



## Standard Test Method for Particle Size Distribution of Refractory Metal Powders and Related Compounds by Turbidimetry<sup>1</sup>

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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

*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).<sup>2</sup> 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 Average Particle Size of Powders of Refractory Metals and Their Compounds by the Fisher Sub-Sieve Sizer<sup>3</sup>

B 821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis<sup>3</sup>

B 859 Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis<sup>3</sup>

E 456 Terminology Relating to Quality and Statistics<sup>4</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>4</sup>

### 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  $\Delta W_{1-2}$  refers to the relative weight for the particle size range between  $d_1$  and  $d_2$ . Values of  $\Delta W$  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. 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.

<sup>1</sup> 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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<sup>2</sup> The boldface numbers in parenthesis refer to the references listed at the end of this test method.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 02.05.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

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<sup>5</sup> 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,<sup>6</sup> 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.<sup>7</sup>

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.<sup>8</sup>

**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)*<sup>9</sup>—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.

<sup>5</sup> 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.

<sup>6</sup> 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.

<sup>7</sup> Ultrasonic tank Model Nos. 2 or 12 as made by Branson Instrument Co., Stamford, CT, have been found satisfactory.

<sup>8</sup> Two-dram Titesal vials, as made by Chemical Rubber Co., Cleveland, OH, have been found satisfactory.

<sup>9</sup> 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.

**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:

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

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*<sub>1</sub>, *d*<sub>2</sub>, et al),
- ρ<sub>x</sub>* = theoretical density of the powder being tested (for tungsten, use 19.3 g/cm<sup>3</sup>),
- ρ<sub>m</sub>* = density of settling medium at ambient temperature (Note 4), and
- g* = gravitational constant (980 cm/s<sup>2</sup>).

NOTE 4—The viscosity and density values at different temperatures that are used for the sedimentation medium in this procedure are the same as for pure water. Some viscosity (from the *Handbook of Chemistry and Physics*, 65th Edition, CRC Press, 1984) and density (from *Metrological Handbook 145*, NIST, 1990) values are given as follows:

Temperature, °C	°F	Viscosity,	Density,
		cP	g/cm <sup>3</sup>
18	64.4	1.0530	0.9986
19	66.2	1.0270	0.9984
20	68.0	1.0020	0.9982
21	69.8	0.9779	0.9980

22	71.6	0.9548	0.9978
23	73.4	0.9325	0.9975
24	75.2	0.9111	0.9973
25	77.0	0.8904	0.9970
26	78.8	0.8705	0.9968
27	80.6	0.8513	0.9965
28	82.4	0.8327	0.9962
29	84.2	0.8148	0.9959
30	86.0	0.7975	0.9956

**9. Conditioning (or De-agglomeration) of the Powder Prior to Analysis**

9.1 For *as-supplied* particle size distribution determinations, this step is not needed.

9.2 For *laboratory-milled* particle size distribution determinations, follow the procedure specified in Practice B 859.

NOTE 5—Since milled powder has a greater tendency than as-supplied powder to pick up moisture and oxidize, the analysis procedure should be initiated immediately after milling is completed. This is particularly important if the powder is to be dispersed using the 5-min hand-shake procedure (see Section 8) where a difference can be seen between determinations made in succession on powders having significant amounts of 1-µm size powder. This difference, related to the size of the powder, is greater for fine powders. For all practical purposes, however, two runs can be made in succession on each milled powder. If more than two runs on the same milled powder are desired using the 5-min shake procedure, provisions may be taken to lessen (elimination is not possible) the effect of humidity on the milled powder such as immediate splitting of the sample and storage under dry nitrogen or in a desiccator. If the 5-min ultrasonic procedure is used to disperse the powder for analysis, the milled powder may be stored for several days without any effect being seen in the distribution results.

**10. Dispersion**

10.1 The powder, either as supplied, or laboratory milled in accordance with 9.2, may be dispersed in the sedimentation medium either by a 5-min ultrasonic treatment procedure or by a 5-min continuous hand-shake procedure. The 5-min ultrasonic treatment procedure is the preferred and recommended procedure.

NOTE 6—The weight of the sample used should give a preferred initial light transmission of between 20 and 30 %. Transmissions between 15 and 40 % are acceptable. If it is desired to change the initial light transmission, reweigh another sample, increasing or decreasing the weight accordingly.

NOTE 7—Table 1 gives likely sample weight ranges for lab-milled tungsten powders having known Fisher sub-sieve sizer average particle diameters in the as-supplied condition. (See Test Method B 330.) These likely sample weight ranges apply for powders that have been lab-milled before testing and either dispersed using the 5-min ultrasonic treatment or the 5-min hand-shake procedure. The table also lists preferred micrometre sizes to be read. For the determination of particle distribution of tungsten in the as-supplied condition, or other powders, proper weights should be determined by trial and error.

10.2 The 5-min ultrasonic treatment dispersion procedure is as follows:

10.2.1 Fill the vial with 2 mL of sedimentation medium or to a height of approximately ¼in. (7.0 mm). Add weighed amount of powder and cap the vial. Place into the ultrasonic tank, handholding the vial for 5 min.

NOTE 8—Depth of the liquid in the tank should be 1½ to 2 in. (approximately 40 to 50 mm) from the bottom. Liquid in the tank is distilled or deionized water, room temperature, with a small amount of detergent. A 1-min warm-up of the ultrasonic tank is recommended prior to vial immersion.

NOTE 9—If any of the powder sample is on the walls of the vial, the liquid may be swirled before and during the ultrasonic treatment to rinse the powder down into the bottom. The vial need not be held in a stationary position nor perpendicular to the bottom. Depth of immersion and location of the vial are generally at the center portion of the tank, but may vary. Where cavitation within the vial is noticeable, as evidenced by rapid agitation of the powder dispersion, the bottom of the vial could even be at the surface of the tank liquid. Agitation within the vial should be noticeable. Where agitation is not evident within the vial, the vial should be moved until agitation is evident. The vial generally is immersed to a depth where powder dispersion is at or below tank liquid level with the vial bottom not closer than ½ in. (about 10 mm) to the bottom of the tank. Immersion is generally not within 1 in. (about 25 mm) from any tank wall. During ultrasonic treatment, a slight tingling feeling at the fingertips, where they touch the vial, might be present. Also, while vial and contents are slightly warmed during treatment, no temperature correction need be made because of the subsequent dilution in the sedimentation cell.

10.2.2 Wipe dry or rinse the outside of the vial immediately after ultrasonic treatment to prevent ultrasonic tank liquid contamination in the sedimentation cell.

10.2.3 Quantitatively transfer the powder dispersion into an empty sedimentation cell. Thoroughly rinse the vial, making sure that all the powder is in the cell.

NOTE 10—A 250 or 500-mL plastic wash bottle that has had the nozzle straightened to an upright position has been found to be convenient to flush the vial of remaining traces of powder as it is inverted over and into the sedimentation cell at a slight angle. Care must be taken not to flush the vial so strongly that liquid and powder splashes out over the sedimentation cell. (See Note 2 regarding cleansing of this equipment.)

NOTE 11—Usually no difficulty is encountered in the transfer of fine powders into the sedimentation cell. However, where coarse powders are ultrasonically dispersed, there is a tendency for some of this powder to remain in the vial and the transfer is a little more difficult. Experience will solve this problem.

10.2.4 Fill the sedimentation cell to the proper height (see Note 3). Adjustment of the final liquid level may be done by using an eye-dropper filled with sedimentation medium.

10.2.5 Close the cell and redisperse the powder in the sedimentation medium by holding it at the top and the bottom and turning it upside down and shaking it for approximately 5

**TABLE 1 Lab-Milled Tungsten Metal Powders**

"As-Supplied" Average Particle Diameter by Fisher Sub-Sieve Sizer, µm	Likely Sample Weight Range, mg		Micrometre Sizes Read
	5-min Ultrasonic Dispersion	5-min Hand-Shake Dispersion	
0 to 1.0	0 to 10	5 to 15	Each µm interval from 1 to 5
1.0 to 1.8	5 to 15	10 to 20	Each µm interval from 1 to 5
1.8 to 3.0	15 to 30	20 to 35	Each µm interval from 1 to 10
3.0 to 5.0	30 to 60	35 to 75	Each µm interval from 1 to 10 plus at 15, 20, and 25
5.0 to 7.0	40 to 100	50 to 100	Each µm interval from 1 to 15 plus at 20 and 25
7.0 to 12.0	75 to 150	75 to 150	Each µm interval from 1 to 15 plus at 20 and 25

to 10 s to remove any powder that has settled to the bottom. Then give the cell 1½ to 2-min second shake (not quite as vigorous as described in 10.3.2) ending with a gentle end-over-end *complete* 360° facewise rotation that allows the air bubble contained in the cell to “wipe” both faces for approximately 10 s to rehomogenize the contents. *Continue this facewise rotation until the cell is placed in the instrument.* During this time, visually check the contents of the cell for uniformity of dispersion and recheck the liquid level.

10.2.6 Proceed *immediately* to step 11.1.

10.3 The 5-min hand-shake dispersion procedure is as follows:

10.3.1 Fill the sedimentation cell with sedimentation medium to approximately 1 to 2 mm below the graduated line that signifies a 25-mm height of fall.

10.3.2 Transfer the weighed sample into the sedimentation cell. Close the cell with a cover and, holding it at the top and bottom between the forefingers and the thumb, shake vigorously for 4½ to 4¾min. Shake the cell by moving it in an arc of 12 to 15 in. (305 to 380 mm) in length, back and forth approximately one cycle per second. The sedimentation medium movement is distinctly heard as the cell is shaken. After the vigorous shake, remove the cover from the cell and adjust the liquid level to the graduated line using an eye dropper filled with sedimentation medium.

10.3.3 Continue by performing steps listed in 10.2.5 and 10.2.6 except to eliminate 1½ to 2-min second shake.

## 11. Procedure

11.1 During the last 2 to 10 s of the gentle shake, start the chart paper motor, recheck the reference light transmission value (see 7.3), adjusting the instrument accordingly, and move the cell carriage to block completely the light transmission. The reading of the millivolt recorder or microammeter changes from 100 to 0 % transmission. As soon as the transmission is 0 %, cautiously drop (Note 12) the cell into the carriage, and then immediately position in the light path. Exercise care to position the cell vertically (top to bottom) in the carriage before moving it into the light path and that the cell carriage is recentered before starting the run.

11.2 As the powder settles, record the light transmission values either manually at the appropriate times determined in 8.1, or continuously through the use of the potentiometer and millivolt recorder. If a recorder is used, read the light transmission for the appropriate times from the graph paper after sedimentation is complete.

NOTE 12—If the cell is dropped too hard, it might crack. To reduce this possibility, place the thumb and forefinger of the other hand around the

cell carriage at the side so they are on top of the block that the cell will sit on during the run. Then drop the cell into the cell carriage, and as soon as it hits the thumb and forefinger, remove them, allowing the cell to have a reduced shock.

## 12. Calculations

12.1 Use Eq 2 to calculate the  $\Delta W$  values from the light intensity measured (either in percent or millivolts) at the upper and lower limit of each chosen range of particle diameters and from the arithmetic means of the particle range.

## 13. Report

13.1 The report may be of a single determination or an average with or without the individual determinations being listed, and should be so identified.

13.2 The report shall be identified with the condition of the powder analyzed, that is, either “as supplied” or “lab milled”, and, if dispersed by the 5-min hand-shake procedure, with “hand-shake.” Conversely, if the powder is dispersed by the 5-min ultrasonic treatment procedure, only the powder condition is identified.

13.3 Values shall be reported in weight percent to the nearest 0.1 % for each micrometre size interval calculated.

## 14. Precision and Bias <sup>10</sup>

14.1 *Precision*—At this time no full interlaboratory study on the precision of this test method exists. However, the user of this test method may get some indication of its precision from ASTM Research Report No. B9-1007, which presents the results of a study done in only three laboratories on tungsten and tungsten carbide powders with the two dispersants included in 6.1 (analyzed according to Practice E 691).

14.1.1 The within-laboratory repeatability limit,  $r$ , as defined by Terminology E 456, was found to be 3 to 5 weight % in each individual particle size range.

14.1.2 The between-laboratory reproducibility limit,  $R$ , as defined by Terminology E 456, was found to be 5 to 7 weight % in each individual particle range.

14.2 *Bias*—No absolute method of determining particle size distribution is universally recognized. Therefore, it is not possible to discuss the bias of results by this test method.

## 15. Keywords

15.1 de-agglomeration; laboratory milled; light-attenuation; particle size distribution; Photometer; rod milled; sedimentation; turbidimeter

<sup>10</sup> Supporting data are available from ASTM Headquarters. Request RR: B9-1007.



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