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# Standard Guide for Powder Particle Size Analysis<sup>1</sup>

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 ( $\epsilon$ ) 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, off-line in a laboratory. It does not apply to in-line (on-line) analysis, nor 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 significant detail regarding the numerous particle size analysis methods available. Although this guide is extensive, it may not be 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, 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.*

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee E29 on Particle and Spray Characterization and is the direct responsibility of Subcommittee E29.02 on Non-Sieving Methods.

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## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

[B215 Practices for Sampling Metal Powders](#)

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

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

[B859 Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis](#)

[C322 Practice for Sampling Ceramic Whiteware Clays](#)

[E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves](#)

[E161 Specification for Electroformed Material and Test Sieves](#)

[E1617 Practice for Reporting Particle Size Characterization Data](#)

[E1638 Terminology Relating to Sieves, Sieving Methods, and Screening Media](#)

[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  $\mu\text{m}$  (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

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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

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 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 or shape factor, or both, 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.<sup>3</sup> 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).<sup>4</sup>

## 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).

<sup>3</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC, [www.chemistry.org](http://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>.

<sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

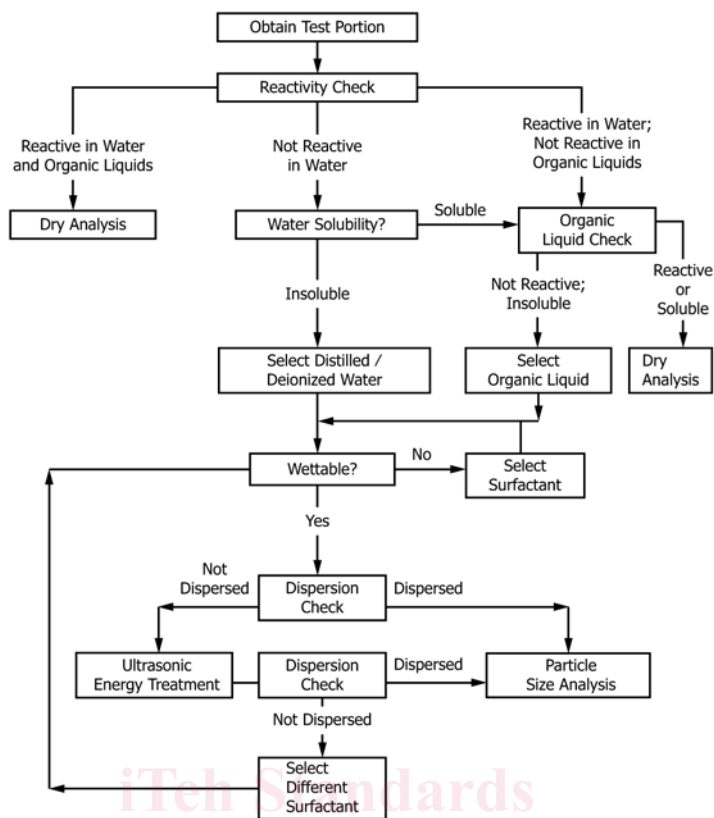


FIG. 1 General Dispersion Procedure (5)

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 seconds after the discontinuation of stirring, particle size analysis can then be performed and the results evaluated. In addition, the use of optical microscopy to directly observe the state of dispersion is recommended.

7.3.4.2 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.

7.3.4.3 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 (7.3.3.1), 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 7.3.4.1. 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 with 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 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 in Guide B821.

7.3.4.4 Check for dispersion, as in 7.3.4.1. If the powder is now well-dispersed, continue with the particle size analysis.

7.3.4.5 If the powder is still not well-dispersed after ultrasonic energy treatment, select a different surfactant, or combination of surfactants, and repeat the steps given in 7.3.3 and 7.3.4 (and their relevant subparagraphs). Continue with this repetitive process until dispersion is attained.

7.4 Dry dispersion is sometimes preferred to wet dispersion, especially when working with static image analysis techniques. Sometimes, dry dispersion is the only alternative (see 7.3.2.1).

7.4.1 Many dry dispersion units are based on vacuum or pressurized gas flow, or both. The generated gas flow sprays the particles inside a closed chamber or through an adjustable orifice, and they fall back on suitable media like microscope glass slides, or are directly introduced into the instrument's measurement zone. The user should adjust the unit's parameters to obtain a good dispersion, with a minimum of agglomerates, and without damaging the particles. The gas flow path, the pressure difference, and the gas release interval are examples of variables that should be adjusted by consulting the manufacturer's guide.

## PARTICLE SIZE ANALYSIS TECHNIQUES

### 8. Sieving

8.1 *Principle of Operation*—Sieving consists of passing a powder through a screen (sieve) with a specified opening size and measuring, by weighing, the amount of powder either remaining on the sieve or passing through. A particle size distribution may be obtained by stacking sieves of increasing opening size and measuring the amount collected on each sieve.

8.2 *Particle Size Range of Application*—Although sieves are available with aperture sizes down to about 5  $\mu\text{m}$ , the practical lower limit for sieve analysis depends on the method of sieving, sieve shaker effectiveness and ability of user material to flow through desired sieve sizes in use. Specifications E11 and E161 specify aperture sizes and tolerances for sieve openings.

8.3 *Specific Requirements*—Because there are many ways of agitating sieves (sieve shakers, ultrasonics, etc.), the method of agitation and the duration of agitation must be standardized. Guidelines for establishing sieve analysis procedures can be found in the *Manual on Test Sieving Methods*(6).

#### 8.4 *Limitations:*

8.4.1 Sample size varies dependent on sieve shaker, sieve diameter and sieve opening sizes in use. Sample size is variable dependent on sieving area and user material specific gravity. Usually not more than two layers of material should be retained on any one sieve.

8.4.2 Information about the largest and smallest particles in the powder is not available, only that they are larger than, or smaller than, a specified size.

### 9. Sedimentation

9.1 *Principle of Operation*—Sedimentation analysis is based on Stokes Law (7), which mathematically states that the time of fall of a particle settling in a straight line through a viscous medium is proportional to the particle's size and to its density. The rate of sedimentation is sometimes measured by weighing the settling powder at the bottom of a sedimentation column as a function of time (referred to as a "sedimentation balance"). More often, sedimentation is monitored by attenuation of a light or X-ray beam: The intensity of a collimated beam transmitted through a suspension (usually liquid) of

powder increases with time, indicating the concentration of particular particle sizes at specific times. This intensity distributed over time is a measure of the particle size distribution.

9.2 *Particle Size Range of Application*—The usual range of sedimentation analysis is 0.1 to 500  $\mu\text{m}$ . The range can be extended down to 0.01  $\mu\text{m}$  by combining sedimentation with centrifugation (see Section 10).

#### 9.3 *Specific Requirements:*

9.3.1 A powder suspension that remains dispersed in a static liquid is necessary. (See Section 7.)

9.3.2 For light or X-ray sedimentation, the suspension must be very dilute in order to transmit a measurable intensity at full dispersion.

9.3.3 Vibration must be limited during analysis, as vibration will affect sedimentation in unpredictable ways.

9.3.4 A constant temperature must be maintained during analysis, as temperature changes will affect the liquid viscosity, thereby changing the sedimentation rate.

9.3.5 The density of the powder material, plus the viscosity and density of the liquid dispersing medium, at the analysis temperature, must all be known in order to calculate the particle size distribution in accordance with Stokes Law.

9.3.6 For X-ray sedimentation, the powder material must fully absorb X-rays.

#### 9.4 *Limitations:*

9.4.1 Since sedimentation analysis depends on the density of the material analyzed, only homogeneous materials of a single, known density may be analyzed in this way if fundamental particle size is desired. However, sedimentation analysis may be used to comparatively *characterize* mixtures of materials of differing densities, with caution as to the significance of the particle size information obtained.

9.4.2 Since a dilute suspension is necessary, concentrated slurries cannot be analyzed by sedimentation.

9.4.3 Coarse, high-density materials may decrease the upper particle size limit, as it is difficult to keep these materials suspended long enough to obtain the analysis results.

## 10. Centrifugal Sedimentation

10.1 *Principle of Operation*—Like ordinary sedimentation analysis, centrifugal sedimentation analysis is based on Stokes Law (7), which mathematically states that the time of fall of a particle settling through a viscous medium is proportional to the particle's size and to its density. The rate of sedimentation is most often monitored by attenuation of a light or X-ray beam: The intensity of a collimated beam transmitted through a suspension (usually liquid) of powder either increases or decreases with time, indicating the concentration of particular particle sizes at specific times. This intensity distributed over time is a measure of the particle size distribution. In the case of low density small particles, a centrifuge is employed to accelerate the analysis and to be certain that Reynolds number requirements are met.

NOTE 8—Settling in centrifugal sedimentation is not in a straight line, but parabolic.

10.2 *Particle Size Range of Application*—The usual range of centrifugal sedimentation analysis is 0.01 to 50  $\mu\text{m}$ .