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StandardGuide for Powder Particle Size Analysis[1]

This standard is issued under the fixed designation E2651; 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 use of many available techniques for particle size measurement and particle size distribution analysis of solid particulate (powder) materials. It does not apply to analysis of liquid droplets or liquid aerosols. The guide is intended to serve as a resource for powder/particle technologists in characterizing their materials.

1.2 This guide provides more detail regarding the particle size analysis methods listed in Guide E1919, which is a compilation of worldwide published standards relating to particle and spray characterization. Although Guide E1919 and this guide are both extensive, neither is all inclusive.

1.3 The principle of operation, range of applicability, specific requirements (if any), and limitations of each of the included particle size analysis techniques are listed and described, so that users of this guide may choose the most useful and most efficient technique for characterizing the particle size distribution of their particular material(s).

1.4 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 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

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

C322 Practice for Sampling Ceramic Whiteware Clays

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E1617 Practice for Reporting Particle Size Characterization Data

E1638 Terminology Relating to Sieves, Sieving Methods, and Screening Media

E1919 Guide for Worldwide Published Standards Relating to Particle and Spray Characterization

E2589 Terminology Relating to Nonsieving Methods of Powder Characterization

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this guide, refer to Terminologies E1638 and E2589.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *powder, n*—a collection of solid particles that are usually less than 1000 μm (1 mm) in size.

4. Significance and Use

4.1 The myriad array of particle size analysis techniques available to the modern-day powder technologist is both daunting and confusing. Many of the techniques are applicable only to certain types of materials, and all have limited ranges of applicability with respect to powder particle size. This guide is an attempt to describe and define the applicability of each of the available techniques, so that powder technologists, and others interested in powders, may make informed and appropriate choices in characterizing their materials.

4.2 This guide is intended to be used to determine the best and most efficient way of characterizing the particle size distribution of a particular powder material. It may also be used to determine whether a reported powder particle size, or size distribution, was obtained in an appropriate and meaningful way.

4.3 Most particle size analysis techniques report particle size in terms of an "equivalent spherical diameter": the diameter of an ideal spherical particle of the material of interest that would be detected in the same manner during analysis as the (usually irregular-shaped) actual particle under the same conditions. The different techniques must necessarily use different definitions of the equivalent spherical diameter, based

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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.

on their different operating principles. However, when analyzing elongated particles, the size parameter most relevant to the intended application should be measured; for example, length (maximum dimension).

4.4 Reported particle size measurement is a function of both the actual dimension and/or shape factor as well as the particular physical or chemical properties of the particle 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. Reagents

5.1 *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.

5.2 *Surfactants*—Suitable surfactants are listed in references (**1-3**).[4]

6. Sampling

6.1 The first step in performing a powder particle size analysis is obtaining a sample of the powder that is intended to be representative of the entire amount. There are two conditions necessary for obtaining an accurate sample of a powder (**4**): The first is that the sampling must be probabilistic; that is, every increment of the powder must have *some* probability of being selected in the sampling process. The sampling must not only be probabilistic, it must also be correct. That means that *every* sample increment must have an equal probability of being chosen. No method of sampling can *guarantee* a representative sample, but adherence to certain "Golden Rules of Sampling" (**1**) will satisfy these two conditions and ensure a sample as close to representative as possible. These rules are:

6.1.1 Always sample a powder in motion (for example, pouring from a blender, or off the end of a conveyor).

6.1.2 Take small portions for many short increments of time from the *whole* stream of powder.

6.1.3 *Never* scoop a sample from a heap or container of powder.

6.2 The preferred method of sampling is to use a spinning (rotary) riffler; however, this is not always possible. Devices that adhere to these rules, such as chute rifflers, spinning rifflers, and stream samplers, are available commercially. Examples of good powder sampling practices may be found in Practices B215 and C322.

7. Dispersion

7.1 The method of powder dispersion has a significant effect on the results of a particle size distribution analysis. The analysis will show a too-coarse, unstable, or non-repeatable 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.

7.2 Many particle size analysis instruments are capable of, or require, dispersing powders in a liquid medium. Guide B821 contains recommended liquid dispersion procedures for certain metal powders and related compounds. That guide also contains general procedures for dispersing powders in liquids, and assessing dispersion. Those general procedures are repeated here:

7.3 The general procedure for determining and achieving proper dispersion is outlined in Fig. 1 (**5**) and described in detail below:

7.3.1 Place a test portion of the powder to be analyzed in a beaker containing the carrier liquid, selected according to 7.3.2.

7.3.2 *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.

7.3.2.1 If the powder reacts with, or is soluble in, water and organic liquids, it must be analyzed in the dry state. Some particle size analysis instruments have built-in systems for de-agglomerating and dispersing dry powders. Consult the instrument manufacturer's operating manual. See 7.4 for further guidance on dry dispersion.

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

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

7.3.3 *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—Suitable surfactants are listed in references (**1-3**).

7.3.3.1 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.

7.3.4 *Dispersion Check:*

7.3.4.1 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

[3] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC, www.chemistry.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, http://www.usp.org.

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.