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Standard Guide for
Development of Laser Diffraction Particle Size Analysis Methods for Powder Materials[1]

This standard is issued under the fixed designation E3340; 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 sets out the general approach to the particle size distribution measurement of powders, suspensions, or slurries using an appropriate wet or dry methodology by the laser diffraction technique. It is recommended for use in measurements of broad particle size distributions.

1.2 The guide provides guidelines to the parameters that should be specified and a generalized guideline to reasonable and acceptable tolerances for points in the volume-based distribution curve such as x_{10} (D_v10), x_{50} (D_v50), x_{90} (D_v90), and D[4, 3] (volume moment mean). It is noted that ISO prefers the term x for particle size as opposed to other usage of d or D (implying diameter).

1.3 This guide provides guidance on the verification of instrument performance in conjunction with the internal quality control (QC) audit functions of the instrument owner. Results should be reported in the format indicated by Practice E1617 and ISO 13320.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

E1617 Practice for Reporting Particle Size Characterization Data

E2490 Guide for Measurement of Particle Size Distribution of Nanomaterials in Suspension by Photon Correlation Spectroscopy (PCS)

2.2 *ISO Standards:*[3]

ISO 13320 Particle size analysis — Laser diffraction methods

ISO 14887 Sample preparation — Dispersing procedures for powders in liquids

ISO 14488/AMD 1:2019 Particulate materials — Sampling and sample splitting for the determination of particulate properties

3. Terminology

3.1 *Definitions:*

3.1.1 Some of the definitions in 3.2 will differ slightly from those used within other (non-particle sizing) standards. For further details, see the Terminology section of Guide E2490.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *differential pressure (ΔP), n*—the pressure difference across a venturi that causes acceleration (and thus shear forces) leading to dispersion (and possibly attrition) in a dry laser diffraction measurement.

3.2.2 *laser diffraction, n*—light scattering technique primarily used for particle size distribution analysis, applicable to the approximate range 0.1 – 3000 µm, where multiple angles are

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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.

[3] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

used to analyze the (laser light) scattering from an ensemble of particles in liquid or dry suspension. See ISO 13320.

3.2.3 *method development, n—in laser diffraction*, this refers to the actions involved in understanding how energy affects the apparent particle size distribution of the material in line with the measurement objectives (that is, bulk or dispersed size required).

3.2.4 *repeatability, n—in particle sizing techniques*, this usually refers to the precision of repeated consecutive measurements on the same group of particles and is normally expressed as a relative standard deviation (RSD) or coefficient of variation (C.V.).

3.2.4.1 *Discussion*—The repeatability value reflects the stability (instrumental, but mainly the sample) of the system over time. Changes in the sample could include dispersion and settling.

3.2.5 *reproducibility, n—in particle sizing*, this usually refers to second and further aliquots of the same bulk sample (and therefore is subject to the heterogeneity of the starting material and the sampling method employed).

3.2.5.1 *Discussion*—In a slurry system, it is often the largest error when repeated samples are taken. Other definitions of reproducibility also address the variability among single test results gathered from different laboratories when interlaboratory testing is undertaken. It is to be noted that the same group of particles can never be measured in such a system of tests and therefore reproducibility values are typically considerably in excess of repeatability values.

3.2.6 *robustness, n*—a measure of the change of the required parameter with deliberate and systematic variations in any or all of the key parameters that influence it.

3.2.6.1 *Discussion*—For example, dispersion time (ultrasound time and power setting) almost invariably will affect the reported results. Variation in pH is likely to affect the degree of agglomeration as will the addition (deliberate or accidental) of any ionic species and so forth. Changes to the input optical constants may also have an effect on the end result.

3.2.7 *standard operating procedure (SOP), n*—the formulation of a fixed measurement protocol once method development has taken place.

4. Summary of Guide

4.1 This guide provides a general overview of the methodologies involved with the development of wet and dry dispersion techniques for measuring the particle size distribution of powdered systems. Specific details are provided to the approach taken when a powder is either measured directly with dry dispersion or wetted in a liquid for wet dispersion.

5. Significance and Use

5.1 The technique of laser diffraction for particle size distribution analysis is extensively used in industry and academia both for on-line control and laboratory needs. Guidance is obviously useful in this regard.

5.2 This guide can be used to develop methods of particle size analysis where well-established analysis procedures do not already exist. See Guide B821 for similar guidance and useful procedures for wet dispersion of metal powders and related compounds.

6. Reagents

6.1 In general, no reagents specific to the laser diffraction technique are necessary. For dry measurement the dispersant 'fluid' is usually dry, particulate-matter-free air. However, in the wet methodology, dispersing, and stabilizing agents can be used for a specific test sample in order to preserve stability during the measurement. A suitable diluent is used to achieve a particle concentration appropriate for a laser diffraction measurement (typically 0.001 – 0.1 volume %). Particle size may undergo change on dilution, as the ionic environment within which the particles are dispersed, changes in nature or concentration. The apparent particle size may change in line with increased concentration (obscuration), especially with particles smaller than around 2 µm, due to multiple scattering.

7. Procedures

7.1 *Overview and Introduction*—The four basic questions to answer prior to performing a particle size distribution measurement are:

7.1.1 What is the acceptable quality level (AQL) of the organization? This sets the desired or required precision for a specification and has an impact on the minimum mass to meet this requirement.

7.1.2 What is the top end point of the distribution that requires specifying or control? This has impact on the minimum mass required.

7.1.3 Is a bulk size ("as is"/with agglomerates) required or is a dispersed (primary) size desired? In other words, "What is the purpose of taking the measurement?" The answers to these questions direct the routes to measurement and the level of energy input required to keep the material in bulk phase or to disperse it to primary particle size.

7.1.4 What is the polydispersity (width/spread) of the particle size distribution and the density of the material? Again, the answer to this question has direct impact on the minimum mass required to meet any proscribed specification or material variation.

7.1.5 To estimate the minimum standard error (fundamental sampling error, FSE) in an experiment due solely to the heterogeneity of the particle size distribution, the following fifth question requires answering:

7.1.5.1 What is the mass of sample that is utilized in the particle size experiment?

7.1.5.2 Note that any formulated specification needs to be 'just good enough' and 'fit for purpose'. The link between the particle size distribution and product performance (product performance indicators) or critical quality attributes to be well understood before any specification is developed.

7.2 The purpose of method development is to understand how the input of energy affects the apparent particle size distribution. The effect of changes in pH and chemistry can also be investigated at this stage. When the effect of these parameters is understood, a standard operating procedure (SOP) can be formulated in line with the objectives of the measurement – bulk or primary size. The input of energy disrupts agglomerated material and excessive energy input can cause comminution (milling) of a powder whether dispersed in a fluid or directly measured dry.

7.3 *Minimum Mass/Best Standard Error (FSE):*

7.3.1 It can be shown (see ISO 14488/AMD 1:2019 and references (**1**)[4] and (**2**)) that the minimum sample mass, M_s (in g), required for any confidence level (standard error, σ) at the x_{95} point (in cm) with (assumed) spherical particles of density ρ g/cm^3 is:

$$M_s = [18 \times (\pi / 6) \times (x_{95})^3 \times \rho] / \sigma^2 \tag{1}$$

7.3.2 Eq 1 gives a good guide as to the mass of the sample needed to meet any predefined specification or AQL and represents the minimum possible variation in the system based solely on the heterogeneity of the material. Other errors, for example, delimitation, segregation, and analytical will add to this FSE. The analytical error is typically 1 or 2 orders of magnitude less than the FSE at x_{95} sizes above 100 µm.

7.4 *Instrument Verification:*

7.4.1 Laser diffraction instruments use first principles measurement techniques and thus there is no concept of calibration in the formal sense where the instrument output would be adjusted to read the correct values (**3**). Verification of instrument performance is carried out using a reference material or reference materials ideally certified by an appropriate national body (for example, National Institute of Standards and Technology (NIST)) for the laser diffraction technique. A reference material should provide the values and ranges for the appropriate points in the distribution. Manufacturer's standard materials usually provide a lower cost option than NIST CRM materials. A standard material should be polydisperse (width of distribution (often defined as a 'span' such as x_{90}/x_{10}) > 10:1) to certify the size (x) axis and the quantity (y, Q_3, volume) axis. Failure to verify the performance of the instrument should be investigated in conjunction with the manufacturer. Usual failure causes include dirty optics, inadequate temperature equilibration especially of volatile fluids, poor mechanical alignment of the optics. Less common causes include damage to any of the optical elements such as scratches on lenses or (a) missing detector(s).

7.5 *Optical Constants:*

7.5.1 For materials smaller than around 25 µm (ISO 13320), the optical property constants exert an effect on the generated particle size distribution. The real part of the refractive index should always be known ideally by measurement if literature values do not exist. There are a number of standard routes for measurement or estimation of the real part of the refractive index and this must always be known prior to measurement. If a robustness study indicates that altering the imaginary/absorption part of the refractive index values, in a small, systematic, sensible manner (the 3S's), has a significant effect on the generated particle size distribution, more information should be sought. Ideally, the volume concentration experiment as described in ISO 13320 can be used for estimation of the imaginary component of the refractive index.

7.6 *Robustness Study:*

7.6.1 Robustness studies only examine the imaginary/absorptive part of this parameter as the real part is already known or assumed. For irregular materials, 0 imaginary is not possible and changes in apparent distribution are examined with orders of magnitude change (0.001/0.01/0.1/1.0) to the imaginary parameter.

7.6.2 Effect on the fine end of the distribution should be assessed. Shoulders at a harmonic of the laser wavelength (for example, for a He-Ne laser this could be 2 × 0.6328 ~ 1.2 µm) are normally indicative of poor optical property selection. The form (shape) of the plot and the fit/residual can aid selection of the 'correct' value. If vital, a volume concentration experiment as described in ISO 13320 can be utilized. More assistance can usually be provided by the instrument manufacturer and application note and webinar information is often available.

7.7 *Method Development:*

7.7.1 The basic approach in laser diffraction method development is to gauge the effect that energy input (and other important parameters such as chemistry/pH) has on the apparent particle size distribution to locate stable measurement

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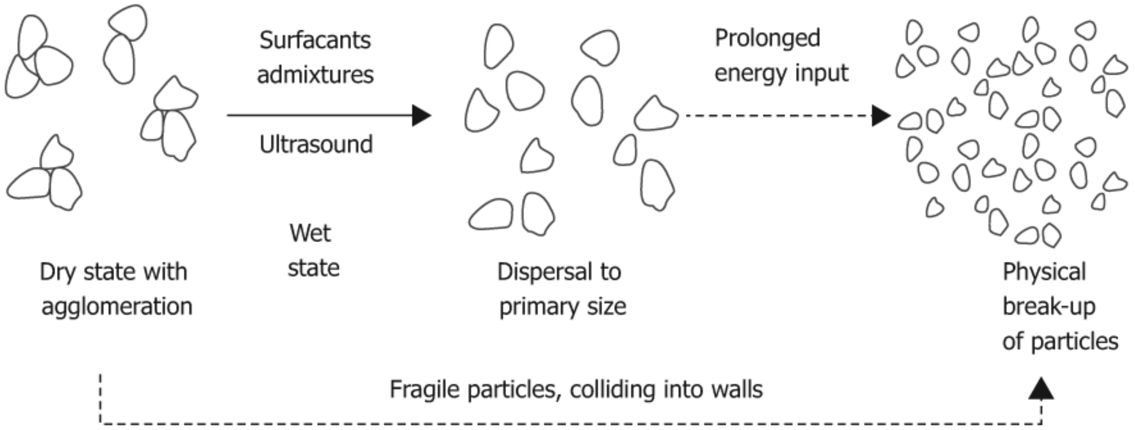
[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.



NOTE 1—The stages of agglomeration, dispersion, and attrition are noted.

FIG. 1 Generic Diagram Indicating the Effect of Increasing Energy on a Particulate System

conditions for any developed SOP (**4**) (see Fig. 1). In some circumstances the laser diffraction technique is rapid enough to examine kinetic effects (for example, dissolution).

7.7.2 In dry method development, the energy input is by means of varying the differential pressure across a venturi. In wet method development, energy input is by means of the pump-stirrer mechanism used to keep the material in suspension and the application of ultrasound energy. Note that in a variable balance that the factors that affect the precision parameters are:

7.7.2.1 Repeatability (applies to wet only as the same particles are recycled in front of the laser beam also called intra-assay precision):

(1) Instrument variability, and

(2) Any changes (desired or undesired) in the material during the measurement duration.

7.7.2.2 Reproducibility (applies to dry as a different group of particles is measured each time during consecutive measurements or to wet with different aliquots taken for measurement):

(1) Instrument variability,

(2) Any changes (desired or undesired) in the material during the measurement duration, and

(3) The inherent heterogeneity of the material in a polydisperse distribution or alternatively, the taking of a representative sample.

7.7.3 There are advantages and disadvantages in both the wet and dry systems.

7.7.3.1 The advantages of dry include:

(1) Much quicker than wet,

(2) No 'sample preparation',

(3) No (hazardous or expensive) solvents,

(4) No recycling or disposal of solvents,

(5) Can take larger amounts of sample so heterogeneity issues may be minimized,

(6) Simple method development, and

(7) More robustness to change in optical parameters as the relative refractive index (RRI) is always higher in a gas (RI ~ 1.000) than in a liquid (RI typically > 1.33).

7.7.3.2 The disadvantages of dry include:

(1) No plateau in the PST except for a monodisperse sample, so needs wet verification for fixing of the working pressure;

(2) The same particles are never remeasured, so the heterogeneity of the material causes variation and thus we are examining reproducibility only;

(3) Will not suspend large or dense material as well as wet (Archimedes effect); and

(4) Occasional occurrence of spurious peaks at large particle size due to refractive index variation in the airstream (can be caused by temperature variation/heating or evaporation of residual solvent from a partially dried powder).

7.7.3.3 Thus, dry dispersion is generally restricted to material > 1 µm– dry submicron dispersion is not possible due to the comminution limit.

7.7.3.4 The advantages of wet dispersion include:

(1) Energy input can be controlled and maximized, if necessary, for small material;

(2) The same particles are measured time and time again (repeatability) and thus sampling heterogeneity issues are not present for a single sample;

(3) The material maybe already in suspension or slurry form, so it makes no sense (and can be extremely dangerous) to dry a material for dry dispersion in laser diffraction; and

(4) An aliquot of the dispersed sample may be taken and examined by optical microscopy, so the state of dispersion can be directly observed. This is a very important advantage for wet dispersion over dry.

7.7.3.5 The disadvantages of wet dispersion include:

(1) Small amounts of sample may be measured so representative sampling is critical,

(2) Possible use of expensive, hazardous, or costly, to recycle solvents – water would be ideal,

(3) Slower than dry dispersion especially with any cleaning cycles, and

(4) Finding a suitable dispersant liquid can be difficult and time-consuming.

DRY METHOD DEVELOPMENT

7.8 This examines the apparent particle size distribution using the pressure-size titration (PST) technique, more correctly differential pressure-size titration (ΔPST). The technique relies on passing a mass of particles, carried in a dry gas (usually air), through a venturi (narrowing in a pipe or tube) such that the resultant acceleration induces shear forces plus collisions with walls which separate (or 'disperse') weak agglomerates of different sizes identical to the operation of an air-jet mill. These shear forces are controlled by the differential pressure (ΔP) across the venturi with higher ΔPs favoring more aggressive dispersion which could fracture or break fragile or friable material. Thus, in general, there is always a balance to be met between adequate dispersion and attrition, or milling/comminution, of the material. Indeed, the slope of the PST can be used to look at attrition of materials (for example, catalysts used in the fluidized catalytic cracking (FCC) process).

7.9 The dispersed particles pass once through the laser beam usually into a vacuum cleaner bag or cyclone. This one-pass technique never remeasures the same group of particles. Thus, the technique is subject to the inherent heterogeneity of the material. It is important to note that there is no plateau or flat portion (indicative of stability) in a PST for a polydisperse distribution even though excellent (sample-to-sample for consecutive measurements) reproducibility can be obtained. The setting of the required working pressure needs to be determined by means of a reference technique – this is often a wet measurement comparison as recommended by ISO 13320. If the results over-plot, the material must be in the same state of dispersion (almost certainly 'complete') under those measurement conditions. If over-plot of the wet and dry results cannot be achieved, the material is undergoing some change in either the dry or wet measurements. This could be attrition in the dry (this dry smaller than wet) or other effects such as swelling or chemical reaction in the wet. This discrepancy will require investigation. See Fig. 2 and Fig. 3.

7.10 The conditions that will need to be optimized in the dry measurement include:

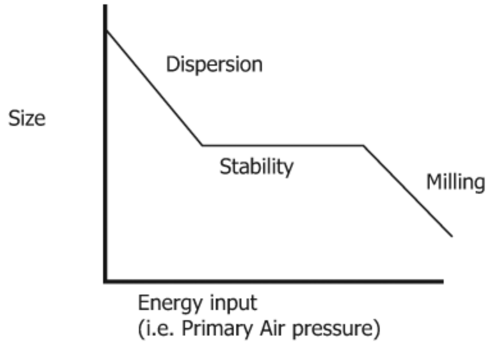


FIG. 2 Theoretical Plot of a Dry Powder Pressure-Size Titration (PST) in Laser Diffraction

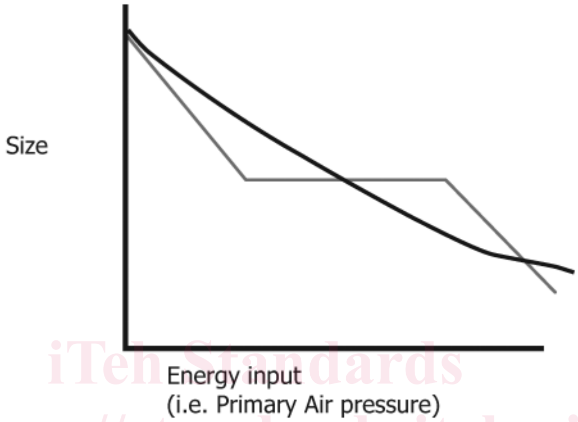


FIG. 3 Real and Practical Plot of a Dry Powder Pressure-Size Titration Plot in Laser Diffraction

7.10.1 The carrier fluid (usually air) needs to be moisture and oil-free (oil is often added to lubricate industrial compressed air). An adequate filter (silica gel for water removal, appropriate filter to remove all dust > 0.5 µm) prior to the dry powder accessory is mandatory.

7.10.2 Optical concentration (often termed obscuration = [1 – Transmission]) rises as particle concentration increases. Usually this is kept low (obscuration < 5 %; transmission > 95 %) to reduce chances of particle recombination after dispersion and to minimize possible multiple scattering. Optical concentration is a function of the particle size distribution (in general, small particles provide higher obscuration but weak scattering; large particles provide low obscuration but high scattering) as well as the experimental conditions. The two factors used to control the mass flow (the key parameter) through the system are the venturi size and the vibration rate of the feed hopper or tray. Changes in differential pressure will not markedly alter the flow rate or mass loading in the system.

7.10.3 Samples of smaller primary particle size will require more aggressive dispersion conditions (smaller venturi size; higher ΔP) generally (if primary size is the objective of the measurement). Delicate material or where agglomerated size is important will typically have less aggressive conditions. Indeed, 0 (zero) ΔP and only vacuum can be used to pass the material through the measurement zone. Gravity feed is possible for large (usually > 1 mm; 1000 µm) particulate matter where dispersion is not needed.

7.10.4 Practical difficulties such as the venturi blocking or ‘throttling’ caused by too high a mass flow and the vibration or feed rate will need to be reduced to an appropriate value for powder passage. Materials that are damp or exhibit poor flowability give rise to changes in the optical concentration and this may be difficult to control. Flow additives (for example, fumed silica) outside the size distribution of the examined material are sometimes utilized but this includes an added complexity to the usually simple measurement. In certain conditions, remembering that this is a dry powder method, wet dispersive techniques will need to be used for successful measurements.

7.10.5 In general, if the dry technique is applicable, it is remarkably simple and quick with no liquid to use or recycle and little or no clean up required. Usually a fine abrasive material (for example, < 325# carborundum) can be used to clean the venturi and any discolored tubing replaced (noting that it is only the high concentration of particles carried in the gas that is actually measured). Certain materials such as powder coatings can fuse/melt under dispersion conditions (friction conditions similar to blowing up a bicycle tire) and regular clean-up will be needed. In the worst scenario, the venturi can be removed and cleaned with an appropriate liquid (for example, methylethylketone. MEK).

7.10.6 It is best to collect data as short consecutive measurements (for example, 10 × 1 second) which can be combined easily at a later stage. This is always preferable to one