



Standard Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering¹

This standard is issued under the fixed designation B822; 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 test method covers the determination of the particle size distribution by light scattering, reported as volume percent, of particulate materials including metals and compounds.

1.2 This test method applies to analyses with both aqueous and nonaqueous dispersions. In addition, analysis can be performed with a gaseous dispersion for materials that are hygroscopic or react with a liquid carrier.

1.3 This test method is applicable to the measurement of particulate materials in the range of 0.4 to 2000 μm , or a subset of that range, as applicable to the particle size distribution being measured.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *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*:²

B215 Practices for Sampling Metal Powders

B243 Terminology of Powder Metallurgy

B821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis

E1617 Practice for Reporting Particle Size Characterization Data

2.2 *ISO Standard*:³

ISO13320-1 Particle Size Analysis—Laser Diffraction Methods—Part 1: General Principles

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 B09.02 on Base 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.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

3. Terminology

3.1 *Definitions*—Definitions of powder metallurgy terms can be found in Terminology B243.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *background*—extraneous scattering of light by elements other than the particles to be measured; includes scattering by contamination in the measurement path.

3.2.2 *Fraunhofer Diffraction*—the optical theory that describes the low-angle scattering of light by particles that are large compared to the wavelength of the incident light.⁴

3.2.3 *Mie Scattering*—the complex electromagnetic theory that describes the scattering of light by spherical particles. It is usually applied to particles with diameters that are close to the wavelength of the incident light. The real and imaginary indices of light refraction of the particles are needed.⁴

3.2.4 *multiple scattering*—the rescattering of light by a particle in the path of light scattered by another particle. This usually occurs in heavy concentrations of a particle dispersion.

4. Summary of Test Method

4.1 A prepared sample of particulate material is dispersed in water, or a compatible organic liquid, and circulated through the path of a light beam or some other suitable light source. A dry sample may be aspirated through the light in a carrier gas. The particles pass through the light beam and scatter it. Photodetector arrays collect the scattered light that is converted to electrical signals, which are then analyzed in a microprocessor. The signal is converted to a size distribution using Fraunhofer Diffraction or Mie Scattering, or a combination of both. Scattering information is analyzed assuming a spherical model. Calculated particle sizes are therefore presented as equivalent spherical diameters. Additional information pertaining to the general principles of particle size distribution analysis by light scattering can be found in ISO Standard 13320-1.

5. Significance and Use

5.1 Reported particle size measurement is a function of both the actual particle dimension and shape factor as well as the

⁴ Muly, E. C., Frock, H. N., "Industrial Particle Size Measurement Using Light Scattering," *Optical Engineering*, Vol 19, No 6, 1980, pp. 861–869.

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

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.

5.1.1 It is important to recognize that the results obtained by this test method, or any other method for particle size determination using different physical principles, may disagree. The results are strongly influenced by the physical principles employed by each method of particle size analysis. The results of any particle sizing method should be used only in a relative sense; they should not be regarded as absolute when comparing results obtained by other methods.

5.2 Light scattering theory has been available for many years for use in the determination of particle size. Several manufacturers of testing equipment now have units based on these principles. Although each type of testing equipment uses the same basic principles for light scattering as a function of particle size, different assumptions pertinent to application of the theory, and different models for converting light measurements to particle size, may lead to different results for each instrument. Therefore, the use of this test method cannot guarantee directly comparable results from different types of instruments.

5.3 Knowledge of the particle size distribution of metal powders is useful in predicting the powder-processing behavior and ultimate performance of powder metallurgy parts. Particle size distribution is related closely to the flowability, moldability, compressibility, and die-filling characteristics of a powder, as well as to the final structure and properties of finished powder metallurgy (P/M) parts.

5.4 This test method is useful to both suppliers and users of powders in determining the particle size distributions for product specifications, manufacturing control, development, and research.

5.5 This test method may be used to obtain data for comparison between lots of the same material or for establishing conformance, as in acceptance testing.

6. Interferences

6.1 Air bubbles entrained in the circulating fluid will scatter light and then be reported as particles. Circulating fluids may not require degassing, but they should be bubble-free upon visual inspection.

6.2 Contaminants, such as nonaqueous solvents, oil, or other organic coatings on the sample, may emulsify in an aqueous carrier, scatter light, and thus be reported as part of the particle size distribution. Samples containing such contaminants may be analyzed in a nonaqueous carrier solvent to dissolve the contaminant, or they may be washed free of the contaminant with a compatible aqueous solvent.

6.3 The presence of oil, water, or foreign substances in a gaseous dispersion will cause clogging or agglomeration or will bias the particle size results. The gas supplied should be free of these substances.

6.4 Reagglomeration or settling of particulates during analysis will cause erroneous results. Dispersions shall be prepared in accordance with Guide B821, and a stable dispersion shall be maintained throughout the analysis.

6.5 Insufficient sample loading may cause electrical noise interference and poor data repeatability. Excessive sample loading may cause excessive light attenuation and multiple scattering, resulting in erroneous particle size distributions.

7. Apparatus

7.1 *Particle Size Analyzer*, based on Fraunhofer Diffraction or Mie Scattering, or a combination of both light scattering analysis techniques. Care must be taken to ensure that the analyzer system or subsystem is optimum for the size range of the powder being tested.

7.2 *Liquid or Gaseous Sample Handling System*.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.⁵ 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.

8.2 *Appropriate Application-Specific Carrier*, as determined by Guide B821. The carrier shall meet the following conditions:

8.2.1 It shall be chemically compatible with the construction material of the sample delivery system,

8.2.2 It shall not cause dissolution of the particles, and

8.2.3 It shall be sufficiently clean and non-absorbing to achieve acceptable background levels.

8.3 *Antifoaming Agent*, or equivalent.

8.4 *Dry, Clean Gas*, for gaseous dispersions.

8.5 *Appropriate Surfactant*, as determined by Guide B821, subject to the conditions listed in 8.2.

9. Sampling and Sample Size

9.1 Obtain a test sample according to Practices B215. The test portion shall be extracted from the test sample using a microsample splitter; quartering shall not be used.

9.2 The maximum test sample for liquid dispersion will be no more than 25 g. No more than 500 g will be needed for a gaseous dispersion.

10. Calibration and Standardization

10.1 Performance of the instrument is defined by the spacing and position of the optical components (refer to the instruction manual provided by the manufacturer).

⁵ *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.

10.2 No absolute standards are available for particle size analysis. Diagnostic powders should be available from the equipment manufacturer to ensure consistent instrument functioning.

11. Procedure

11.1 Allow the instrument to warm up for a minimum of 20 min.

11.2 Install the desired sample delivery system and select the applicable instrument range, as indicated by the instructions provided by the instrument manufacturer.

11.3 Establish correct optical alignment, if necessary, according to the requirements of the manufacturer.

NOTE 1—Optical alignment should be checked upon startup, whenever the sample delivery system is changed, or at least once a day.

11.4 Measure the background in the mode in which the analysis will be conducted. Be sure that the carrier is flowing through the light path while measuring background. Background values shall not exceed the specifications of the manufacturer. If background values exceed the recommendations of the manufacturer, perform the necessary procedures as specified by the manufacturer to bring the background values to within acceptable limits.

11.5 Obtain a representative test sample according to Practices B215. Extract a test portion from the test sample using a microsample splitter. Refer to the recommendations of the equipment manufacturer to ensure that the amount of the test portion is acceptable to achieve optimum light scattering conditions. A wide range of sample sizes is acceptable, depending on median particle size (50 %), particle density (mass/volume), and sample delivery system.

11.6 Select the appropriate run time for the sample. This procedure is very specific to the application and is generally gaged by the run-to-run repeatability.

11.7 Select the desired data output parameters, according to the requirements set forth by the instrument manufacturer.

11.8 For liquid dispersions, disperse the test portion according to the procedure outlined in Guide B821. Gaseous dispersions require no additional sample preparation.

11.9 Transfer the prepared sample directly to the sample delivery system. In the liquid system, allow circulation for 20 s before measuring. In the dry sampling system, engage the sample switch to allow the sample to begin to flow into the light source before starting measurement.

11.10 Perform the sample analysis according to the instructions of the manufacturer.

11.11 In the liquid system, drain and fill the sample dispersion system in preparation for the next sample analysis. Drain and rinse as necessary, to achieve background values within acceptable operating limits, as specified by the manufacturer.

NOTE 2—When changing from either polar to a nonpolar organic liquid or nonpolar to polar liquid, it will be necessary to rinse the sample delivery system several times with a compatible solvent, such as alcohol, to eliminate the formation of an interference emulsion due to cross contamination of the two carriers.

11.12 In the dry gaseous system, brush or vacuum out all particles throughout the sample system. Purge with air to remove particles remaining in the sample delivery system.

11.13 Repeat Steps 11.5 through 11.11 for additional test portion analyses.

12. Report

12.1 Practice E1617 specifies three detail levels for reporting particle size characterization data. It is up to the supplier and the user of the data to determine which level of reporting is needed. As a minimum, report the following information:

12.1.1 The instrument name and model number used and the range selected,

12.1.2 The method of dispersing the test portion,

12.1.3 The instrument analysis run time,

12.1.4 Any curve fit models used (where applicable),

12.1.5 The real and imaginary refractive index of the sample material (where applicable),

12.1.6 The real refractive index of the dispersing liquid,

12.1.7 The volume mean diameter,

12.1.8 The differential volume percent versus diameter, and

12.1.9 The cumulative volume percent versus diameter.

13. Precision and Bias

13.1 An interlaboratory study is being initiated to determine the precision of this test method.

13.2 *Precision*—The repeatability standard deviation of volume mean diameter for one cobalt sample has been determined to be ± 0.4 % relative standard deviation, based upon analysis in one laboratory. The reproducibility of this test method is being determined and will be available on or before June 30, 2012.

13.3 *Bias*—No information can be presented on the bias of the procedure in this test method for measuring particle size distribution because no metal powder having an accepted reference value is available.

14. Keywords

14.1 laser diffraction; light scattering; metal powders; particle size distribution; powder metallurgy

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B822 - 02) that may impact the use of this standard. (May 1, 2010)

(1) Section 13 has been updated to include a temporary precision statement to be used while the interlaboratory study is underway, and to state that bias cannot be defined for this type analysis. Rationale: The repeatability of a single labora-

tory will provide some guidance to the user of the test method as to what to expect from use of the procedures described here until a final precision statement can be completed.

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