



Standard Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis¹

This standard is issued under the fixed designation B 859; 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 practice covers the de-agglomeration of refractory metal powders and their compounds in preparation for particle size analysis.

1.2 Experience has shown that this practice is satisfactory for the de-agglomeration of elemental tungsten, molybdenum, rhenium, and tantalum metal powders, and tungsten carbide. Other metal powders (for example, elemental metals, carbides, and nitrides) may be prepared for particle size analysis using this practice with caution as to effectiveness until actual satisfactory experience is developed.

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.* For specific precautionary statements, see Note 2.

2. Referenced Documents

2.1 ASTM Standards:

- B 243 Terminology of Powder Metallurgy²
- B 330 Test Method for Fisher Number of Metal Powders and Related Compounds²
- B 430 Test Method for Particle Size Distribution of Refractory Metal Powders and Related Compounds by Turbidimetry²
- B 761 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by X-Ray Monitoring of Gravity Sedimentation²
- B 821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis²
- B 822 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering²

2.2 ASTM Adjunct:

ADJB0859 Detailed Drawings of Alternative Steel Milling Bottles³

3. Terminology

3.1 *Definitions*—Definitions of powder metallurgy terms can be found in Terminology B 243.

4. Significance and Use

4.1 Refractory metal powders, such as tungsten and molybdenum, are usually produced by hydrogen reduction at high temperatures. Thus, they usually contain numerous large, strongly-sintered agglomerates. Many of the manufacturing processes using these powders involve a milling step or some similar treatment or depend on the individual particulate size, not on the agglomerate size.⁴ Thus, a knowledge of the individual particulate size distribution, not the agglomerate size distribution, is usually desired from a particle size analysis of these powders. This practice provides a procedure for breaking down agglomerates into their constituent particles (de-agglomeration), without excessive fracture of the individual particles. The procedure is often referred to as *laboratory milling* or *rod milling*.

4.2 The laboratory milling conditions specified in this guide have been in use since 1965 as part of Test Method B 430. This guide was first published as a separate, stand-alone standard in 1995 because of its applicability in preparing powder samples for analysis by other methods (for example, Test Methods B 761 and B 822), in addition to Test Method B 430. Information on the development and establishment of the milling conditions here specified can be found in the footnoted reference.⁵

4.3 The milling procedure described in this practice does not necessarily break down only agglomerates without fracturing individual particles; some particle fracture may occur in certain powders. However, use of this practice *does* provide

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² *Annual Book of ASTM Standards*, Vol 02.05.

³ Available from ASTM International Headquarters. Order Adjunct No. ADJB0859.

⁴ Michaels, A. I., "Turbidimetric Particle Size Distribution Theory: Application to Refractory Metal and Oxide Powders," *1958 Symposium on Particle Size Measurement*, ASTM STP 234, ASTM, 1959, pp. 207–244.

⁵ Buerkel, W. A., "Turbidimetric Particle Size Analysis as Applied to Tungsten Powder and the Carbide Industry," *Handbook of Metal Powders*, A. Poster, ed., Reinhold Publishing Corp., New York, NY, 1966, pp. 20–37.

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

consistent particle size analysis results that have been found to relate well to powder behavior in numerous manufacturing processes.

4.4 This practice shall be used for the de-agglomeration of the refractory metal powders and compounds listed in 1.1, when an evaluation of the individual particulate size distribution is required from the subsequent particle size analysis. It shall not be used when the *agglomerate* (as-is or as-supplied) size distribution is desired.

4.5 This practice may be used in preparing samples for Test Methods B 330, B 430, B 761, B 822, and other particle size analysis methods, prior to the dispersion procedure of Guide B 821, if used.

5. Apparatus

5.1 *Milling Bottle*—There are two alternative materials for the milling container:

5.1.1 *Glass Bottle*—Round laboratory solution bottle, 250-mL capacity⁶, or a 250-mL, 140-mm high, 60-mm diameter, wide-mouth, flat-bottom centrifuge bottle, with cap or stopper, or

5.1.2 *Stainless Steel Bottle*—Fabricated according to the detailed drawings in Adjunct No. ADJB0859.

5.2 *Tungsten Rods*—Fifty rods 75 ± 3 mm long by 4.0 ± 0.3 mm in diameter (approximately 3 in. by 0.15 in.), ground surface.

5.3 *Laboratory Jar Roll Mill*,⁷ capable of rotating the milling bottle at 145 rpm.

NOTE 1—If a jar roll mill is not available to give a bottle rpm of 145, the bottle can be either (1) set up on a lathe, or (2) built up in diameter and used on a faster rpm mill.

5.4 *Screen, No. 20 (850- μ m), and Bottom Pan.*

⁶ The solution bottle No. 16158-128 furnished by VWR Scientific Co., P.O. Box 7900, San Francisco, CA 94120, or bottle No. 05-587A furnished by Fisher Scientific, 711 Forbes Avenue, Pittsburgh, PA 15219, or their equivalent, have been found satisfactory.

⁷ The 0-300 rpm rollspeed, Model 755 Roll Mill, made by U.S. Stoneware, 40 Whitney Rd., Mahwah, NJ 07430, or its equivalent, has been found satisfactory.

6. Procedure

6.1 Place 30 ± 0.1 g of tungsten, molybdenum, rhenium, or tantalum metal, or 50 ± 0.1 g of tungsten carbide powder in the milling bottle containing the 50 tungsten milling rods.

6.2 Seal the milling bottle and rotate on the jar roll mill for $60 \text{ min} \pm 15 \text{ s}$ at a bottle speed of 145 ± 5 rpm. After the first 5 min, check to be sure the rods are cascading inside the bottle by listening for the fast-paced, repeated “clinking” sound that the cascading rods make. If the rods are not cascading, stop the mill, set the bottle upright momentarily, then replace the bottle on the mill and continue milling for the remaining 55 min (if the rods are now cascading; if not, repeat setting the bottle upright and restarting until they are).

6.3 After milling, immediately screen the powder through a No. 20 (850- μ m) screen to remove the milling rods. Dislodge any milled powder that might remain in the bottle by placing a couple of screened rods in the bottle and “whipping” for a few turns.

NOTE 2—**Warning:** The fresh metal surfaces produced during milling may have a tendency to rapidly oxidize when the milling bottle is opened. Use caution when opening the bottle to avoid fire or explosion.

6.4 Remove all the milled powder from the bottom pan and place in a sample container.

7. Particle Size Analysis

7.1 If necessary, disperse the milled powder according to Guide B 821.

7.2 Immediately perform the desired particle size analysis.

NOTE 3—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. 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, 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.

8. Keywords

8.1 de-agglomeration; laboratory-milled; laboratory milling; molybdenum; particle size analysis; powders; refractory metals; rhenium; rod-milled; rod milling; tantalum; tungsten; tungsten carbide

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes made to this standard since the last issue, B 859–95 (2000), that may impact the use of this standard. (Approved Oct. 1, 2003.)

(1) Stainless steel milling bottles were added as an alternative to the glass bottles in Sections 5.1 and 5.1.2.

(2) The adjunct number for the drawings of the stainless steel milling bottles was added in Section 2.2.

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