INTERNATIONAL STANDARD

ISO 13320

Second edition
2020-01

Particle size analysis — Laser diffraction methods

Analyse granulométrique — Méthodes par diffraction laser

Reference number
ISO 13320:2020(E)

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Published in Switzerland

Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

This second edition cancels and replaces the first edition (ISO 13320:2009), which has been technically revised. The main changes compared to the previous edition are as follows:

a) protocols for evaluation of accuracy and qualification of instrument were newly developed;

b) new Annex H (normative) for usage of reference material has been added;

c) new descriptions for wider applications, such as off-line, online, in-line and at-line have been added;

d) some informative parts have been moved to new annexes;

e) minor revisions and updates have been made throughout the document.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The laser diffraction technique has evolved such that it is now a dominant method for determination of particle size distributions (PSDs). The success of the technique is based on the fact that it can be applied to a wide variety of particulate systems. The technique is fast and can be automated, and a variety of commercial instruments is available. Nevertheless, the proper use of the instrument and the interpretation of the results require the necessary caution.

Since ISO 13320-1:1999 was first published, the understanding of light scattering by different materials and the design of instruments have advanced considerably. This is especially marked in the ability to measure very fine particles. Therefore, it was replaced with the first edition of ISO 13320 in 2009, and since then the method has been developed for a wider application. Additionally, demands raised recently not only on establishment of accuracy of measurements but also on necessity of evaluation of the accuracy and of qualification of instrument by users. Therefore, this document incorporates the most recent advances in understanding.

Particle size analysis — Laser diffraction methods

1 Scope

This document provides guidance on instrument qualification and size distribution measurement of particles in many two-phase systems (e.g. powders, sprays, aerosols, suspensions, emulsions and gas bubbles in liquids) through the analysis of their light-scattering properties. It does not address the specific requirements of particle size measurement of specific materials.

This document is applicable to particle sizes ranging from approximately 0,1 µm to 3 mm. With special instrumentation and conditions, the applicable size range can be extended above 3 mm and below 0,1 µm.

For spherical and non-spherical particles, a size distribution is reported, where the predicted scattering pattern for the volumetric sum of spherical particles matches the measured scattering pattern. This is because the technique assumes a spherical particle shape in its optical model. For non-spherical particles the resulting particle size distribution is different from that obtained by methods based on other physical principles (e.g. sedimentation, sieving).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9276-1, *Representation of results of particle size analysis — Part 1: Graphical representation*

ISO 9276-2, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1.1
absorption
reduction of intensity of a light beam not due to scattering

3.1.2
accuracy
closeness of agreement between a test result or measurement result and the true value

Note 1 to entry: In practice, the accepted reference value is substituted for the true value.

Note 2 to entry: The term “accuracy”, when applied to a set of test or measurement results, involves a combination of random components and a common systematic error or bias component.

Note 3 to entry: Accuracy refers to a combination of trueness and precision.

[SOURCE: ISO 3534-2:2006, 3.3.1]

3.1.3
aspect ratio
ratio of the minimum to the maximum Feret diameter

Note 1 to entry: For not very elongated particles.

[SOURCE: ISO 26824:2013, 4.5]

3.1.4
certified reference material
CRM
reference material (3.1.16) characterised by a metrologically valid procedure for one or more specified properties, accompanied by an RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

Note 1 to entry: The concept of value includes a nominal property or a qualitative attribute such as identity or sequence. Uncertainties for such attributes may be expressed as probabilities or levels of confidence.

Note 2 to entry: Metrologically valid procedures for the production and certification of RMs are given in, among others, ISO 17034 and ISO Guide 35.

Note 3 to entry: ISO Guide 31 gives guidance on the contents of RM certificates.

Note 4 to entry: ISO/IEC Guide 99:2007, 5.14 has an analogous definition.

[SOURCE: ISO Guide 35:2017, 3.2]

3.1.5
complex refractive index
$\underline{n}_p$

refractive index of a particle, consisting of a real and an imaginary (absorption) part

Note 1 to entry: The complex refractive index of a particle can be expressed mathematically as

$$\underline{n}_p = n_p - ik_p$$

where

- i is the square root of –1;
- k_p is the positive imaginary (absorption) part of the refractive index of a particle;
- n_p is the positive real part of the refractive index of a particle.

Note 2 to entry: In contrast to ISO 80000-7, this document follows the convention of adding a minus sign to the imaginary part of the refractive index.

3.1.6
deconvolution
<laser diffraction> mathematical procedure whereby the size distribution of an ensemble of particles is inferred from measurements of their scattering pattern

3.1.7
diffraction
<particle size analysis> scattering of light around the contour of a particle, observed at a substantial distance (in the ‘far field’)

3.1.8
equivalent spherical diameter
<laser diffraction> particle size reported from a distribution of spherical particles that creates a scattering pattern that matches the light scattering distribution observed from the measurement

Note 1 to entry: The scattering pattern of the spherical particles is calculated according to an optical model.

3.1.9
extinction
<particle size analysis> attenuation of a light beam traversing a medium through absorption and scattering

3.1.10
intermediate precision
<laser diffraction> accuracy and precision under *intermediate precision conditions* (3.1.11)

[SOURCE: ISO 3534-2:2006, 3.3.15, modified — field of application <laser diffraction> has been added.]

3.1.11
intermediate precision conditions
<laser diffraction> conditions where test results or measurement results are obtained on different laser diffraction instruments and with different operators using the same prescribed method

Note 1 to entry: There are four elements to the operating condition: time, calibration, operator and equipment.

3.1.12
multiple scattering
consecutive scattering of light by more than one particle, causing a scattering pattern that is no longer the sum of the patterns from all individual particles

3.1.13
obscuration
fraction of incident light that is attenuated due to extinction (scattering and/or absorption) by particles

Note 1 to entry: Obscuration can be expressed as a percentage.

Note 2 to entry: When expressed as fractions, obscuration plus *transmission* (3.1.29) equal unity.

[SOURCE: ISO 8130-13:2019, 3.1, modified — words “percentage” and “during a laser diffraction measurement” have been omitted because of context.]

3.1.14
optical model
theoretical model used for computing the model matrix for optically homogeneous and isotropic spheres with, if necessary, a specified complex refractive index

EXAMPLE Fraunhofer diffraction model, Mie scattering model.

3.1.15
precision
closeness of agreement between independent test/measurement results obtained under stipulated conditions

Note 1 to entry: Precision depends only on the distribution of random errors and does not relate to the true value or the specified value.

Note 2 to entry: The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results or measurement results. Less precision is reflected by a larger standard deviation.

Note 3 to entry: Quantitative measures of precision depend critically on the stipulated conditions. Repeatability conditions and reproducibility conditions are particular sets of extreme stipulated conditions.

[SOURCE: ISO 3534-2:2006, 3.3.4]

3.1.16
reference material
RM
material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

Note 1 to entry: RM is a generic term.

Note 2 to entry: Properties can be quantitative or qualitative, e.g. identity of substances or species.

Note 3 to entry: Uses may include the calibration of a measurement system, assessment of a measurement procedure, assigning values to other materials, and quality control.

Note 4 to entry: ISO/IEC Guide 99:2007 has an analogous definition but restricts the term "measurement" to apply to quantitative values. However, ISO/IEC Guide 99:2007, 5.13, Note 3 (VIM), specifically includes qualitative properties, called "nominal properties".

[SOURCE: ISO Guide 35:2017, 3.1]

3.1.17
reflection
<particle size analysis> change of direction of a light wave at a surface without a change in wavelength or frequency

3.1.18
refraction
process by which the direction of a radiation is changed as a result of changes in its velocity of propagation in passing through an optically non-homogeneous medium, or in crossing a surface separating different media

Note 1 to entry: The process occurs in accordance with Snell's law:

$$n_m \sin\theta_m = n_p \sin\theta_p$$

See 3.2 for symbol definitions.

3.1.19
relative refractive index
m_{rel}
ratio of the complex refractive index of a particle to the real part of the dispersion medium

[SOURCE: ISO 24235:2007, 3.3, modified — "absolute refractive index" has been replaced by "complex refractive index" and "the sample" has been replaced by "a particle".]

Note 1 to entry: In many applications, the medium is transparent and, thus, its refractive index has a negligible imaginary part.

Note 2 to entry: The relative refractive index can be expressed mathematically as

$$m_{rel} = \underline{n}_p / n_m$$

where

n_m is the real part of the refractive index of the medium;
$\underline{n}_p$ is the complex refractive index of a particle.

See *single scattering* (3.1.26).

3.1.20
repeatability
precision under *repeatability conditions* (3.1.21)

Note 1 to entry: Repeatability can be expressed quantitatively in terms of the dispersion characteristics of the results.

[SOURCE: ISO 3534-2:2006, 3.3.5]

3.1.21
repeatability conditions
observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in the same test or measuring facility by the same operator using the same equipment within short intervals of time

Note 1 to entry: Repeatability conditions include:

- the same measurement procedure or test procedure;
- the same operator;
- the same measuring or test equipment used under
  - the same conditions;
  - the same location;
- repetition over a short period of time.

[SOURCE: ISO 3534-2:2006, 3.3.6]

3.1.22
method repeatability
closeness of agreement between multiple measurement results of a given property in different aliquots of a sample, executed by the same operator using the same instrument under identical conditions within a short period of time

Note 1 to entry: The variability includes the variabilities of sub sampling technique, of the sampled material together and of the instrument.

3.1.23
scattering
change in propagation of light at the interface of two media having different optical properties

3.1.24
scattering angle
angle between the principal axis of the incident light beam and the scattered light

3.1.25
scattering pattern
angular pattern of light intensity, $I(\theta)$, or spatial pattern of light intensity, $I(r)$, originating from scattering, or the related energy values taking into account the sensitivity and the geometry of the detector elements

3.1.26
single scattering
scattering whereby the contribution of a single member of a particle population to the total scattering pattern remains independent of the other members of the population

3.1.27
single shot
<sample> for an analysis, for which the entire content of a test sample container is used

3.1.28
test sample
sample that is entirely used for a property characterization

[SOURCE: ISO 14488:2007, 3.12]

3.1.29
transmission
<particle size analysis> fraction of incident light that remains un-attenuated by the particles

Note 1 to entry: Transmission can be expressed as a percentage.

Note 2 to entry: When expressed as fractions, *obscuration* (3.1.13) plus transmission equal unity.

3.1.30
true value
quantity or quantitative characteristic supposed to be "true" as the target value of the measurement according to the definition of the measurement

Note 1 to entry: The true value is a theoretical concept and, in general, cannot be known exactly.

Note 2 to entry: For an explanation of the term "quantity", refer to ISO 3534-2:2006.

3.1.31
trueness
closeness of agreement between the expectation of a test result or a measurement result and a true value

Note 1 to entry: The measure of trueness is usually expressed in terms of bias

Note 2 to entry: Trueness is sometimes referred to as "accuracy of the mean". This usage is not recommended.

Note 3 to entry: In practice, the accepted reference value is substituted for the true value.

[SOURCE: ISO 3534-2:2006, 3.3.3]

3.2 Symbols

A_i	extinction efficiency of size class i
C	particulate concentration, volume fraction
CF	coverage factor
D	particle diameter (x may also be used)
$D_{10,3}$	particle diameter corresponding to the 10th percentile of the cumulative undersize distribution (here by volume)
$D_{50,3}$	median particle diameter corresponding to the 50th percentile of the cumulative undersize distribution (here by volume)
$D_{90,3}$	particle diameter corresponding to the 90th percentile of the cumulative undersize distribution (here by volume)
$I(\theta)$	angular intensity distribution of light scattered by particles (scattering pattern)
I_h	intensity of horizontally polarized light at a given angle
$I(r)$	spatial intensity distribution of light scattered by particles on the detector elements (measured scattering pattern by detector)

I_v	intensity of vertically polarized light at a given angle
J_1	first order Bessel Function of the first kind
k	wave number in medium: $2\pi n_m/\lambda$
ik_p	imaginary (absorption) part of the refractive index of a particle
l_a	distance from scattering object to detector
l_b	illuminated path length containing particles
$\boldsymbol{L}_n$	vector of photocurrents (i_1, i_2, ... i_n)
m_{rel}	relative, complex refractive index of particle to medium
$\boldsymbol{M}$	model matrix, containing calculated detector signals per unit volume of particles in all size classes
n_m	real part of refractive index of medium
n_p	real part of refractive index of particle
$\underline{n}_p$	complex refractive index of particle
O	obscuration (1 – transmission);
r	radial distance from focal point in focal plane
u_p	standard uncertainty for the parameter and value specified
u_{crm}	standard uncertainty of the certified value
u_{house}	standard uncertainty of in-house reference material value
U_{crm}	expanded uncertainty of the certified value
U_{house}	expanded uncertainty of in-house reference material value
U_{lim}	expanded tolerance limit defined by calculation
V_i	volume content of size class i
v	velocity of particles in dry disperser
x	particle diameter (D may also be used)
x_i	geometric mean particle size of size class i
$x_{10,3}$	particle diameter corresponding to 10th percentile of the cumulative undersize distribution (here by volume)
$x_{50,3}$	median particle diameter corresponding to the 50th percentile of the cumulative undersize distribution (here by volume)
$x_{90,3}$	particle diameter corresponding to 90th percentile of the cumulative undersize distribution (here by volume)
$\bar{x}_{1,3}$	volume-weighted mean diameter
α	dimensionless size parameter: $\pi x n_m/\lambda$

$\Delta Q_{3,i}$	volume fraction within size class i
θ	scattering angle with respect to forward direction
θ_m	angle with respect to perpendicular at boundary for a light beam in medium (see definition 3.1.18)
θ_p	angle with respect to perpendicular at boundary for a light beam in particle (see definition 3.1.18)
λ	wavelength of illuminating light source in vacuum
σ	standard deviation
ω	angular velocity

4 Principle

4.1 General

The laser diffraction or scattering technique[1] for the determination of particle size distributions, PSDs, is based upon the phenomenon that the angular distribution of the intensity of scattered light by a particle (scattering pattern) is dependent on the particle size. When the scattering is from a cloud or ensemble of particles the intensity of scattering for any given size class is related to the number of particles and their optical properties, present in that size class[5][20].

A test sample, dispersed at an adequate concentration in a suitable liquid or gas, is passed through the beam of a monochromatic light source, usually a laser. The light scattered by the particles, at various angles, is measured by an array of photo detectors. The numerical values from each detector are recorded for subsequent analysis. Within certain limits, such as of particle concentration in measuring zone, the scattering pattern of an ensemble of particles is identical to the sum of the individual scattering patterns of all particles. The theoretical scattering patterns of unit volumes of particles in selected size classes are used to build a matrix and together with a mathematical procedure are used to solve the inverse problem, providing a volumetric particle size distribution (PSD), iterated to provide a best fit to the measured scattering pattern[18].

4.2 Theory

The theoretical scattering pattern of a single spherical homogeneous particle is given by Mie-theory in general[4]. If the particle size is relatively large (in terms of size parameter, $\alpha = \pi \times n_m/\lambda > 10$) and is opaque, Fraunhofer diffraction theory is available only for small angle forward scattering[4][5]. The Fraunhofer approximation is an analytical method that does not require the optical properties of the material.

Some other theoretical approximations are available for numerical realization of the Mie-theory, and these are called optical models in general. Choosing a relevant optical model for the inverse problem to yield a proper PSD is important.

Laser diffraction records the scattering pattern from the particles presented. This composite pattern is converted to a size distribution of spherical particles that would provide the same composite scattering pattern using an appropriate optical model and data inversion routine. It therefore provides a size distribution of laser diffraction equivalent spheres. If the test sample is not spherical, the same basic procedure is used and the resulting size distribution is formed. Thus, PSD's for non-spherical particles

1) Early instruments had very limited computer capacity and were restricted to using a laser with Fraunhofer Diffraction. Often a model form of particle size distribution was iterated to fit the scattering data. The term Laser Diffraction rapidly became the dominant descriptor. This has continued despite the technique having advanced to use different light sources and more sophisticated optical theories and data analysis.

are likely to be different from other particle sizing techniques measuring the same material. The details of the theory are given in Annex A.

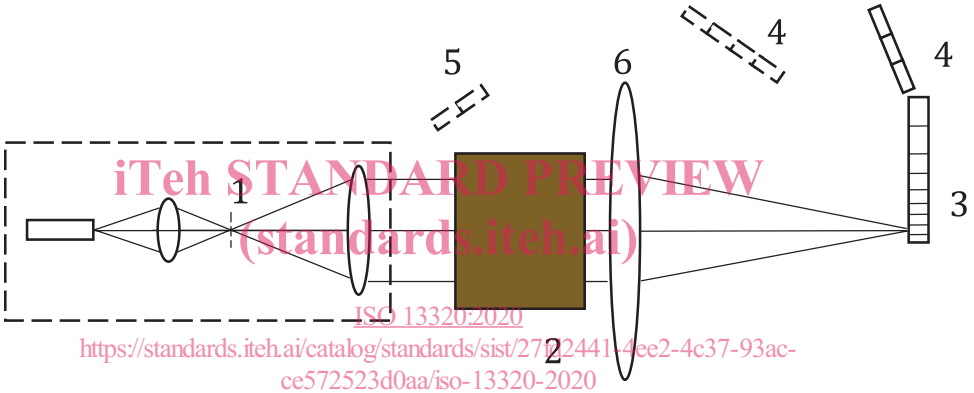
4.3 Typical instrument and optical arrangement

The system consists of a monochromatic light source, sample feeder, optical system, light detectors, and control-calculation device. To extend the applicable range of particle size and its analysis, multiple light sources, additional light detecting systems and related optical systems can be used.

The light source is typically a laser or other narrow-wavelength source to generate a monochromatic beam. This is followed by a beam-processing unit producing an extended and nearly ideal, Gaussian distributed beam to illuminate the dispersed particles. The illuminating light beam passes through the measuring zone of the optical system.

A computer is used to control the measurement, to store and to process the data, and to solve the inversion problem from the data of the detected signals to the particle size distribution. It may provide automated instrument operation.

Typical diagrams of the set-up of laser diffraction/scattering instruments are given in Figures 1 to 4.



Key

1 light source assembly [with one or more light source(s)] including beam expansion and collimation
2 measurement zone (for details, see Figure 2)
3 forward scattering multi-element detector (with obscuration/transmission detector)
4 wide angle scattering detector(s)
5 back scattering detector
6 Fourier lens

Figure 1 — Fourier optical arrangement