Designation: B761 – 17 (Reapproved 2021)

Standard Test Method for Particle Size Distribution of Metal Powders and Related Compounds by X-Ray Monitoring of Gravity Sedimentation[1]

This standard is issued under the fixed designation B761; 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 distributions of metal powders. Experience has shown that this test method is satisfactory for the analysis of elemental tungsten, tungsten carbide, molybdenum, and tantalum powders, all with an as-supplied estimated average particle size of 6 µm or less, as determined by Test Method B330. Other metal powders (for example, elemental metals, carbides, and nitrides) may be analyzed using this test method with caution as to significance until actual satisfactory experience is developed (see 7.2). The procedure covers the determination of particle size distribution of the powder in the following two conditions:

1.1.1 As the powder is supplied (as-supplied), and

1.1.2 After the powder has been deagglomerated by rod milling as described in Practice B859.

1.2 This test method is applicable to particles of uniform density and composition having a particle size distribution range of 0.1 up to 100 µm.

1.2.1 However, the relationship between size and sedimentation velocity used in this test method assumes that particles sediment within the laminar flow regime. This requires that the particles sediment with a Reynolds number of 0.3 or less. 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 with Reynolds number greater than 0.3 if particles greater than 25 µm 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 flowing equation

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

where

D = the diameter of the largest particle expected to be present,
ρ = the particle density,
ρ_0 = the suspending liquid density,
g = the acceleration due to gravity, and
η = is the suspending liquid viscosity.

A table of the largest particles that can be analyzed with Reynolds number of 0.3 or less in water at 35°C is given for a number of metals in Table 1. A column of the Reynolds number calculated for a 30–µm particle sedimenting in the same liquid system is given for each material also.

1.3 *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, the values in SI units are to be regarded as standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Specific hazard information is given in Section 7.*

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]

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

B821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis

[1] This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is 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.

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

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

Particle Composition	Particle Density	Maximum Particle Diameter	Reynolds Number for 30 μm[A]
Cobalt	8.90	33.19	0.22
Copper	8.92	33.16	0.22
Iron	7.86	34.79	0.19
Molybdenum	10.20	31.55	0.26
Nickel	8.90	33.19	0.22
Tantalum	16.60	26.46	0.44
Tantalum carbide	13.90	28.19	0.36
Titanium carbide	4.93	41.88	0.11
Tungsten	19.35	25.06	0.51
Tungsten carbide	15.63	27.03	0.41
Vanadium	6.11	38.37	.014
Vanadium carbide	5.77	39.26	0.13

[A] Reynolds number calculated for 30 μm particle sedimenting in water at 35°C, with a density of 0.9941 g/cm^3 and viscosity of 0.7225 cp.

B859 Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis
E456 Terminology Relating to Quality and Statistics
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 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.

4. Significance and Use

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

4.2 Users should be aware that sample concentrations used in this test method may not be what is 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 are believed to be significant.

4.3 Reported particle size measurement is a function of both 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. Apparatus

5.1 Gravitational sedimentation particle size analyzer utilizing X-ray extinction to determine particle concentration.[3]

6. Reagents and Materials

6.1 *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.[4] 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.

6.2 *Dispersing Medium*—Dissolve 0.10 g of sodium hexametaphosphate [$(NaPO_3)_6$] in 1000 mL of distilled or deionized water.

6.3 *Cleaning Solution*—Dissolve 0.5 g of laboratory detergent in 1000 mL of distilled or deionized water, or prepare a 0.1 % solution by volume of Triton X-100 using distilled or deionized water.[5]

7. Hazards

7.1 Precautions applying to the use of low intensity X-ray units should be observed.

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[3] The sole instrument of this type known to the committee as 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.

[4] *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.

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