Designation: C1730 − 17

Standard Test Method for Particle Size Distribution of Advanced Ceramics by X-Ray Monitoring of Gravity Sedimentation[1]

This standard is issued under the fixed designation C1730; 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 This test method covers the determination of particle size distribution of advanced ceramic powders. Experience has shown that this test method is satisfactory for the analysis of silicon carbide, silicon nitride, and zirconium oxide in the size range of 0.1 up to 50 µm.

1.1.1 However, the relationship between size and sedimentation velocity used in this test method assumes that particles sediment within the laminar flow regime. It is generally accepted that particles sedimenting with a Reynolds number of 0.3 or less will do so under conditions of laminar flow with negligible error. Particle size distribution analysis for particles settling with a larger Reynolds number may be incorrect due to turbulent flow. Some materials covered by this test method may settle in water with a Reynolds number greater than 0.3 if large particles are present. The user of this test method should calculate the Reynolds number of the largest particle expected to be present in order to judge the quality of obtained results. Reynolds number (Re) can be calculated using the following equation:

$$\mathrm{Re} = \frac{D^3(\rho - \rho_0)\rho_0 g}{18\eta^2} \tag{1}$$

where:

D = the diameter of the largest particle expected to be present, in cm,
ρ = the particle density, in g/cm^3,
ρ_0 = the suspending liquid density, in g/cm^3,
g = the acceleration due to gravity, 981 cm/sec^2, and
η = the suspending liquid viscosity, in poise.

1.1.2 A table of the largest particles that can be analyzed with a suggested maximum Reynolds number of 0.3 or less in water at 35 °C is given for a number of materials in Table 1. A column of the Reynolds number calculated for a 50-µm particle sedimenting in the same liquid system is also given for each material. Larger particles can be analyzed in dispersing media with viscosities greater than that for water. Aqueous solutions of glycerine or sucrose have such higher viscosities.

1.2 The procedure described in this test method may be applied successfully to other ceramic powders in this general size range, provided that appropriate dispersion procedures are developed. It is the responsibility of the user to determine the applicability of this test method to other materials. Note however that some ceramics, such as boron carbide and boron nitride, may not absorb X-rays sufficiently to be characterized by this analysis method.

1.3 The values stated in cgs units are to be regarded as the standard, which is the long-standing industry practice. The values given in parentheses are for information only.

1.4 *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.* Specific hazard information is given in Section 8.

1.5 *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]

C1145 Terminology of Advanced Ceramics
E1617 Practice for Reporting Particle Size Characterization Data

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology C1145.

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[1] This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.03 on Physical Properties and Non-Destructive Evaluation.

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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.

TABLE 1 Maximum Diameter of Ceramic Powders That Can Be Analyzed with Reynolds Number of 0.3 or Less in Water at 35 °C

Particle Composition	Particle Density	Maximum Particle Diameter, µm	Reynolds Number for 50 µm[A]
Aluminum Nitride	3.26	50.36	0.29
Aluminum Oxide	3.965	46.01	0.39
Cerium Dioxide	7.132	36.13	0.80
Silicon Carbide	3.217	50.68	0.29
Silicon Nitride	3.44	49.09	0.32
Yttrium Oxide	5.01	41.61	0.52
Zirconium Oxide	5.89	38.95	0.63

[A] A Reynolds number calculated for 50-µm particles sedimenting in water at 35 °C, with a density of 0.9941 g/cm^3 and viscosity of 0.7225 cp. Entries with Reynolds numbers that exceed the recommended upper limit of 0.30 are included to indicate that samples of these materials containing 50-µm particles should not be analyzed using water as a dispersing liquid without addition of a viscosity modifier such as glycerol or sucrose.

4. Summary of Test Method

4.1 A carefully dispersed homogeneous suspension of the powder is permitted to settle in a cell scanned by a collimated X-ray beam of constant intensity. The net X-ray signal is inversely proportional to the sample concentration in the dispersing medium, and the particle diameter is related to the position of the X-ray beam relative to the top of the cell. Cumulative mass percent versus equivalent spherical diameter are recorded to yield a particle size distribution curve.

5. Significance and Use

5.1 This test method is useful to both suppliers and users of powders, as outlined in 1.1 and 1.2, in determining particle size distribution for product specifications, manufacturing control, development, and research.

5.2 Users should be aware that sample concentrations used in this test method may not be what are considered ideal by some authorities, and that the range of this test method extends into the region where Brownian movement could be a factor in conventional sedimentation. Within the range of this test method, neither the sample concentration nor Brownian movement is believed to be significant. Standard reference materials traceable to national standards, of chemical composition specifically covered by this test method, are available from NIST,[3] and perhaps other suppliers.

5.3 Reported particle size measurement is a function of the actual particle dimension and shape factor as well as the particular physical or chemical properties 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.

5.4 Suppliers and users of data obtained using this test method need to agree upon the suitability of these data to provide specification for and allow performance prediction of the materials analyzed.

6. Apparatus

6.1 *X-Ray Gravitational Sedimentation Particle Size Analyzer*—The analyzer shall utilize extinction of collimated X-rays to determine particle mass concentration as a homogeneous dispersion of sample sediments under the influence of gravity.[4]

6.2 *Ultrasonic Probe,* consisting of a 200 to 300 W power unit, ultrasonic transducer, and 13-mm (½-in.) diameter probe.

6.3 *Balance,* top-loading, accurate to 100 ± 0.1 g.

6.4 *Stirrer,* magnetic, with 19-mm (¾-in.) stirrer bar.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Typical Dispersing Media*—Dissolve 5.0 g of sodium hexametaphosphate [$(NaPO_3)_6$] in 1000 mL of distilled or deionized water. Alternately, dissolve 5.0 g Darvan C[6] (ammonium salt of polymethacrylic acid) in 95 mL of distilled or deionized water. This latter solution is typically used after dilution of 0.03 g per 20 mL of distilled or deionized water.

7.3 *pH Adjusters*—Fresh ammonium hydroxide (NH_4OH) at 5 to 10 weight % strength is a common reagent used to raise pH, while fresh nitric acid (HNO_3) at 5 to 10 weight % strength is a common reagent to lower pH.

7.4 *Cleaning Solution*—Prepare a 0.1 % solution by volume of Triton X-100 using distilled or deionized water,[7] or other suitable laboratory cleaning solution.

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[3] National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 2300, Gaithersburg, MD 20899-2300, http://www.nist.gov.

[4] The sole instrument of this type known to the committee at this time is the SediGraph X-ray gravity sedimentation particle size analyzer, available from Micromeritics Instrument Corporation, 4356 Communications Drive, Norcross, GA 30093. 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] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[6] Darvan C is a trademarked product of R.T. Vanderbilt Company, Inc., Norwalk, CT 06856-5150.

[7] Triton X-100 is a trademarked product of Rohm & Haas, Philadelphia, PA and is available from a number of laboratory supply companies.

8. Hazards

8.1 Precautions applying to the use of low intensity X-ray units shall be observed. Do not attempt to disable mechanical and electrical X-ray safety interlocks. Do not attempt to operate the instrument with panels removed.

9. Procedure

9.1 *Analyzer Preparation:*

9.1.1 See the manufacturer's manual for general operating instructions.

9.1.2 To warm up the analyzer, apply power for a minimum of 30 min prior to testing.

9.1.3 Set up the instrument to report either cumulative mass finer than, or passing, or cumulative mass coarser than, or retained, as desired. Ensure proper operating conditions by periodically performing baseline scan (analysis of X-ray profile with no particles present) and beam split test (determination of top of sedimentation cell) if necessary, and by analyzing reference samples with known, accepted distributions. No specific calibration tests are needed as results are based upon first principles. However, there are some electronic calibrations, such as for temperature of the sample dispersion, performed at time of instrument manufacture or repair, by trained personnel, using proper equipment with calibrations traceable to national standards.

9.2 *Sample Preparation and Dispersion:*

9.2.1 Add appropriate sample mass to a beaker containing the proper amount of appropriate dispersing medium suggested in analyzer instruction manual. Generally a 100-mL tall-form glass beaker is used, with 80 to 100 mL of dispersing medium. Typically the concentration of sample required is that which reduces the intensity of X-rays by 30 to 40 % prior to the start of sedimentation.

NOTE 1—Suggested approximate starting masses for silicon carbide, silicon nitride, and zirconium dioxide in 100 mL of dispersing medium are listed in Table 2.

NOTE 2—Dispersion of some advanced ceramic materials is aided by the use of wetting agents and surfactants. Many times sodium polyacrylates, sodium polysulfonates, and sodium polyphosphates are used in 0.05 % to 0.3 % concentrations (w/w). For others, such as zirconium oxide and silicon nitride, dispersion in water after pH adjustment is sufficient.

9.2.2 Place the beaker containing the sample on a magnetic stirrer. Add a stirrer bar to the beaker. Adjust the stirrer to provide a small vortex in the sample surface.

9.2.3 Adjust the pH of the sample, if necessary, to ensure adequate dispersion aided by the chemical environment. For some materials, this occurs at pH greater than 9.0.

TABLE 2 Suggested Approximate Starting Masses for Silicon Carbide, Silicon Nitride, and Zirconium Oxide

Material	Mass,^{A} g, per 100 mL of Dispersing Medium
Silicon Carbide	5.0
Silicon Nitride	5.0
Zirconium Dioxide	2.0

[A] The amount of sample required will vary. Increase or decrease the sample mass as needed to provide the level of X-ray attenuation recommended in analyzer instruction manual.

9.2.4 If it is desired to analyze a de-agglomerated sample, while continuing to stir the sample, insert, for example, an ultrasonic transducer into the beaker of the dispersing liquid containing the sample. Ensure that the transducer is approximately 10 mm above the bottom of the sample beaker. Apply ultrasonic energy at 40 W or higher for 1.0 min. Allow the sample to sit for 1.0 min. Repeat the procedure two additional times so that the total ultrasonic energy application time is 3 min. This high level of mechanical energy application is necessary to accomplish de-agglomeration of the particles. The extent of de-agglomeration is expected to be different for powders produced by different processes. An alternate approach is to ultrasonicate for 3 min continuously before pH and temperature check. These specific dispersion instructions are provided to illustrate the importance of proper, repeatable dispersions, in order to obtain repeatable analysis results. The dispersion method given has been shown to be appropriate for the materials specified in the scope of this test method. The total ultrasonic power needs to be limited for friable samples, subject to primary particle fracture, and when the determination of size of agglomerated sample is desired. Typically the amplitude, and thus the power, of the vibrating ultrasonic transducer can be adjusted by the operator. Also, ultrasonic baths and laboratory blenders can be employed when use of a more powerful ultrasonic transducer would result in primary particle fracture. The total sheer force applied to the particles during dispersion should be appropriate for the final application of the material under study.

9.2.5 Readjust the pH, if necessary, after ultrasonic treatment. Keep stirring the sample continuously prior to commencement of the analysis, and test it as soon as practical to prevent re-agglomeration.

9.3 *Temperature Adjustment:*

9.3.1 If the temperature of the solution is above that of the cell chamber after ultrasonic dispersion, cool the solution to within 1 °C of the cell chamber prior to the introduction into the cell chamber by stirring outside the cell chamber. This cooling must be accomplished as soon as possible.

9.3.2 If the temperature is below the cell chamber temperature, load the sample into the cell or sample chamber of the analyzer and allow sample to circulate until sample temperature is within 1 °C of the chamber in which the sample analysis cell is housed. The temperature of the analysis compartment and sample circulation system are generally controlled and reported, usually with indication of whether or not the sample temperature is within acceptable limits.

9.4 Load the prepared sample into analyzer according to analyzer instruction manual. Adjust the built-in magnetic stirrer to produce a slight vortex, typically 0.5 cm deep, in the sample dispersion.

9.5 Follow analyzer instruction manual to begin analysis of sample. Be sure to include the use of any necessary sedimentation parameters, such as sample density, liquid density and viscosity, and analysis size range.

NOTE 3—Be aware that bubbles may need to be removed from the analysis cell prior to analysis. Some instrumentation performs an automatic scan for bubbles and, if detected, follows with a bubble elimination routine.

9.6 Rinse the sedimentation cell and sample chamber thoroughly, up to three times, with fresh dispersing medium, according to analyzer instruction manual. If it is necessary to clear the cell further at this time, rinse three times with the dilute cleaning solution, followed by an additional cycle of rinses with fresh dispersing medium.

9.7 It is advised that a repeat analysis be performed on a separately weighed portion of the sample, thus providing two distributions on the same powder. Compare the results of the two analyses. The values for cumulative mass distribution at specific diameters within the analysis range should be within ±2 mass percent for samples with measured particle size distribution spanning one or more orders of magnitude. (For samples with narrower distributions, the difference between the two tests should be less than 3 mass percent.) If so, results of either analysis, or the average of the two, can be reported. If the results differ by more than 2 mass percent, steps should be taken to determine the reason for excessive sample analysis result variation. Proper instrument operation should be verified, as described in 9.1. If repeated analyses continue to exceed the expected sample reproducibility, and proper instrument operation has been verified, incomplete sample dispersion stabilization is usually the cause.

10. Report

10.1 A copy of all the data, either in graphical or tabular form, shall be supplied. Users and suppliers of powder should agree on the reporting requirements. Practice E1617 provides requirements for various levels of result reporting in order to ensure better understanding and reliability of analysis results.

11. Precision and Bias

11.1 *Precision*—An interlaboratory study has been conducted previously by the International Energy Agency, and data from that study are available from the National Institute of Standards and Technology (NIST) as publication SP-879.[8] Data of the previous study have been determined by ASTM Subcommittee C28.03 to be appropriate for this temporary precision statement. Due to the variability in the sample preparation methods used by the various participants, a new interlaboratory study will be conducted using a number of advanced ceramic materials, to be completed within five years of publication of this test method.

11.1.1 A silicon nitride powder with measured median diameter of 1.0 µm was used in the IEA interlaboratory study. The median diameter repeatability standard deviation has been determined to be 0.07 µm and the 95 % repeatability limit is 0.20 µm. The reproducibility standard deviation has been determined to be 0.10 µm and the 95 % reproducibility limit is 0.27 µm for this silicon nitride sample.

11.2 *Bias*—No absolute method of determining powder particle size exists, nor are there any universally recognized standards or reference powders for this measurement. Therefore, it is not possible to discuss the bias results by this test method.

12. Keywords

12.1 advanced ceramics; gravity sedimentation; particle size distribution; silicon carbide; silicon nitride; zirconium oxide

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[8] Available by special request from the NIST Research Library via the NIST website at www.nist.org.