INTERNATIONAL STANDARD

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Particle size analysis — Laser diffraction methods

Analyse granulométrique — Méthodes par diffraction laser

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13320 was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

This first edition of ISO 13320 cancels and replaces ISO 13320-1:1999.

This corrected version of ISO 13320:2009 incorporates the following correction:

— in Figure A.2, lower graph, the symbols for datapoints corresponding to “1,39 – 0,0i” and “2,19 – 0,0i” have been changed to match the plots to which they refer.

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 various kinds of particulate systems, is fast and can be automated, and that 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 the publication of ISO 13320-1:1999, 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, this International Standard has been prepared to incorporate the most recent advances in understanding.

Particle size analysis — Laser diffraction methods

1 Scope

This International Standard 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 International Standard 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 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. 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 referenced documents are indispensable for the application 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*

ISO 9276-4, *Representation of results of particle size analysis — Part 4: Characterization of a classification process*

ISO 14488, *Particulate materials — Sampling and sample splitting for the determination of particulate properties*

ISO 14887, *Sample preparation — Dispersing procedures for powders in liquids*

3 Terms, definitions and symbols

3.1 Terms and definitions

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

3.1.2
coefficient of variation
CV
relative standard deviation (deprecated)
⟨positive random variable⟩ standard deviation divided by the mean

NOTE 1 The coefficient of variation is commonly reported as a percentage.

NOTE 2 Adapted from ISO 3534-1:2006[24], 2.38.

3.1.3
complex refractive index
$\underline{n}_p$
refractive index of a particle, consisting of a real and an imaginary (absorption) part

NOTE 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.

In contrast to ISO 80000-7:2008[27], item 7-5, this International Standard follows the convention of adding a minus sign to the imaginary part of the refractive index.

3.1.4
relative refractive index
m_{rel}
ratio of the complex refractive index of a particle to the real part of the dispersion medium

NOTE 1 Adapted from ISO 24235:2007[26].

NOTE 2 In most applications, the medium is transparent and, thus, its refractive index has a negligible imaginary part.

NOTE 3 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.

3.1.5
deconvolution
⟨particle size analysis⟩ mathematical procedure whereby the size distribution of an ensemble of particles is inferred from measurements of their scattering pattern

3.1.6
diffraction
⟨particle size analysis⟩ scattering of light around the contour of a particle, observed at a substantial distance (in the 'far field')

3.1.7
extinction
⟨particle size analysis⟩ attenuation of a light beam traversing a medium through absorption and scattering

3.1.8
model matrix
matrix containing vectors of the scattered light signals for unit volumes of different size classes, scaled to the detector's geometry, as derived from model computation

3.1.9
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

NOTE See **single scattering** (3.1.20).

3.1.10
obscuration
optical concentration
fraction of incident light that is attenuated due to extinction (scattering and/or absorption) by particles

NOTE 1 Adapted from ISO 8130-14:2004[25], 2.21.

NOTE 2 Obscuration can be expressed as a percentage.

NOTE 3 When expressed as fractions, obscuration plus **transmission** (3.1.22) equal unity.

3.1.11
optical model
theoretical model used for computing the model matrix for optically homogeneous and isotropic spheres with, if necessary, a specified complex refractive index

EXAMPLES Fraunhofer diffraction model, Mie scattering model.

3.1.12
reflection
⟨particle size analysis⟩ change of direction of a light wave at a surface without a change in wavelength or frequency

3.1.13
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

[IEC 60050-845:1987[28]]

NOTE 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.

3.1.14
repeatability (instrument)
⟨particle size analysis⟩ closeness of agreement between multiple measurement results of a given property in the same dispersed sample aliquot, executed by the same operator in the same instrument under identical conditions within a short period of time

NOTE This type of repeatability does not include variability due to sampling and dispersion.

3.1.15
repeatability (method)
⟨particle size analysis⟩ closeness of agreement between multiple measurement results of a given property in different aliquots of a sample, executed by the same operator in the same instrument under identical conditions within a short period of time

NOTE This type of repeatability includes variability due to sampling and dispersion.

3.1.16
reproducibility (method)
⟨particle size analysis⟩ closeness of agreement between multiple measurement results of a given property in different aliquots of a sample, prepared and executed by different operators in similar instruments according to the same method

3.1.17
scattering
⟨particle size analysis⟩ change in propagation of light at the interface of two media having different optical properties

3.1.18
scattering angle
⟨particle size analysis⟩ angle between the principal axis of the incident light beam and the scattered light

3.1.19
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

3.1.20
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

3.1.21
single shot analysis
analysis, for which the entire content of a sample container is used

3.1.22
transmission
⟨particle size analysis⟩ fraction of incident light that remains unattenuated by the particles

NOTE 1 Transmission can be expressed as a percentage.

NOTE 2 When expressed as fractions, **obscuration** (3.1.10) plus transmission equal unity.

3.1.23
width of size distribution
the width of the particle size distribution (PSD), expressed as the x_{90}/x_{10} ratio

NOTE For normal (Gaussian) size distributions, often the standard deviation (absolute value), σ, or the coefficient of variation (CV) is used. Then, about 95 % of the population of particles falls within $\pm 2\sigma$ from the mean value and about 99,7 % within $\pm 3\sigma$ from the mean value. The difference $x_{90} - x_{10}$ corresponds to $2{,}6\sigma$.

3.2 Symbols

A_i	extinction efficiency of size class i
C	particulate concentration, volume fraction
CV	coefficient of variation
f	focal length of lens
i	square root of –1
i_n	photocurrent of detector element, n
$I(\theta)$	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_i	first order Bessel Function
k	wavenumber in medium: $2\pi n_\mathrm{m}/\lambda$
k_p	imaginary (absorption) part of the refractive index of a particle
l_a	distance from scattering object to detector
l_b	illuminated pathlength 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
$\underline{n}_\mathrm{p}$	complex refractive index of particle
O	obscuration (1 – transmission); only true for single scattering
r	radial distance from focal point in focal plane
$\boldsymbol{V}$	vector of volume content in size classes (V_1, V_2 ... V_i)
V_i	volume content of size class i
v	velocity of particles in dry disperser
x	particle diameter
x_i	geometric mean particle size of size class i
x_{50}	median particle diameter; here used on a volumetric basis, i.e. 50 % by volume of the particles are smaller than this diameter and 50 % are larger
x_{10}	particle diameter corresponding to 10 % of the cumulative undersize distribution (here by volume)
x_{90}	particle diameter corresponding to 90 % of the cumulative undersize distribution (here by volume)
α	dimensionless size parameter: $\pi x n_\mathrm{m}/\lambda$
$\Delta Q_{3,i}$	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 (as used in Snell's law; see 3.1.13, Note)

θ_p angle with respect to perpendicular at boundary for a light beam in particle (as used in Snell's law; see 3.1.13, Note)

λ wavelength of illuminating light source in vacuum

σ standard deviation

ω angular frequency

4 Principle

A 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 multi-element detectors, and numerical values relating to the scattering pattern are recorded for subsequent analysis. These numerical scattering values are then transformed, using an appropriate optical model and mathematical procedure, to yield the proportion of the total volume of particles to a discrete number of size classes forming a volumetric particle size distribution (PSD).

The laser diffraction technique for the determination of PSDs is based on the phenomenon that particles scatter light in all directions with an intensity pattern that is dependent on particle size. Figure 1 illustrates this dependency in the scattering patterns for two sizes of spherical particles. In addition to particle size, particle shape and the optical properties of the particulate material influence the scattering pattern.

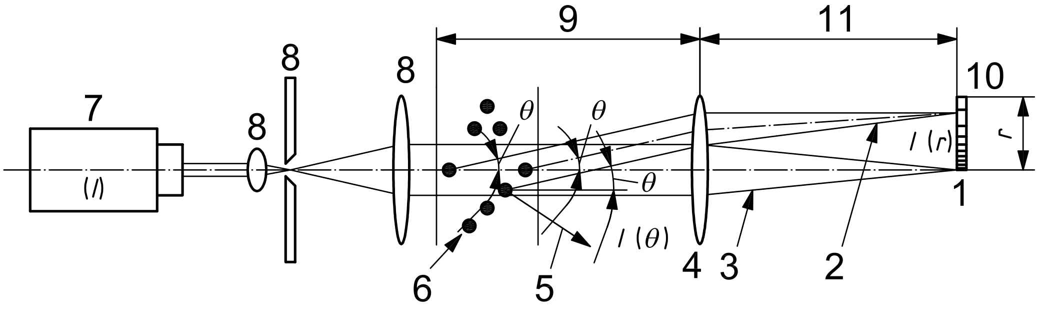
a) b)

Figure 1 — Scattering pattern for two spherical particles: the particle generating pattern a) is twice as large as the one generating pattern b) (simulated images for clarity)

5 Laser diffraction instrument

A set-up for a laser diffraction instrument is given in Figure 2.

In this Fourier set-up, a light source (typically a laser or other narrow-wavelength source) is used to generate a monochromatic, coherent, parallel beam. This is followed by a beam processing unit, usually a beam expander with integrated filter, producing an extended and nearly ideal beam to illuminate the dispersed particles.



Key

1 obscuration/optical concentration detector
2 scattered beam
3 direct beam
4 fourier lens
5 scattered light not collected by lens 4
6 ensemble of dispersed particles
7 light source (e.g. laser)
8 beam processing unit
9 working distance of lens 4
10 multi-element detector
11 focal distance of lens 4

NOTE For explanations of symbols, see 3.2.

Figure 2 — Fourier set-up of a laser diffraction instrument

A sample of particles, dispersed at an adequate concentration, is passed through the light beam in a measuring zone by a transporting medium (gas or liquid). This measuring zone should be within the working distance of the lens used. Sometimes, the particle stream in a process passes directly through the laser beam for measurement. This is the case in the measurement of sprays and aerosols. In other cases (e.g. when measuring emulsions, pastes and powders), samples can be dispersed in fluids and caused to flow through the measurement zone. Often dispersants (wetting agents; stabilizers) and/or mechanical forces (agitation; sonication) are applied for deagglomeration of particles and for stabilization of the dispersion. For these liquid dispersions, a recirculation system is most commonly used, consisting of an optical measuring cell, a dispersion bath usually equipped with stirrer and ultrasonic elements, a pump and tubing.

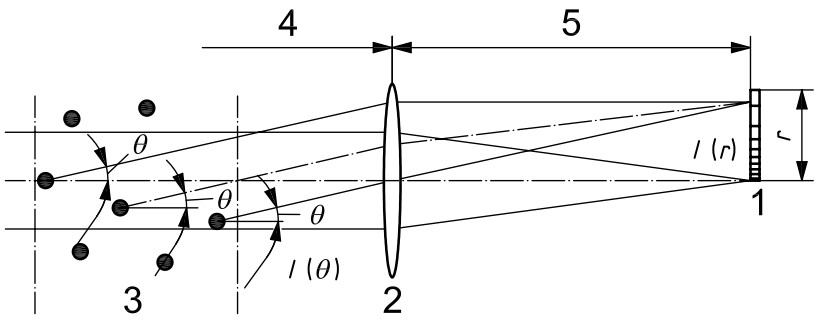
Dry powders can also be converted into aerosols through application of dry powder dispersers, which apply mechanical forces for deagglomeration. Here, a dosing device feeds the disperser with, ideally, a near-constant mass flow of sample. The disperser uses the energy of a compressed gas or the differential pressure to a vacuum to disperse the particles. It outputs an aerosol that is blown through the measuring zone, usually into the inlet of a vacuum pipe that collects the particles. Coarse, non-agglomerated powders can be transported through the measurement zone by gravity.

There are two positions in which the particles can enter the laser beam. In the Fourier optics case, the particles enter the parallel beam before and within the working distance of the collecting lens [see Figure 3a)]. This allows for the measurement of spatially extended particle systems. In the reverse Fourier optics case, the particles enter behind the lens and, thus, in a converging beam [see Figure 3b)].

The advantage of the Fourier set-up is that a reasonable pathlength for the sample is allowed within the working distance of the lens. The reverse Fourier set-up demands small pathlengths but provides one solution that enables the measurement of scattered light at larger angles.

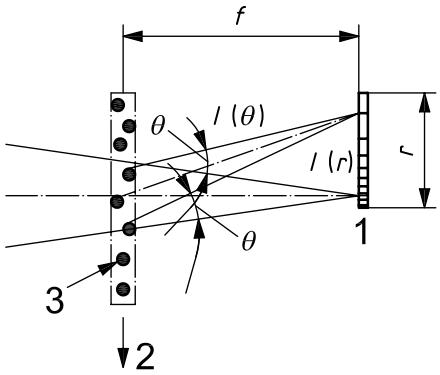
The interaction of the incident light beam and the ensemble of dispersed particles results in a scattering pattern with different light intensities scattered at various angles (see Annex A for the theoretical background of laser diffraction). The total angular intensity distribution $I(\theta)$, consisting of both direct and scattered light, is then focused by a positive lens or an ensemble of lenses onto a multi-element detector. The lens(es)

provide(s) for a scattering pattern which, within limits, is not dependent upon the location of the particles in the light beam. The continuous angular intensity distribution $I(\theta)$ is converted into a discrete spatial intensity distribution $I(r)$ on a set of detector elements.



Key

1 detector
2 fourier lens
3 ensemble of dispersed particles
4 working distance
5 focal distance



Key

1 detector
2 flow through cuvette for dispersed particles
3 particle

NOTE For explanations of symbols, see 3.2.

a) Fourier set-up: particles are in parallel beam before and within working distance of lens

b) Reverse Fourier set-up: particles are in converging beam between lens and detector

Figure 3 — Illustrations of optical arrangements used in laser diffraction instruments

Some instruments contain extra features to improve particle size analysis:

a) an extra light source at the same optical axis having a different wavelength;

b) one or more off-axis light sources, either at less or at more than 90° with respect to the optical axis;

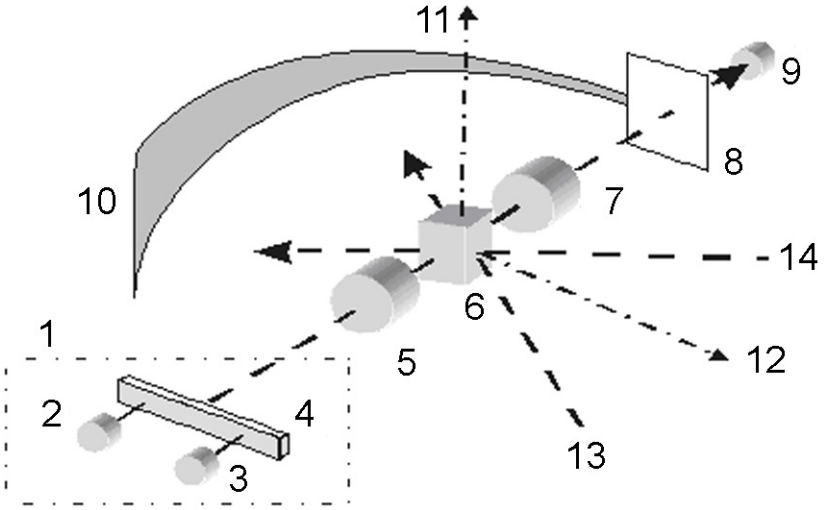
c) polarization filters for light source and detectors;

d) scattered light detectors at angles smaller than 90° but larger than the conventional angular range (forward scattering);

e) scattered light detectors at around 90° for measurement of intensities in different polarization directions;

f) scattered light detectors at angles larger than 90° (backscattering).

These possibilities are illustrated in Figure 4.



Key

1	light source assembly including beam expansion and/or collimation	8	low angle detector(s), either bespoke design or pixel array
2	light source wavelength 1	9	transmission or obscuration detector
3	light source wavelength 2	10	high angle detector array
4	beam switching arrangement	11	horizontally polarized light detector
5	reverse Fourier lens(es) position	12	vertically polarized light detector
6	measurement cell or general measurement zone	13	alternative entry point for light source
7	Fourier lens(es) position	14	alternative entry point for light source

Figure 4 — Possibilities for optical arrangements in laser diffraction instrument

It is assumed that the recorded scattering pattern of the particle ensemble is identical to the sum of the patterns from all individual particles (single scattering). Furthermore, the scattering pattern is assumed to come from spherical particles.

Detection of the scattering pattern is done by a number of silicon detectors or photodiodes and/or a pixel array detector. These detectors convert the spatial intensity distribution $I(r)$ into a series of photocurrents, i_n. Subsequent electronics then convert and digitize the photocurrents into a set of energies, L_n, representing the scattering pattern. A central element measures the intensity of the scattered and non-scattered light and, thus, with a calculation, provides a measure of optical concentration or obscuration. Some instruments provide special geometries of the central element in order to automatically re-centre or re-focus the detector by moving the detector or the lens. It is desirable that the detector elements are positioned so as to prevent the light reflected from internal surfaces from re-traversing the optical system.

A computer controls the measurement and is used for storage and manipulation of the detected signals, for storage and/or calculation of a proper form of the optical model (usually as a model matrix containing light-scattering vectors per unit of volume per size class, scaled to the detector's geometry and sensitivity) and for calculation of the PSD (see Annex A for the theoretical background of laser diffraction). Also, it may provide automated instrument operation.

Significant differences exist, both in hardware and software, not only between instruments from different manufacturers but also between different types from one company. The instrument specifications should give adequate information for proper judgement of these differences. Annex B contains recommendations for the specifications of laser diffraction instruments.