Designation: B 821 – 02

Standard Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis[1]

This standard is issued under the fixed designation B 821; 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 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 are currently in general use for certain materials.

1.2 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.

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

B 243 Terminology of Powder Metallurgy[2]

B 430 Test Method for Particle Size Distribution of Refractory Metal Powders and Related Compounds by Turbidimetry[2]

B 761 Test Method for Particle Size Distribution of Refractory Metals and Their Compounds by X-Ray Monitoring of Gravity Sedimentation[2]

B 822 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering[2]

3. Terminology

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

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 established powder dispersion techniques for certain materials and the means of deriving techniques for materials not listed. 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 This guide should be used in the preparation of powders for use in Test Methods B 430, B 761, and B 822 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*, ½ -in. (25.4-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*—Suggested surfactants are listed in Table 1 and footnotes 4 through 6.[4,5,6]

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[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] *Annual Book of ASTM Standards*, Vol 02.05.

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

TABLE 1 Recommended Dispersion Procedures

Material	Carrier Liquid	Surfactant	Surfactant Concentration	Ultrasonic Treatment		
				Type	Power Level, W	Time, min
Chromium carbide	water	none	...	none	...	...
				or[A]		
				bath	25	5
Copper	water	Tween 21[B]	3–5 drops[C]	bath	80	1
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	160	3
				or		
				bath	80	10
				or[A]		
				bath	25	5
Nickel	water	Tween 21[B]	3–5 drops[C]	bath	80	1
Tantalum	water	sodium hexametaphosphate	0.01 %	probe	160	3
				or		
				bath	80	10
Tantalum carbide	water	sodium hexametaphosphate	0.01 %	probe	160	3
				or		
				bath	80	10
				or[A]		
				bath	25	5
Tungsten	water	sodium hexametaphosphate	0.01 %	probe	160	3
				or		
				bath	80	10
				or[A]		
				bath	25	5
Tungsten carbide	water	sodium hexametaphosphate	0.01 %	probe	160	3
				or		
				bath	80	10
				or[A]		
				bath	25	5

[A] As described in Test Method B 430.

[B] Tween 21, chemically known as polyoxyethylene[6] sorbitan monolaurate, is available from ICI Americas, Inc., Specialty Chemicals Division, Murphy and Concord Pike, Wilmington, DE 19897.

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

7. General Dispersion Procedure

7.1 The general procedure for determining and achieving proper dispersion is outlined in Fig. 1[7] and described in detail below:

7.1.1 Place a test portion of the powder to be analyzed 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, it must be analyzed in the dry state, and the remainder of this guide is then not applicable.

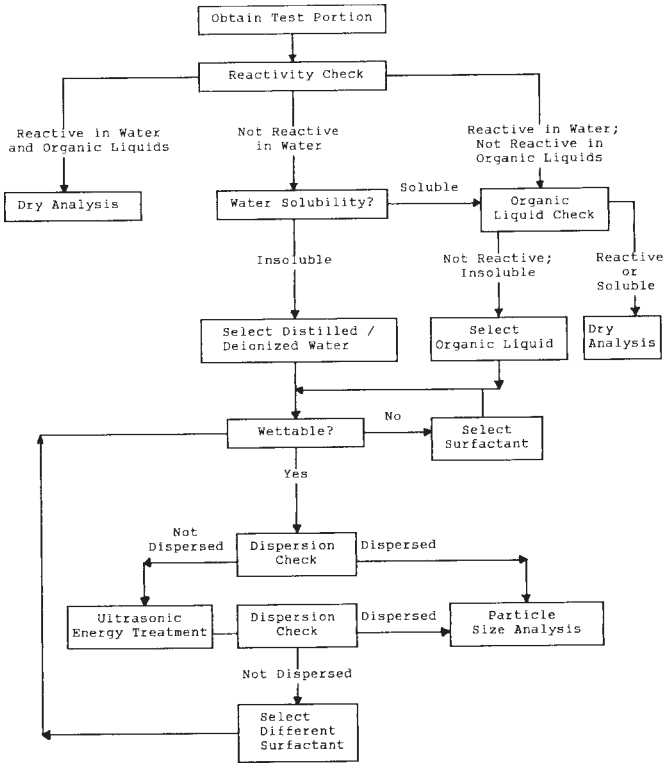

FIG. 1 General Dispersion Procedure

[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 5100 Windows Software Operator's Manual*,, Micromeritics Corporation, Norcross, GA, 1998, pp. C-1, C-2, H-1, and H-2.

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

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 Surfactant*—If the powder is not wettable by the chosen carrier liquid, select a suitable surfactant (dispersing agent).

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/surfactant solution.

NOTE 3—Suggested surfactants are listed in Table 1 and footnotes 4 through 6.[4,5,6]

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

NOTE 4—Excess surfactant may cause 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.

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 of the liquid/sample mixture may be necessary through part or all of the ultrasonic treatment. As with surfactant selection (7.1.3.1), the appropriate time and power level for ultrasonic treatment must be determined by trial and error. Select the time and power level by using the minimums necessary to ensure precision and adequate dispersion, as determined in 7.1.4.1. The optimum ultrasonic treatment is usually that which produces the finest particle size distribution results without fracturing the individual particles.

NOTE 6—Particle fracture can be evaluated by examining the treated powder in a suitable microscope and noting whether the particle shape or distribution has changed significantly as the power level or treatment time has been increased. Fracture of particles is also often indicated by a shift from a unimodal to bimodal particle size distribution as the ultrasonic power level or treatment time is increased.

NOTE 7—Some indication of the type of equipment, starting times, and power levels for ultrasonic energy treatment may be obtained from Table 1.

7.1.4.4 Check for dispersion, as in 7.1.4.1. If the powder is now well-dispersed, continue with the particle size analysis (9.1).

7.1.4.5 If the powder is still not well-dispersed after ultrasonic energy treatment, select a different surfactant and repeat the steps given in 7.1.3 and 7.1.4 (and their relevant subparagraphs). Continue with this repetitive process until dispersion is attained.

8. Recommended Dispersion Procedures

8.1 Table 1 lists the dispersion procedures currently in general use for several metals and metal compounds. These procedures have been shown by experience to produce consistent, reproducible particle size analysis results for the materials listed.

9. Particle Size Distribution Analysis

9.1 After dispersion has been achieved by one of the above techniques, immediately perform the required particle size analysis by whatever method is applicable (for example, Test Method B 430, B 761, or B 822).

10. Keywords

10.1 liquid dispersion; metal powders; particle size analysis; powder metallurgy

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B 821 – 92(1997)) that may impact the use of this standard.

(1) Paragraphs 4.2, 4.3, and 6.1 were modified to replace the word “shall” with the word “should” in all occurrences. Rationale: This standard is a guide and not a practice. Since a guide is used to recommend a course of action rather than to specify one, each occurrence of the word “shall” was replaced with the word “should.”

(2) Footnote 6 was modified to bring it up to date with the current version of the referenced document. Rationale: The referenced document is no longer available, but a new version contains the pertinent information.