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Determination of particle size distribution — Single-particle light interaction methods —

Part 1:
Light interaction considerations

Détermination de la distribution granulométrique — Méthodes d'interaction lumineuse de particules uniques —

Partie 1: Considérations relatives à l'interaction lumineuse

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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 3.

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 part of ISO 13323 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13323-1 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

ISO 13323 consists of the following parts, under the general title *Determinatrion of particle size distribution — Single-particle light interaction methods*:

— *Part 1: Light interaction considerations*

— *Part 2: Light-scattering single-particle light interaction device design, performance specifications and operation requirements*

— *Part 3: Single-particle light-extinction device design, performance specifications and operation requirements*

Annexes A, B and C of this part of ISO 13323-1 are for information only.

Introduction

Measurement of individual particles by interaction with light has been carried out for many years using a variety of instruments. These instruments vary in optical design, light-source types, and means of particle presentation to the light. For these reasons, data from nearly identical particle sources frequently differ when different instruments are used for measurement. In addition, the extent of light interaction produced by a particle is affected by several physical parameters in addition to the particle size. The purpose of this part of ISO 13323 is to define the basis for, and to reduce the variability of, data produced by light interaction methods of particle size measurement.

Particle size measurement by single-particle light interaction devices normally involves either determination of the light scattered as a result of the light interaction with a single-particle or the amount of light extinction caused by the presence of the particle in the light beam. This part of ISO 13323 will discuss the principle of the light interaction phenomena that are measured. The general performance and operational parameters that are pertinent to the instruments and to the particle/fluid environment in which the instruments operate will be summarized. Specific instrument types, operation, and performance are not discussed in this part of ISO 13323.

Determination of particle size distribution — Single-particle light interaction methods —

Part 1:
Light interaction considerations

1 Scope

This part of ISO 13323 provides guidance on the selection and operation of devices that determine the size and number of particles by measuring the phenomena resulting from light interaction with individual particles present in a gas or liquid. The reported particle size is defined as an equivalent optical size based upon the response of the measurement system to calibration particles. This definition requires that the instrument be calibrated with well-defined materials.

This part of ISO 13323 applies to particles ranging in size from approximately 0,05 µm in diameter to the millimetre size range. Gas-borne particles in sizes from approximately 0,05 µm to 20 µm or so are measured primarily by light-scattering. Larger particles can be measured using light extinction sensors. Liquid-borne particles in the size range from approximately 0,05 µm to a few micrometres are measured by light-scattering. Light extinction is used to measure liquid-borne particles in sizes from approximately 1 µm to the millimetre size range. The size range capability of any single instrument is usually approximately 100:1. Particles larger than approximately 100 times the size of the smallest particle that can be measured with good sizing resolution are reported as “greater than or equal to the threshold size” of the largest size channel of the instrument.

The response that is considered in this part of ISO 13323 is the change in collected light flux resulting from the presence of a single-particle within the optical sensing zone of the measuring instrument. For this reason, instruments, which rely upon optical interaction to produce data only indicating the extent of particle motion, are not discussed here.

NOTE Instruments not discussed here include devices such as aerodynamic particle sizers or phase Doppler particle analysers, which produce data primarily dependent upon the aerodynamic size of the particles. Those instrument types do not use the extent of light interaction to measure the particle size. The particle size is defined by residence time during motion through a defined distance or by particle velocity. These instruments report a particle size that is related to fluid-dynamic measurements.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13323. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 13323 are encouraged to investigate the possibility of applying the most recent editions of the normative documents 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 3165, *Sampling of chemical products for industrial use — Safety in sampling*.

ISO 6206, *Chemical products for industrial use — Sampling — Vocabulary*.

ISO 14887[1)], *Sample preparation — Dispersing procedures for powders in liquid*.

3 Terms and definitions

For the purposes of this part of ISO 13323, the following terms, definitions and symbols apply.

3.1 Definitions

3.1.1
absorption
reduction of intensity of a light beam traversing a medium (fluid or particle) by energy conversion in the medium

3.1.2
coincidence
presence of more than one particle within the sensing zone of an instrument at any time

NOTE The effects include decreased indication of particle population and increased indication of particle size, since several particles can be reported as a single larger one.

3.1.3
relative complex refractive index
refractive index of a particle relative to that of the fluid medium (n_{m}) in which it is suspended, consisting of a real part (n_{p}) and an imaginary (absorption) part (ik_p)

$$m = \frac{n_{\mathrm{p}} - ik_p}{n_{\mathrm{m}}} \quad (1)$$

3.1.4
counting accuracy
ratio of the reported population to the true population in the measured sample

NOTE The counting accuracy may be expressed as counting efficiency by multiplying the ratio by 100.

3.1.5
equivalent optical diameter
diameter reported by a single-particle light interaction device, based upon the light interaction signal from that single-particle being equivalent to that from a calibration particle of known dimensions and optical properties

NOTE This diameter will vary with the optical system of the device and particle/fluid optical properties and some physical properties.

3.1.6
extinction
attenuation of light through absorption and scattering when passing through or otherwise interacting with a medium

3.1.7
multiple scattering
three-dimensional spatial pattern of light intensity emitted from a particle from scattering of light from the primary light source and light scattered from other particles in the sensing volume which is directed to the particle of concern in the sensing zone

3.1.8
reflection
return of radiation by a surface without change in wavelength

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1) To be published.

3.1.9
refractive index
ratio of the velocity of light in a medium to the velocity in a vacuum which is expressed as the combination of a real and an imaginary term

NOTE The real term expresses the light velocity ratio and the imaginary term expresses the fraction of incident light absorbed by the medium through which the light passes.

3.1.10
refraction
change in the direction of light propagation as a result of change in the velocity of propagation in passing from one medium to another

3.1.11
reported size range
size channel
size range defined by a particle sizing instrument

NOTE When several size ranges are reported, the lower and upper range limits are shown. The upper limit of all but the largest size range is equal to the lower limit of the next larger range. The size limits of the largest range is typically defined as “equal to or greater than x”, where x is the lowest size limit of that range.

3.1.12
scattering
general term describing the change in light propagation at the interface of two media

3.1.13
scattering pattern

three-dimensional spatial pattern of light intensity emitted from a particle as a result of scattering of light transmitted from the primary light source to the particle being measured in the optical sensing zone

3.1.14
sensing zone
sensing volume
volume within the instrument that is optically and physically defined where particle interaction with light is observed and used to develop data on particle size and quantity

3.1.5
Stoke's number
St
product of particle relaxation time (t), time for a particle to accommodate to a fluid velocity change and actual particle velocity (v), divided by the sample probe inlet size (d_i)

$$St = \frac{tv}{d_i} \qquad (2)$$

3.1.6
extinction coefficient
E
ratio of total light flux scattered and/or absorbed by a particle to the light flux incident upon the particle

3.2 Symbols

a Particle radius

A Projected area of particle(s) illuminated by incident light

c_n Numerical particle concentration

E Extinction coefficient

$I(\theta)$ Angular intensity distribution of light scattered by a particle

$I(r)$ Light flux scattered by a particle at a specific solid angle

I_1 Scattered light polarized in a direction perpendicular to the incident light beam

I_2 Scattered ight polarized in a direction parallel to the incident light beam

k_p Imaginary (absorption) part of a particle refractive index

l Beam path through a sensing zone

n Refractive index of a particle, relative to that of the suspension medium

n_m Real part of the refractive index of the suspension medium

n_p Real part of the refractive index of the particle

x Particle diameter, in micrometres. Unless otherwise specified, the equivalent optical diameter is reported

y Ratio of scattered or transmitted light flux to incident light flux

α The term of particle projected area divided by the illumination wavelength, $2\pi A/\lambda$

θ Scattering angle with respect to forward direction in degrees. The scattering angle may consist of a significantly large solid angle, but is typically defined as the centre angle of the light collection system with respect to the centre line of the illumination source

λ Wavelength of the illumination source, in nanometres. The illumination source may emit light at a single wavelength or over a broad range of wavelengths

4 Light interaction principles

4.1 Introduction

A brief summary of the parameters affecting light interaction with single-particles is presented in this clause. Further details are provided in annex A. In single-particle light interaction devices, the output data are affected by illumination wavelength and intensity, illumination source and collection optics configurations, as well as light collection and capabilities of the data handling system. Particle and suspension fluid physical properties affect response, as well. The particle size, shape, and orientation within the sensing zone may also affect the response. The relative refractive index of the particle in the suspension fluid also affects the response.

4.2 Light scattering

Most particles measured by light-scattering will be in the size range from approximately 50 nm to 100 µm. When light interacts with the particle, the scattered light flux varies roughly with the projected area of the particle for particles with radius significantly larger than the light wavelength. For smaller particles, the variation of the scattered light flux changes with particle radius, increasing to the sixth power as particle size decreases to approximately 0,2 µm. A light-scattering system used for submicrometer particles will normally be used to measure particles over a size range up to 50:1. A system that is used to measure particles larger than approximately 1 µm can measure over a size range of approximately 100:1. The limitations are connected with the linearity of electronic data processing systems over wide ranges (e.g. 5×10^6) and the need to ensure that the smallest signal that is processed is larger than the electronic and optical background noise level.

NOTE Further details on the operation of light-scattering instruments for counting and/or sizing particles can be obtained from the reference [3] in the bibliography.

4.2.1 Physical principles

Measurement of scattered light from fluidborne particles is carried out by observing the scattered light from the particle at a specific solid angle that is defined by a particular instrument configuration. The particle(s) within the sensing zone of the instrument may be present within a known volume of fluid moved through the instrument or may be moving with “normal” fluid flow through a defined known sensing zone established within the instrument. In the first case, the particle concentration per unit fluid volume is defined; in the second case, the particle flux per unit area through which particles are moving is defined on a time basis. The instruments which report the particle concentration are normally used to characterize particle size distribution and concentration in fluids which are moving under specific conditions at pressures ranging from near ambient to approximately 500 kPa. Instruments defining particle flux are often used when flow is random or at pressures from approximately 1 kPa to ambient.

4.2.2 Optical system designs

Typical optical system bases for single-particle light-scattering instruments are shown in Figure 1. The configurations shown here describe essentially every optical design of the light-scattering instrument used for this purpose. The original designs, laid out in 1965, used incandescent-filament illumination sources, lens and aperture systems to define sensing zones. A summary of single-particle light-scattering instruments designed for aerosol studies was shown recently. Current optical systems use either gas or diode-laser illumination that may not require the same type of beam-shaping systems. Even so, the basic optical system designs for light collection are still followed. The choice for selection is based upon the particle size range of concern, available components, construction funds, and the environments in which particle measurements are to be made. Essentially, the same optical design base can be used for measurements in gas or in liquid suspension. The major differences are in the fluid control systems used for the two applications. The problems of defining the edges of the sensing zones are greater when working with liquid systems. Larger particles are more frequently measured in liquid than in gas and a small portion of a liquid-borne large particle may move through an optically defined sensing zone, scattering as much light as a small particle entirely within the sensing zone. Procedures for minimizing this effect and other problem areas will be discussed in ISO 13323-2 and ISO 13323-3.

NOTE Further information on the optical design of airborne-particle counting systems can be obtained from reference [4] in the bibliography.

In general, particle counters using monochromatic light sources and forward-scattering systems with a small solid angle produce a multi-valued response of scattered light flux as a function of particle size. The response will increase and decrease with particle size over some portions of the instrument dynamic range. Particle counters with polychromatic light, and especially those with scattering systems with a large solid angle, produce the desirable response where scattered light flux does not increase and decrease as the particle size increases, but the instrument sensitivity for small particle measurement may be decreased unless illumination intensity is increased and the design of the optical and electronic systems minimizes background noise levels. In this connection, laser illumination systems can generate light flux intensity levels of several watts in the sensing zone. The use of light-collection optical systems with a large angle here will also minimize multi-valued response.

