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Standard Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis¹

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1. Scope*

1.1 This guide covers the dispersion in liquids of metal powders and related compounds for subsequent use in particle size analysis instruments. This guide describes a general procedure for achieving and determining dispersion; it also lists procedures that have been found useful for certain materials.

1.2 This guide does not include specific procedures for *dry* dispersion of particulate materials. It only indicates when liquid dispersion is not appropriate and dry dispersion must be utilized (see 7.1.2.1). For guidance on development of methods of *dry* dispersion, see Guide E3340.

1.3 This guide is limited to metal powders and related metal compounds. However, the general procedure described herein may be used, with caution as to its significance, for other particulate materials, such as ceramics, pigments, minerals, etc.

1.4 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This guide 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. Referenced Documents

2.1 *ASTM Standards*:²

B215 Practices for Sampling Metal Powders

B243 Terminology of Powder Metallurgy

B761 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by X-Ray Monitoring of Gravity Sedimentation

B822 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering

E3340 Guide for Development of Laser Diffraction Particle Size Analysis Methods for Powder Materials

3. Terminology

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

4. Significance and Use

4.1 The method of powder dispersion in a liquid has a significant effect on the results of a particle size distribution analysis. The analysis will show a too-coarse, unstable, or nonrepeatable distribution if the powder has not been dispersed adequately. It is therefore important that parties wishing to compare their analyses use the same dispersion technique.

4.2 This guide provides ways of deriving dispersion techniques for a range of metal powders and compounds. It should be used by all parties performing liquid-dispersed particle size analysis of all of the materials covered by this guide (see 1.1, 1.2, and 4.1).

4.3 Table 1 provides some dispersion procedures that have been found useful and consistent for the particular materials listed there. These are only *suggested* dispersion procedures; the procedures and dispersion checks of 7.1.2 – 7.1.4, or the more detailed method development procedures of Guide

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.

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

TABLE 1 Useful Dispersion Procedures for Some Specific Materials

Material	Carrier Liquid	Additive Surfactant or Stabilizer	Additive Concentration	Ultrasonic Treatment ^A		
				Type	Power Level, W	Time, min
Chromium carbide	water	none	...	none or bath
Copper	water	Tween 21 ^B	3–5 drops ^C	bath	25	5
Ferroalloys	isopropyl alcohol	Tween 21 ^B	10 %	bath	80	1
Iron/steel	water	Tween 21 ^B	3–5 drops ^C	bath	80	1
Manganese sulfide	water	Tween 21 ^B	3–5 drops ^C	bath	80	1
Molybdenum	water	sodium hexametaphosphate	0.01 %	probe or bath or bath	160 80	3 10
Nickel	water	Tween 21 ^B	3–5 drops ^C	bath	25	5
Tantalum	water	sodium hexametaphosphate	0.01 %	probe or bath	80 160	1 3
Tantalum carbide	water	sodium hexametaphosphate	0.01 %	probe or bath or bath	160 80	3 10
Tungsten ($D_{50} \leq 20 \mu\text{m}$)	water	sodium hexametaphosphate	0.01 %	probe or bath	160	3
($D_{52} > 20 \mu\text{m}$, Run Dry)				probe or bath	80	10
Tungsten carbide ($D_{50} \leq 20 \mu\text{m}$)	water	sodium hexametaphosphate	0.01 %	probe or bath	160	3
($D_{52} > 20 \mu\text{m}$, Run Dry)				probe or bath	80	10
				probe or bath	25	5

^A Stated ultrasonic power and duration times are given as an indication only. Specific conditions should be sought for the particular system in question during the method development phase.

^B Tween 21, chemically known as polyoxyethylene⁶ sorbitan monolaurate, is manufactured by Croda International PLC, and is available from various chemical suppliers.

^C Three to five drops Tween 21 in 30 to 50 mL water.

E3340, should still be used to verify adequate dispersion for each particular material and particle size range.

4.4 This guide should be used in the preparation of powders for use in Test Methods **B761** and **B822** and other procedures that analyze metal powder particle size distributions in liquid-dispersed systems.

5. Apparatus

5.1 *Microscope*, suitable for observation of particles in the size range of 5 to 1000 μm .

5.2 *Ultrasonic Probe*, 12.7 mm tip, with the power level to be determined by this guide.

5.3 *Ultrasonic Bath*—Power level to be determined by this guide.

6. Reagents

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

Society.³ 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.

6.2 *Surfactants/Stabilizers*—Some suggested surfactants or stabilizers are listed in **Table 1** and in the references of Footnotes 4 through 6.^{4,5,6}

³ *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.

⁴ Allen, T., *Particle Size Measurement*, 4th Edition, Chapman and Hall, London, UK, 1991.

⁵ Nelson, R. D., *Dispersing Powders in Liquids*, Elsevier, New York, NY, 1988.

⁶ *SediGraph III 5120 Operator's Manual*, Micromeritics Corporation, Norcross, GA, 1998, pp. C-3, C-4, and H-3.

7. General Dispersion Procedure

7.1 The general procedure for determining and achieving proper dispersion is outlined in Fig. 1⁷ and described in detail below:

7.1.1 After observing the powder in a microscope to get a general idea of size, brittleness, and agglomeration state, place a test portion of the powder to be analyzed, obtained according to Practices B215, in a beaker containing the carrier liquid, selected according to 7.1.2.

7.1.2 Selection of Carrier Liquid:

NOTE 1—The selected carrier liquid must be compatible with the components of the instrument used for the particle size analysis.

7.1.2.1 If the powder reacts with, or is soluble in, water and organic liquids, or settles in the beaker while stirring, it must be analyzed in the dry state, and the remainder of this guide is then not applicable.

7.1.2.2 If the powder reacts with, or is soluble in, water, but not organic liquids, select an appropriate organic liquid.

7.1.2.3 If the powder is neither reactive nor soluble in water, select distilled or deionized water as the carrier liquid.

7.1.3 Selection of Additive—If the powder is not wettable by the chosen carrier liquid, select a suitable additive (dispersing agent or stabilizer).

NOTE 2—Ultrasonic energy treatment may be necessary to separate particles so that the individual particles may be wetted by the carrier liquid or liquid/additive solution.

NOTE 3—Suggested additives are listed in Table 1 and Footnotes 4 through 6.^{4,5,6}

7.1.3.1 The appropriate additive and its concentration are determined by trial and error; a series of concentrations of different candidate additives must be tried on separate samples and the resultant particle size distribution analyses compared. The optimum additive and concentration are usually those that produce the finest particle size distribution results.

NOTE 4—Excess additive may cause foaming or a coarser particle size distribution in the subsequent particle size analysis.

7.1.4 Dispersion Check:

7.1.4.1 Determine whether the powder is dispersed in the liquid by examining it carefully in a beaker during and after stirring. If the powder appears to be distributed uniformly throughout the liquid, and does not flocculate within a few seconds after the discontinuation of stirring, particle size analysis can then be performed (9.1) and the results evaluated. Some large particles of high-density materials may not remain suspended and settle out before the particle size analysis can be performed; in this case, *dry* analysis is recommended. See Guide E3340.

7.1.4.2 Ultrasonic Energy Treatment—Even if the powder appears to be uniformly dispersed, ultrasonic energy treatment may be necessary.

NOTE 5—Ultrasonic treatment may also be necessary to break up agglomerates in powders that appear to be dispersed, unless the agglomerate distribution is desired from the subsequent analysis.

7.1.4.3 Disperse the sample by placing the carrier liquid/sample beaker in an ultrasonic bath or by inserting an ultrasonic probe into the liquid/sample mixture. Continuous stirring

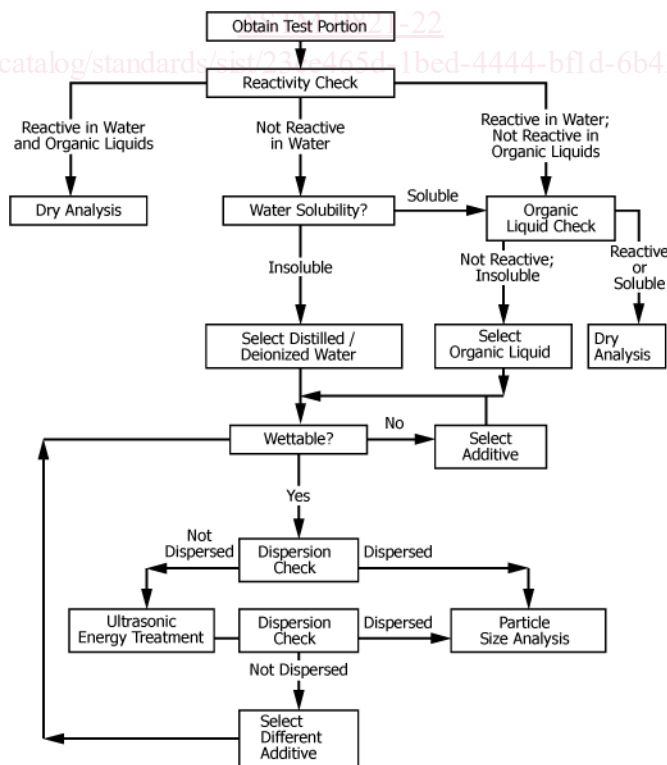


FIG. 1 General Dispersion Procedure

⁷ Microtrac Course Manual, Leeds and Northrup Company, St. Petersburg, FL, 1989.