



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

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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 With the exception of the values for mass, for which the use of the gram (g) unit is the long-standing industry practice, the values stated in SI are to be regarded as standard. No other units of measure are included in this 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 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:*²

[B243 Terminology of Powder Metallurgy](#)

[B330 Test Methods for Estimating Average Particle Size of Metal Powders and Related Compounds Using Air Permeability](#)

[B761 Test Method for Particle Size Distribution of Metal](#)

¹ This practice 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.

[Powders and Related Compounds by X-Ray Monitoring of Gravity Sedimentation](#)

[B821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis](#)

[B822 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering](#)

2.2 *ASTM Adjunct:*

[ADJB0859 Detailed Drawings of Alternative Steel Milling Bottles](#)³

3. Terminology

3.1 *Definitions*—Definitions of powder metallurgy terms can be found in Terminology [B243](#).

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, initially as part of a particle size analysis test method. 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

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.

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