Designation: B 430 – 97 (Reapproved 2001)

Standard Test Method for Particle Size Distribution of Refractory Metal Powders and Related Compounds by Turbidimetry[1]

This standard is issued under the fixed designation B 430; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of particle size distribution of refractory metal powders with a turbidimeter **(1)**.[2] Experience has shown that this test method is satisfactory for the analysis of elemental tungsten, molybdenum, rhenium, tantalum metal powders, and tungsten carbide powders. Other refractory 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. The procedure covers the determination of particle size distribution of the powder in two conditions:

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

1.1.2 After the powder has been de-agglomerated by rod milling (laboratory milled) according to Practice B 859.

1.2 Where dual units are given, inch-pound 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:*

B 330 Test Method for Fisher Number of Metal Powders and Related Compounds[3]

B 821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis[3]

B 859 Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis[3]

E 456 Terminology Relating to Quality and Statistics[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

3. Summary of Test Method

3.1 A uniform dispersion of the powder in a liquid medium is allowed to settle in a glass cell. A beam of light is passed through the cell at a level having a known vertical distance from the liquid level. The intensity of the light beam is determined using a photo cell. This intensity increases with time as sedimentation of the dispersion takes place.

3.2 The times at which all particles of a given size have settled below the level of the transmitted light beam are calculated from Stokes' law for the series of sizes chosen for the particle size analysis.

3.3 The intensity of the light beam at these times is measured as percent of the light transmitted through the cell with the clear liquid medium. The size distribution in the powder can be calculated from these relative intensities using the Lambert-Beer law in the modified form (also see Refs **2, 3, 4**).

$$\Delta W_{1-2} = d_m(\log I_{d1} - \log I_{d2}) \quad (1)$$

where I_{d1} and I_{d2} are the intensities measured at the times when all particles having diameters larger than d_1 and d_2 respectively have settled below the level of the light beam, d_m is the arithmetic mean of particle sizes d_1 and d_2, and $\Delta W_{1\text{-}2}$ refers to the relative weight for the particle size range between d_1 and d_2. Values of ΔW are determined for each of the particle size ranges chosen. The sum of these values is $\Sigma\Delta W$. The weight percent of particles in the size range from d_1 to d_2 can then be calculated as:

$$\text{Weight, \%} = (\Delta W_{1-2}/\Sigma\Delta W) \times 100 \quad (2)$$

4. Significance and Use

4.1 Knowledge of the particle size distribution of refractory metal powders is useful in predicting powder-processing behavior, and ultimate performance of powder metallurgy parts.

[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] The boldface numbers in parenthesis refer to the references listed at the end of this test method.

[3] *Annual Book of ASTM Standards*, Vol 02.05.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

Particle size distribution is closely related to the flowability, compressibility, and die-filling characteristics of a powder, as well as to the final structure and properties of the finished parts. However, the degree of correlation between the results of this test method and the quality of powders in use has not been fully determined quantitatively.

4.2 This test method is suitable for manufacturing control and research and development in the production and use of refractory metal-type powders, as indicated in 1.1.

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 also can affect reported particle size results.

5. Apparatus

5.1 *Turbidimeter* **(5)**—The recommended instrument is one[5] using a cell rectangular in cross section, approximately 50 mm high, 40 mm wide, and 10-mm sedimentation medium thickness, and having optically parallel faces.

5.2 *Millivolt Recorder*, 0 to 10-mV range, 10-in. (254-mm) wide strip chart, 0 to 100 graduations, 120 in./h (50 mm/min) chart speed,[6] or microammeter with 0 to 100 graduations, 15-μA full scale, 4.5-mV full scale.

NOTE 1—While a 120-in./h (50-mm/min) chart speed is recommended, other speeds may be satisfactory.

5.3 *Ultrasonic Cleaning Tank*, with tank dimensions approximately 5 by 5 by 3 in. (127 by 127 by 76 mm) deep and an output of 50 W, or approximately 3½ by 3½ by 2⅝ in. (89 by 89 by 67 mm) deep and an output of 25 W.[7]

5.4 *Glass Vial*, nominal 2-dram, flat-bottom, with a tight-fitting cap. The vial shall be approximately 2 in. (51 mm) in height with a ⅝-in. (16-mm) outside diameter and approximately a 1/32-in. (0.8-mm) wall.[8]

6. Reagents

6.1 *Sedimentation Medium*:

6.1.1 *Base Medium*, distilled or deionized water (see Note 4).

6.1.2 Use either one of the following as recommended in Guide B 821:

6.1.2.1 *Daxad* (No. 11)[9]—Dissolve 25 mg in 1 L of base medium.

6.1.2.2 *Sodium Hexametaphosphate*—Dissolve 0.1 g in 1 L of base medium.

NOTE 2—Use water that is pure. Do not store the sedimentation medium longer than a week, and do not use rubber tubing in any storage container. Clean thoroughly all sedimentation medium containers every week.

7. Preparation of Apparatus

7.1 Warm up equipment by turning on the light source and recorder for a minimum of 1 h prior to use.

7.2 Fill the cell with sedimentation medium to a height sufficient to cover the light beam path by at least 10 mm and place the cell in the turbidimeter (Note 3). If a microammeter is used to measure light intensity, adjust the light transmission to 100 % using the diaphragm. If a millivolt recorder is used, adjust the potentiometer so that the photovoltaic cell output is 10 mV or 100 %. In this case, the diaphragm is not adjusted and is completely open.

NOTE 3—For convenience in filling the cell to the proper height, inscribe a line on each face of the cell at the desired liquid-level height. The height of fall is usually 25 mm. To determine the location of the line, the center of the light beam path must be established and 25 mm added to this value.

7.3 After the instrument is adjusted to 100 % light transmission through the sedimentation cell and medium, move the cell carriage until light is passing through a reference glass held in another slot of the cell carriage. Read and record the percent of reference light transmission. Having been selected to have approximately 70 to 95 % of the transmission of the sedimentation cell and medium, the reference glass will indicate 100 % light transmission through the sedimentation cell when the recorder reads this value through the reference cell.

8. Calculation of Times at Which Light Intensity is Measured

8.1 The times at which the light transmission values should be read are calculated from Stokes' law. A uniform 1-μm interval should be used in making measurements through the 10-μm size and, depending upon the particular powder, either 1-μm or 5-μm intervals thereafter. The form of Stokes' law used is as follows:

$$t = (18 \times 10^8 Nh)/d^2(\rho_x - \rho_m)g \quad (3)$$

where:

t = time, s,
N = viscosity of settling medium at ambient temperature, P (Note 4),
h = height of fall, cm (distance from liquid level height to midpoint of light beam),
d = diameter of particle, μm (d_1, d_2, et al),
ρ_x = theoretical density of the powder being tested (for tungsten, use 19.3 g/cm ^{3}),

[5] The recommended instrument is a Cenco Photelometer (not made anymore) of original or modified designs or any proven equivalent instrument. A schematic diagram of the Photelometer is shown in the papers referenced at the end of this test method. Copies of detailed drawings of an acceptable instrument are available from ASTM Headquarters. Order ADJA0430. A fabricated instrument can be secured from WAB Instruments Co., 5171 Hickory Dr., Cleveland, OH 44124.

[6] The 69800-Q1, Model S, Type G, Speedomax W, or XL630 Series recorder as made by the Leeds and Northrup Co., have been found satisfactory.

[7] Ultrasonic tank Model Nos. 2 or 12 as made by Bransonic Instrument Co., Stamford, CT, have been found satisfactory.

[8] Two-dram Titeseal vials, as made by Chemical Rubber Co., Cleveland, OH, have been found satisfactory.

[9] Daxad No. 11 powder as made by the W. R. Grace and Co., Polymers and Chemicals Div., 62 Whittemore Ave., Cambridge, MA 02140, has been found satisfactory.