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Particle size analysis — Laser diffraction methods

Analyse granulométrique — Méthodes par diffraction laser

(Revision of ISO 13320-1:1999)

ICS 19.120

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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.

ISO 13320 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

Annexes A to E of this part of ISO 13320 are for information only.

Introduction

The laser diffraction technique has evolved such that it is now a dominant method for determination of particle size distributions. 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 first version of this ISO Standard 13320-1 was published in 1999, the understanding of light scattering by different materials and the design of instruments has advanced considerably. This is especially marked in the ability for the measurement of very fine particles. Therefore, it is necessary to revise this International Standard to capture the most recent advances in understanding.

Particle size analysis — Laser diffraction methods

1 Scope

This ISO 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. ISO 13320 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. Some advance is also noted for particles smaller than 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 will be different from those obtained by methods based on other physical principles (e.g. sedimentation, sieving).

2 Normative references

The following normative documents contain provisions that, through reference in this text, constitute provisions of ISO 13320. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on ISO 13320 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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: Characterisation of a classification process*

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

ISO/FDIS 14488:2007, *Particulate materials — Sampling and sample splitting for the determination of particulate properties*

NOTE ISO/FDIS 14488 is under development.

3 Terms, definitions and symbols

3.1 Terms and definitions

3.1.1
absorption
reduction of intensity of a light beam traversing a medium; the energy is lost as heat or may be re-radiated as fluorescence and/or phosphorescence

3.1.2
coefficient of variation (also known as relative standard deviation)
relative measure (%) for precision: standard deviation divided by mean value of population and multiplied by 100

3.1.3
complex refractive index
N_p refractive index of a particle, consisting of a real and an imaginary (absorption) part[1]

$$\boldsymbol{N_p = n_p - ki}$$

3.1.4
relative refractive index
m complex refractive index of a particle, relative to that of the medium[2]

$$m = N_p/n_m$$

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

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

3.1.7
extinction
attenuation of a light beam traversing a medium through absorption and scattering

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

3.1.9
multiple scattering
subsequent 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 (in contrast to single scattering)

[1] This document follows the convention of adding a minus sign to the imaginary part of the refractive index. Both n and k are positive numbers; i stands for $\sqrt{(-1)}$.

[2] In most applications, the medium is transparent and, thus, its refractive index has no imaginary part.

3.1.10
obscuration
optical concentration
percentage or fraction of incident light that is attenuated due to extinction (scattering and/or absorption) by the particles (obscuration = 1 – transmission, when expressed as a fraction.)

3.1.11
optical model
theoretical model used for computing the model matrix for optically homogeneous spheres with, if necessary, a specified complex refractive index, e.g. calculation by Fraunhofer diffraction or Mie scattering

3.1.12
reflection
change of direction of a light wave at a surface without a change in wavelength or frequency

3.1.13
refraction
change of the direction of propagation of light determined by change in the velocity of propagation in passing from one medium to another; in accordance with Snell's law

$$n_m \sin \Theta_m = n_p \sin \Theta_p$$

3.1.14
repeatability (instrument)
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)
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)
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
general term describing the change in propagation of light at the interface of two media having different optical properties

3.1.18
scattering angle
angle between the principal axis of the transmitted light beam and the scattered light

3.1.19
scattering pattern
angular or spatial pattern of light intensities [$I(\theta)$ and $I(r)$ respectively] 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
percentage or fraction of incident light that remains un-attenuated by the particles (transmission = 1 – obscuration, when expressed as fraction)

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) or the coefficient of variation (relative percentage) is used. Then, about 95 % of the population of particles falls within ± 2 standard deviations from the mean value and about 99,7 % within ± 3 standard deviations from the mean value. The difference $x_{90} - x_{10}$ corresponds to 2,6 σ.

3.2 Symbols

A_i	extinction coefficient of size class i
a	distance from scattering object to detector
b	illuminated path length containing particles, mm
C	particulate concentration, volume fraction
CV	coefficient of variation, %
f	focal length of lens, mm
$I(\theta)$	angular intensity distribution of light scattered by particles (scattering pattern)
I_h	Intensity of horizontally polarised 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 polarised light at a given angle
i	square root of (-1)
i_n	photocurrent of detector element n, µA
k	wave number: $2\pi/\lambda$
k_p	imaginary (absorption) part of particle's refractive index
L	vector of photocurrents (i_1, i_2,... i_n)
M	model matrix, containing calculated detector signals per unit volume of particles in all size classes
m	relative, complex refractive index of particle to medium
n_m	real part of refractive index of medium
n_p	real part of refractive index of particle
N_p	complex refractive index of particle
O	obscuration (1 – transmission)
r	radial distance from focal point in focal plane, µm
V	vector of volume concentrations in size classes (V_1, V_2 , … V_i)
V_i	volume concentration of size class i
v	velocity of particles in dry disperser

x particle diameter, µm

x_{50} median particle diameter, µm; here used on a volumetric basis, i.e. 50 % by volume of the particles is smaller than this diameter and 50 % is larger

x_{10} particle diameter corresponding to 10 % of the cumulative undersize distribution (here by volume), µm

x_{90} particle diameter corresponding to 90 % of the cumulative undersize distribution (here by volume), µm

α dimensionless size parameter: $\pi x/\lambda$

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 refraction)

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

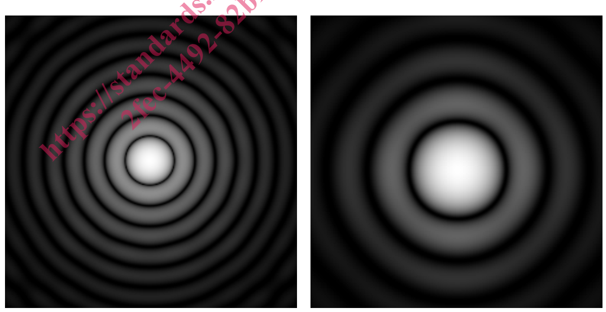
λ wavelength of illuminating light source in vacuum, nm

σ standard deviation

4 Principle

A representative 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.

The laser diffraction technique for determination of particle size distributions 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)

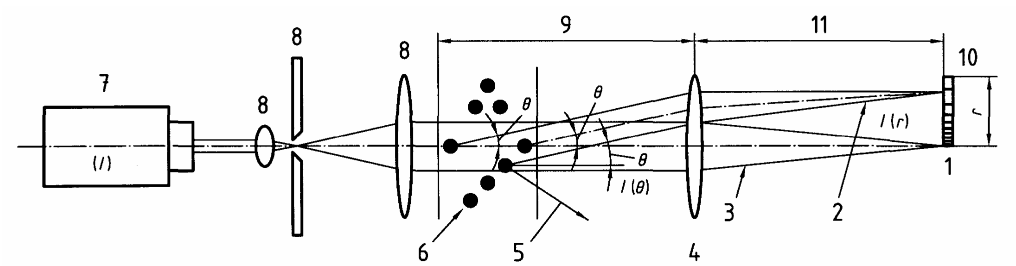
Figure 1— Scattering pattern for two spherical particles: the particle generating pattern a) is twice as large as the one generating pattern b) (simulated images for clarity)

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	7	Light source (e.g. laser)
2	Scattered beam	8	Beam processing unit
3	Direct beam	9	Working distance of lens 4
4	Fourier lens	10	Multi-element detector
5	Scattered light not collected by lens 4	11	Focal distance of lens 4
6	Ensemble of dispersed particles		

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

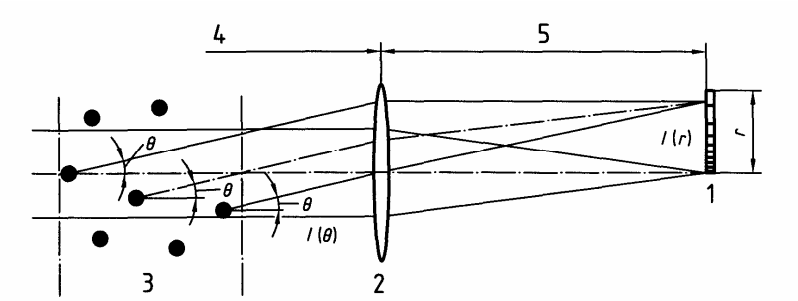
A representative sample of particles, dispersed at an adequate concentration, is passed through the light beam in a measuring zone by a transporting medium (gas or liquid). This measuring zone should be within the working distance of the lens used. Sometimes, the particle stream in a process passes directly through the laser beam for measurement. This is the case in measurement of sprays and aerosols. In other cases (such as emulsions, pastes and powders), representative samples can be dispersed in fluids and caused to flow through the measurement zone. Often dispersants (wetting agents; stabilisers) and/or mechanical forces (agitation; sonication) are applied for de-agglomeration of particles and for stabilisation 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 de-agglomeration. 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 path length for the sample is allowed within the working distance of the lens. The Reverse Fourier set-up demands small path lengths but provides one solution that enables the measurement of scattered light at larger angles.

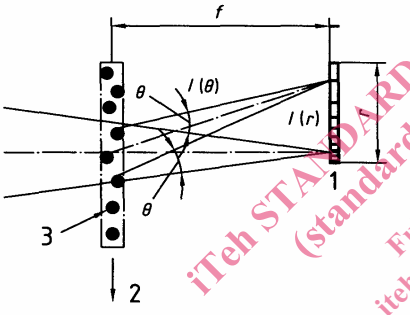
The interaction of the incident light beam and the ensemble of dispersed particles results in a scattering pattern with different light intensities scattered at various angles (see Annex A for theoretical background of laser diffraction). The total angular intensity distribution $I(\theta)$, consisting 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	4	Working distance
2	Fourier lens	5	Focal distance
3	Ensemble of dispersed particles		

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



Key

1	Detector
2	Flow through cuvette for dispersed particles
3	Particle

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:

- An extra light source at the same optical axis having a different wavelength.
- One or more off-axis light sources, either at less or at more than 90 degrees with respect to the optical axis.
- Polarisation filters for light source and detectors.
- Scattered light detectors at angles smaller than 90 degrees but larger than the conventional angular range (forward scattering).
- Scattered light detectors at around 90 degrees for measurement of intensities in different polarisation directions.
- Scattered light detectors at angles larger than 90 degrees (backscattering).