Designation: B761 – 06 (Reapproved 2011)

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

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 Fisher number 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 *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 7.*

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
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

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

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 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.

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.

7.2 Most carbides and nitrides are brittle materials and may be partially deagglomerated or fractured, or both, during the manufacturing process. Different manufacturing processes or changes in the process may affect the apparent particle size distribution as determined by this test method. Thus, caution should be used in evaluating the results, especially for brittle materials.

8. Sample Preparation

8.1 For the as-supplied particle size distribution determinations, this step is not needed.

8.2 For laboratory-milled particle size distribution determinations, use the rod milling technique as outlined in Practice B859.

9. Procedure

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

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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, 1 Micromeritics 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.

9.2 Set up the instrument in the "percent finer than" mode if necessary. Ensure proper operating conditions by periodically performing base line scan and beam split test if necessary.

9.3 Add appropriate sample weight to the amount of dispersing medium suggested in analyzer instruction manual.

NOTE 1—Suggested approximate starting weights for tungsten and tungsten carbide are listed in Table 2.

9.4 *Sample Dispersion*—Follow procedure recommended in Guide B821.

9.5 *Temperature Adjustment:*

9.5.1 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 and pumping outside the cell chamber (see Note 2). This cooling must be accomplished as soon as possible.

NOTE 2—It may be convenient to use a separate magnetic stirrer and stirring rod.

9.5.2 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 cell chamber.

9.6 Load the prepared sample into analyzer according to analyzer instruction manual.

9.7 Follow analyzer instruction manual to begin analysis of sample. Be sure to include the use of any necessary sedimentation parameters where necessary.

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

NOTE 4—It is recommended that the stirrer be turned off simultaneously with activation of the instrument.

9.8 Rinse the sedimentation cell and sample chamber thoroughly 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.

TABLE 2 Suggested Approximate Starting Weights for Tungsten or Tungsten Carbide

Nominal Fisher Number According to Test Method B330 of As-Supplied Powder, µm	Weight^A, g per 25 mL of Dispersing Medium
1	0.21
2	0.23
3	0.26
4	0.28
5	0.30
6	0.33

^{A} The amount of sample required will vary. Increase or decrease the sample weight as needed to provide the level of X-ray attenuation recommended in analyzer instruction manual.

9.9 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.

10. Report

10.1 A copy of all the data, either in graphical or tabular form, shall be supplied.

11. Precision and Bias

11.1 *Precision*—The results of an interlaboratory study to determine the precision of this test method are available in Research Report No. B09–1011,[6] which is a report on a study done in nine laboratories on two tungsten carbide powders in the rod-milled condition. Although this is not in conformance with the requirements of Practice E691 (three materials are required; six or more recommended), the user of this test method may infer its precision from this interlaboratory study. The pertinent conclusions are presented below:

11.1.1 The within-laboratory repeatability limit, r, for the median particle size (r as defined by Terminology E456), was found to be estimated by the following equation:

$$r = 0.133M - 0.009 \tag{2}$$

where

M = the measured median particle size (µm), in the range of 1.4 to 4.2 µm (r = 0.15 to 0.55 µm in this range).

Duplicate median particle size results from the *same* laboratory should not be considered suspect unless they differ by more than r.

11.1.2 The between-laboratory reproducibility limit, R, for the median particle size (R as defined by Terminology E456) was found to be estimated by the following equation:

$$R = 0.482M - 0.489 \tag{3}$$

where

M = the measured median particle size (µm) in the range of 1.4 to 4.2 µm (R = 0.19 to 1.54 µm in this range).

Median particle size results from two *different* laboratories should not be considered suspect unless they differ by more than R.

11.2 *Bias*—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 results by this test method.

12. Keywords

12.1 metal powders; particle size; particle size distribution; powdered metals; refractory metal powders; sedimentation particle size distribution

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1011.

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue, B761 – 02$^{\varepsilon 1}$, that may impact the use of this standard. (Approved April 1, 2006.)

(1) A footnote containing sole source information for apparatus meeting specifications of this test method was inserted. Rationale: The subcommittee is only aware of one source of commercial apparatus.

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