section 6.3.7.

(7) Use of the NIST Cement No. 114 as a reference material was eliminated.

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

(9) Much of the wording now follows Test Methods B330, also revised recently to include the new instrument.

(10) Preliminary precision data for the HEL SAS was added and the precision statement for the FSSS was rephrased.