



Standard Guide for Powder Particle Size Analysis¹

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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

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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.

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 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 riffles, spinning riffles, 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 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.

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.

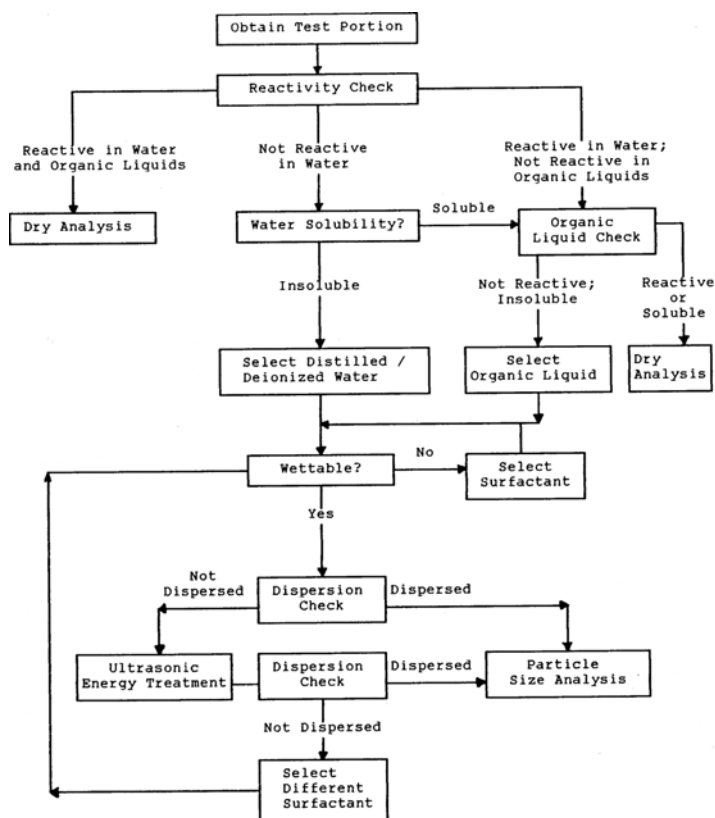


FIG. 1 General Dispersion Procedure (5)

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 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 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 μm , the practical lower limit for sieve analysis is usually considered to be 38 μm (400 mesh). At the upper end, Specification E11 specifies aperture sizes and tolerances for sieve openings up to 125 mm.

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 A relatively large sample, 50 to 100 g, is usually required for an accurate measurement of the mass of powder retained on each 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 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 μm . The range can be extended down to 0.01 μm 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.

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.

10.2 Particle Size Range of Application—The usual range of centrifugal sedimentation analysis is 0.01 to 50 μm .

10.3 Specific Requirements:

10.3.1 A powder suspension that remains dispersed in a circulating liquid is necessary.

10.3.2 For either type of sedimentation analysis, the suspension must be very dilute in order to transmit a measurable intensity at full dispersion.

10.3.3 The temperature of the liquid must be known in order to input the proper value of the liquid viscosity.

10.3.4 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.

10.3.5 The powder material must absorb or block light or X-rays, depending on which is used to monitor the sedimentation rate.

10.4 Limitations:

10.4.1 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.

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

11. Electrical Sensing Zone

11.1 Principle of Operation—Particles suspended in a liquid electrolyte are passed through a small aperture, across which is applied an electric field. As each particle passes through the aperture (or "sensing zone") it displaces its own

volume of conducting liquid, momentarily increasing the impedance of the aperture by an amount proportional to the particle's size. Instruments using this principle count individual particles and sort them according to size.

11.2 *Particle Size Range of Application*—0.4 to 1200 μm .

11.3 *Specific Requirements:*

11.3.1 An electrolyte solution compatible with the material being analyzed must be formulated. The electrolyte must have an electrical conductivity significantly different from that of the analyte.

11.3.2 A powder suspension that remains dispersed in the electrolyte liquid is necessary. (See Section 7.)

11.3.3 The suspension must be very dilute, so that particles may pass individually through the instrument aperture.

11.4 *Limitations:*

11.4.1 The entire particle size range of application stated above cannot be covered in one analysis. The size range of a particular analysis depends on the size of the aperture used. The particle size must be a significant fraction of the aperture size in order to detect a measurable difference in impedance. Therefore, some foreknowledge of the powder particle size is necessary to choose the appropriate aperture.

11.4.2 Since a dilute suspension is necessary, concentrated slurries cannot be analyzed by the electrical sensing zone method.

12. Static Image Analysis

12.1 *Principle of Operation*—A sample of particles is dispersed on a slide or other carrier and observed under an optical or electron microscope, stereoscope, or with a simple zoom. Particles generally fall on their more stable side so it is easier to evaluate the longest side, especially when analyzing elongated particles. Images are captured using a digital camera and analyzed using image analysis software. Both size and shape information can be obtained. Static image analysis allows measuring, among others, the longest and shortest dimensions (length and width) as well as the elongation factor (aspect ratio) for each particle.

12.2 *Particle Size Range of Application*—Using a good resolution digital camera, the minimum size that can be measured is based on the minimum resolution of the microscope optics. Typically, it ranges from 0.7 μm at 1000 $\times$ up to any size depending on the microscope. As long as the image of the particles can be captured by a digital camera, they can be analyzed.

12.3 *Specific Requirements:*

12.3.1 Particles must be dispersed as much as possible on the slide.

12.3.2 A tool that allows automatic separation of connected particles should be available in the image analysis software.

12.3.3 A tool to discriminate and eliminate remaining connected particles should be available in the image analysis software.

12.4 *Limitations:*

12.4.1 Several techniques and tools are available to considerably improve the sample dispersion. Even using them, some agglomerates may remain and be evaluated as single particles. If no tools are available in the image analysis software to identify and separate or eliminate them, the particle size may

be overestimated and the shape measured may then also be rougher than in reality.

12.4.2 Since elongated particles tend to present their largest dimension to the microscope, the equivalent spherical diameter measured using static image analysis may be larger than that measured by other techniques

13. Dynamic Digital Image Processing (Photo Optical)

13.1 *Principle of Operation*—A sample of particulate material is dispersed in a gas (usually air) or liquid stream. As the particles pass through the measurement zone, they are illuminated, either from front or back, to create two-dimensional projected images that are captured by a digital camera and then analyzed using image analysis software. Both size and shape information can be obtained.

13.2 *Particle Size Range of Application*—0.7 μm to 300 μm .

13.3 *Specific Requirements:*

13.3.1 A powder suspension that remains dispersed in flowing air or liquid is necessary. (See Section 7.)

13.3.2 The powder must be free-flowing in the carrier medium (gas or liquid).

13.4 *Limitations*—Touching or overlapping particles are seen as single particles; thus, this technique may overestimate the particle size if these conditions occur.

14. Laser Diffraction (Light Scattering)

14.1 *Principle of Operation*—Particles suspended in a liquid or gaseous medium are passed through a laser beam, the smaller particles scattering (diffracting) the laser light to larger off-axis angles than the larger particles. The angular distribution of the scattered light is measured using photodetectors and analyzed using Mie scattering theory (or Fraunhofer diffraction theory, a special case of Mie theory), relating the angular intensity distribution to the particle size.

14.2 *Particle Size Range of Application*—0.02 μm to 2 mm.

14.3 *Specific Requirements:*

14.3.1 A powder suspension that remains dispersed in flowing air or liquid is necessary. (See Section 7.)

14.3.2 The powder suspension must be very dilute, to avoid multiple scattering—light scattering from one particle, then re-scattering from other particles, before reaching the detector.

14.3.3 For transparent materials, knowledge of the refractive index is necessary.

14.4 *Limitations:*

14.4.1 Since light scattering analysis depends on the optical properties of the material analyzed, only homogenous materials of a single, known refractive index may be analyzed in this way if fundamental particle size is desired. However, light scattering analysis may be used to comparatively characterize mixtures of materials of differing optical properties, with caution as to the significance of the particle size information obtained.

14.4.2 Since a dilute suspension is necessary, concentrated slurries cannot be analyzed by the laser diffraction method.

15. Aerosol Electrical Mobility

15.1 *Principle of Operation*—A suspension containing either solid particles in a liquid, or a non-volatile component

dissolved into a liquid, is atomized into an aerosol. The liquid portion of the aerosol is evaporated leaving only solid particles. These particles are charge-neutralized and drawn into the electric mobility analyzer (or differential mobility analyzer (DMA)). The DMA subjects the aerosol stream to an electric field. Particles with the correct electric mobility will travel across the electric field and be selected out of the aerosol stream. The electric mobility is a function of the field strength, the aerodynamic diameter of the particle and the charge on the particle. The output of the DMA is a monodisperse aerosol stream that is generally counted with an absolute counter such as a condensation particle counter. The particle size can be determined by the voltage applied to the DMA and the associated aerosol flow rates.

15.2 *Particle Size Range of Application*—6 nm to 1.0 μm .

15.3 *Specific Requirements*—The particle suspension must be very dilute to avoid doublets and aggregates within one droplet.

15.4 *Limitations*—The accuracy of this method is dependent on the accuracy of the voltage and air flow metering.

16. Acoustic Attenuation

16.1 *Principle of Operation*—An ultrasonic wave is passed through particles suspended in a liquid, usually water. The ultrasonic wave is scattered and absorbed by the particulate phase such that its intensity is diminished (attenuated), the extent of attenuation being dependent on the ultrasonic frequency and physical properties of the particles. The attenuation spectrum is measured by ultrasonic transducers and analyzed using a theoretical or empirical model relating the attenuation spectrum to particle size.

16.2 *Particle Size Range of Application*—0.01 to 100 μm .

16.3 *Specific Requirements*:

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

16.3.2 The powder suspension must be relatively concentrated (0.1 to 50 % by volume).

16.3.3 Knowledge of the particle and liquid physical properties, such as density, sound speed, shear viscosity, thermal conductivity and expansion, and heat capacity, is necessary.

16.3.4 Since many of the physical properties mentioned above are temperature-dependent, a constant temperature must be maintained during analysis.

16.4 *Limitations*:

16.4.1 Since a relatively concentrated suspension is necessary, very dilute suspensions cannot be analyzed by the acoustic attenuation method.

16.4.2 Since acoustic attenuation analysis depends on the density and other physical properties of the material analyzed, only homogeneous materials of specific, known physical properties may be analyzed in this way; no mixtures of powders are possible.

17. Representation and Treatment of Data

17.1 Report particle size analysis data according to the guidelines in Practice E1617, at whatever level of reporting is appropriate and agreed upon.

17.2 Data may be reported in tabular form, as the entire particle size distribution, or in graphical form, as a cumulative distribution (percent less than or percent greater than) or a frequency distribution (histogram). Any combination of these may be reported.

17.3 Some particle size measurement instrumentation fits the collected data to certain reference models, such as normal, log-normal, or Rosin-Rammler distributions. The fitted reference model should also be reported.

17.4 Other size parameters calculated from the measured particle size distribution may also be reported (for example, mean, modal, or median diameter, 10 % size, 90 % size, etc.).

18. Keywords

18.1 acoustic attenuation; aerosol electrical mobility; centrifugal sedimentation; dispersion; electrical sensing zone; electrozone; image analysis; laser diffraction; light scattering; particle size; particle size analysis; particle size distribution; powder; sampling; sedimentation; sieving; static image analysis

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