## INTERNATIONAL **STANDARD**

**ISO 13323-1**

> First edition 2000-11-01

## **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



Reference number ISO 13323-1:2000(E)

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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 welldefined 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 um to 20 um 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<sup>1</sup>), 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  $(n_n)$  and an imaginary (absorption) part  $(ik_n)$ 

$$
m = \frac{n_{\rm p} - ik_p}{n_{\rm m}}\tag{1}
$$

#### **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

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  $(d_i)$ 

$$
St = \frac{tv}{d_i} \tag{2}
$$

#### **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*<sup>n</sup> 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*<sup>1</sup> Scattered light polarized in a direction perpendicular to the incident light beam
- *I*<sub>2</sub> Scattered ight polarized in a direction parallel to the incident light beam
- $k<sub>n</sub>$  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
- $n_{\rm m}$  Real part of the refractive index of the suspension medium
- *n*<sub>p</sub> Real part of the refractive index of the particle
- *x* Particle diameter, in micrometres. Unless otherwise specified, the equivalent optical diameter is reported  $\mathbf{a}$
- *y* Ratio of scattered or transmitted light flux to incident light flux
- $\alpha$  $\alpha$  The term of particle projected area divided by the illumination wavelength,  $2\pi A/\lambda$
- $\theta$  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
- $\lambda$  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  $\mu$ m 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.  $5 \times 10^6$ ) 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 lightcollection optical systems with a large angle here will also minimize multi-valued response.



**Key**

- 1 Light
- 2 Aperture
- 3 Traps
- 4 Aerosol
- 5 Collection

#### **Figure 1 — Basic optical designs for light-scattering particle counters**

#### **4.3 Light extinction**

#### **4.3.1 Physical principles**

Light extinction is mainly used for counting and sizing particles in liquid. These instruments are used for particles larger than approximately 1 µm in diameter. This limitation arises from the effects of variations in the refractive index ratio on sizing response in liquids. In some cases, these instruments are used for sizing and counting dry particles in sizes that permit moving them through the sensing zone at a rate so that residence time for counting or sizing is no more than 50 µs or so. Most liquids have refractive index values quite close to those of many particles; most gas refractive indexes are much lower than that of the particles so a small change in particle type has little effect upon the refractive index ratio for gas substrates. Illumination shall be provided by a gas or solid-state laser or by an incandescent filament lamp. During operation, the light flux level of the beam through the sensing zone is continually measured by the photodetector. When a particle passes through the sensing zone, some light is removed due to scattering out of the direct beam and/or by absorption by the particle. The scattering coefficient is strongly dependent upon the particle/fluid refractive index ratio, particularly for particles less than or equal to 3 µm to 5 µm. For this reason, extinction counters using different types of illumination can report different sizes for identical particles with shape or optical characteristics that differ from those of the calibration particles.

#### **4.3.2 Optical system design**

Figure 2 shows the basic operation of an extinction optical system that observes a full sample stream. The crosssectional area of the flow passage is defined by a transparent rectangular or circular tube with the sensing zone height usually defined by the illumination beam. Although the illustration indicates a gas laser as the illumination source, many extinction particle counters still in use are fitted with an incandescent filament lamp for illumination. In the time period 1990 to 1995, most particle-counter producers began using solid-state lasers for illumination because of their small size, high power and stability. As advancement in power levels continues to increase, these light sources will continue to be more widely used in this field.

#### **Key**

- 1 Reference diode
- 2 Laser
- 3 Condenser lenses
- 4 Liquid out
- 5 Liquid in
- 6 Signal diode



**Figure 2 — Operation of an extinction optical system**

#### **5 Performance of particle measurement device**

#### **5.1 Particle-sizing accuracy**

Data produced by optical single-particle counting instruments are affected by several system parameters in addition to the size of the particles being measured. For this reason, the statement of particle-sizing accuracy is based upon the capability for accurate measurement of calibration particles. There are two types of particles used for calibration. In some application areas (pharmaceutical or fine chemicals production), isotropic spheres of known refractive index are used; each batch of these calibration particles is monodisperse with a Gaussian particle size distribution with a known small standard deviation. In areas where there are a wide variety of irregularly shaped particles with the possibility that the nature of the material may not be known or may vary with time, the standard calibration particles are polydisperse with a known particle size distribution and are prepared as a suspension with a carefully determined concentration of the particles in the liquid which is of concern. When a batch of monodisperse calibration particles is measured by an instrument, the median value of the reported particle size distribution for monodisperse calibration particles shall not vary from the correct value (defined by a suitable reference method) of the standard particle batch by more than 5 %. When a batch of polydisperse calibration particles is measured by an instrument, the cumulative concentration at any selected size shall not differ from the specified value by more than 10 % unless the specified population of particles at the selected size is so small that statistical effects for "sparse" sample data indicate that an anticipated confidence level of 90 % cannot be expected.

In either case, the instrument performance in terms of variation from the specified values shall be verified by calibration at intervals no greater than that specified by the instrument producer or by reference to an operational standard calibration method agreed upon between the producer and purchaser of a product whose quality is defined by data from the particle-sizing instrument. A maximum interval of no more than one year is recommended.

#### **5.2 Particle-sizing resolution**

The particle-sizing resolution defines how well a particle counter can differentiate between particles of nearly the same size. This capability affects particle counting and sizing accuracy, particularly at the small size ranges. Counters with poor resolution identify a broader range of particles both above and below the threshold size as being at that size than do counters with better resolution. The result is that counters with poor resolution indicate a larger number of particles at the smallest size range of the counter because of the steep slope of the power-law particle size distribution seen in many particle systems.

The particle-counter-sizing resolution is stated as the increased width of the size distribution reported for nearmonodisperse particles. A counter with acceptable resolution shall increase the reported standard deviation for monosize particles by no more than 10 %. Values of 3 % to 5 % are common for instruments with sample rates up to a few hundred millilitres per minute. For higher flow rates, the sensing zone area in the direction orthogonal to the flow path is increased to ensure that the particle residence time in that sensing zone is sufficient for accurate sizing. When sensing volume dimensions are increased, the scattering angles for particles passing through different parts of the sensing zone may differ to the point where significant variation in signal will result from identical particles, depending upon their location in the sensing zone.

#### **5.3 Particle-counting accuracy and concentration limits**

When the particle counter is operating correctly, the counting accuracy for monodisperse particles at the upper and lower boundaries for each size channel shall be 50  $% \pm 5$  %. This means that half of the particles equal to the lower boundary size channel threshold are reported within the channel and half are reported within the contiguous smaller size range channel. Similarly, half of the particles equal in size to the upper boundary size of that channel are reported as being within the channel and half are reported as being within the contiguous larger size range channel. If the channel of concern is the smallest size range of the instrument, then all particles that produce a signal approximately larger than the minimum size threshold or smaller than the maximum threshold for that channel should be counted with 100 % accuracy. This situation is normal for single-particle counting instruments with a number of contiguous size-range reporting levels. However, if the optical system is misaligned so that some sampled liquid bypasses the sensing zone, the counting efficiency will also be low. If the flow system of the counter is designed or constructed so that flow eddies exist within the sensing zone, then an excessively high counting rate will be reported, due to sample recirculation through the sensing zone. --`,,`,-`-`,,`,,`,`,,`---

The sample fluid measuring system also affects the counting accuracy. Flow-measurement accuracy is a greater problem with gas-borne particle counters than with liquid-borne particle counters. Many gas-borne particle counters are used in areas where concentration is reported as particles per unit volume of gas at ambient pressure and temperature rather than at standard conditions. If the counter uses a gas-flow metering system based on the mass flow measurement, then moving the instrument from a sea level environment to a mountain area can result in signficant errors in the reported particle concentration.

If the particle concentration in the sample is greater than the design limit for the counter, particle coincidence within the sensing zone will cause low counting efficiency. This is due to more than one particle being present within that zone at any time. In that situation, the coincident particles are reported as an individual particle with a size equivalent to the summed size (based on the viewed area) of the individual particles simultaneously present within the sensing zone. The resulting data are then incorrect, both in terms of inadequate particle number and incorrrect (usually oversize) particle size being reported. If the particle concentration in the sample is very low, the statistical validity of the reported data will also be low. This situation arises simply because particles are randomly situated in any suspension and very small samples are not representative of the entire population from which they are procured. Required minimum data quantities for specific instruments shall be based upon sample-volume measurement capabilities and specified confidence limits. However, when measuring suspensions with very low concentration, it may not be possible to report a specified confidence limit unless large sample volumes are measured or many small samples are observed.

#### **6 Particle-counter operation**

No matter how well an instrument is designed and constructed, it must be operated correctly in order to produce valid data. The instrument shall be in good working order and it shall have a current calibration report. The standard procedures for ensuring operability and calibrating the instruments require preparation of documentation verifying the status of the instrument. The operator shall ensure that the instrument is in good condition and that the documentation is valid and current. Further details will be provided in the discussions on specific instrument types in ISO 13323-2 and ISO 13323-3.

The instrument shall not be used in an environment which exceeds its capabilities. The external environmental temperature and relative humidity, as well as the sample fluid composition, temperature and pressure shall meet the limits defined by the manufacturer. In addition, sample acquisition and handling procedures, which will not degrade the sample in any way before or during the measurement, shall be used.

#### **6.1 Environmental constraints**

For most particle measurement instruments, environmental requirements are defined by the manufacturer. These include information on allowable environmental temperature, pressure, and relativd humidity ranges; electrical power, voltage, and frequency range limits are provided; sample-fluid condition limits may also be given. These may include fluid composition limits or sample line pressures, particularly if particles in corrosive fluids or in very high pressure systems are to be characterized.

#### **6.2 Sample-acquisition requirements**

Detailed discussions of powder system sampling and powder dispersion into fluids are not considered here. A brief discussion of sample acquisition from fluid systems which contain particles is presented here. In addition, consideration of fluid flows within the measuring instruments is included in the discussions of specific instruments in ISO 13323-2 and ISO 13323-3.

Sample acquisition shall not change the environment from which the sample is taken or change the sampled fluid in any way during the sampling process. Samples shall be acquired at a sufficient number of locations to ensure that any variations which may be present in the entire fluid system will be encompassed by that sampling process. At each sample location, a sufficient volume shall be sampled to permit generation of enough data to allow the application of normal statistical processing to the measurements collected at that location in order to define data with a satisfactory confidence limit.

When sampling from a flowing fluid, differences between the velocities of the stream and the sampled fluid entering the sample-probe inlet affect the sample-probe inlet efficiency because of particle inertia. When these velocities are identical, sampling is isokinetic. This requires that the probe inlet faces into the fluid stream flow and shall be parallel with that flow (isoaxial sampling). In sampling gas flows, anisokinetic sampling effects are trivial for situations where the Stoke's number is less than one. In this situation, particles accommodate quickly to a change in velocity between that of the sampled air stream and that of the sample probe. This situation exists for particles smaller than about 2 µm in diameter and for conditions where the mismatch between the sample probe and sampled-air velocity is less than a factor of 1,5 or so. For larger particles and greater velocity differences, sampling errors of more than 5 % can be expected. When sampling from liquid streams, particle inertia forces are decreased due to the greater density and viscosity of liquids as compared to gas. In that situation, anisokinetic sampling has little effect when measuring particles less than 10 um in diameter with the stream/probe velocity mismatch of 1.5.  $\ddotsc$ 

When sampling from a turbulent or static fluid body, isokinetic sampling is not possible. It is necessary to ensure that particles in the fluid are well dispersed and that the dispersion process has not introduced artifacts or removed particles from the fluid. Artifact introduction is a problem when working with liquid; dispersion involves agitation or mixing with possible introduction of gas bubbles during mixing. Care shall be taken to control the mixing intensity to a level that provides suitable dispersion within the liquid without introducing air, which forms bubbles that can be interpreted as particles. When working with static gas suspensions, particle loss due to deposition on surfaces can occur due to Brownian motion, sedimentation, electrostatic effects, etc. A more serious problem arises from atypical gas movement due to introduction of the sample probe into the body of static gas. This may cause changes in particle trajectories within the gas. In that case, exact definition of the gas volume from which the sampled particles were acquired becomes very difficult. Again, as with isokinetic sampling, the smaller particles (about 3 µm) will follow the air streamlines quite closely, but the larger particles will not do so.

After a sample has been collected, it may be necessary to transport it to the measuring instrument through a transit line. Particles may be lost as a result of deposition on the transit line walls. Such losses should be minimized by keeping the lines vertical, minimizing the residence time in the line, avoiding abrupt changes in line dimensions and configuration, and by maximizing any radius of curvature if line direction changes are required. These precautions will tend to minimize large particle losses by gravitational deposition and small particle losses by turbulent or laminar deposition.

## **Annex A**

(normative)

## **Theoretical background of light scattering**

The derivation of equations defining light-scattering is available in the literature and will not be repeated here. Without going into detail on the derivation, the important parameters which define scattering from a particle include the particle size, the relative refractive index ratio (both real and imaginary components) of the particle and of the suspension medium, the illumination wavelength, and the solid angle over which the light is collected. The theoretical relationship for light-scattering by small particles is shown in equations A.1 and A.2, the Rayleigh scattering equations for small particles. Equation A.1, for perpendicular polarized light, shows nearly equal scattering through all angles, while equation A.2 for parallel polarized light, shows scattering equal in intensity to that for perpendicular polarization at  $\theta$  = 0 rad (0°) and  $\theta$  =  $\pi$  rad (180°), while decreasing to zero at  $\theta$  = 0,5 $\pi$  rad (90°), as shown in Figure A.1.

$$
I_1 = \frac{\alpha^4}{2\pi} \times \left(\frac{n^2 - 1}{n^2 + 2}\right)^2
$$
 (A.1)

$$
I_2 = \frac{\alpha^4}{2\pi} \times \left(\frac{n^2 - 1}{n^2 + 2}\right)^2 \cos^2 \theta
$$
 (A.2)





Equation A.3 is the Mie scattering equation describing scattering for particles of larger size with vertically polarized light. Equation A.3 describes scattering for horizontally polarized light. Figure A.2 shows the scattering pattern expected from slightly larger particles and Figure A.3 shows the scattered-light flux variation with a scattering angle for particles significantly larger than the light wavelength.

$$
I_2 = \left| \sum_{n=1}^{\infty} \frac{2n+1}{n(n+1)} \left[ a_n \tau_n \left( \cos \theta \right) + b_n \pi_n \left( \cos \theta \right) \right] \right|^2 \tag{A.3}
$$

where

 $a_n$  and  $b_n$  are complex amplitude functions dependent on *n* and *x*.

 $\pi_n$  and  $\tau_n$  are angular functions depending on  $\theta$ .







 $x \gg$ illumination wavelength

**Figure A.3 — Scattered-light flux variation with scattering angle for very large particles**

## **Annex B**

(informative)

## **Theoretical background of light extinction**

If a well-collimated beam of light is passed through a fluid, any particle with a light extinction coefficient other than zero which is present in the light path will remove light by a combination of scattering out of the direct beam and by absorption. If the absorption part of the particle refractive index  $(k<sub>n</sub>)$  is zero, light is removed from the beam solely by scattering. The particle extinction coefficient defines the combined effects of scattering and absorption. Beer's law, relating the transmitted and incident light flux, may be written as shown in equation B.1.

$$
I = \exp(-Ac_n l) \tag{B.1}
$$

Figure B.1 shows some calculated and measured particle extinction coefficients for spheres with common refractive indices. The extinction coefficient oscillates about the value 2 for small particles, with differences that vary with refractive index ratio. However, the extinction coefficient approaches the value 2 very closely for larger particles of almost any refractive index.

Extinction cœfficient  $\overline{5}$  $n = 1,5$  $n = 1,33$  $\frac{1}{4}$  $n = 1,5-0,6i$  $\overline{\mathbf{3}}$  $\overline{2}$  $\mathbf{1}$  $0<sup>1</sup>$  $\sigma$  $0,5$  $\overline{1}$  $1,5$  $\overline{2}$  $2,5$  $\overline{\mathbf{3}}$ Particule diameter, um a) Mie theory Extinction cœfficient 1  $\overline{\mathbf{3}}$  $\overline{2}$  $\overline{c}$  $\overline{1}$  $\circ$ <sup>0</sup>  $\overline{10}$  $\overline{15}$  $\overline{25}$ 20  $30<sup>°</sup>$ Particule size parameter,  $\alpha$ b) Measured data

# $-1, \, \cdots, \, \cdots, \, \cdots, \, \cdots$

#### **Key**

- 1 Diamond particles
- 2 Quartz particles

**Figure B.1 — Some particle extinction coefficients**

## **Annex C**

(informative)

## **Applications for single-particle light interaction devices**

Measurement of the particle size or population in a fluid suspension is carried out with dispersions produced from powders added to a fluid or with particle suspensions already in the fluid. Dispersions are prepared with an appropriate particle concentration to ensure that individual particles, rather than flocs or agglomerates, are measured. Single-particles already present in gas or liquid are sized and/or counted by light-scattering or light extinction to define cleanliness of the fluid where particles may be present. For most measurements of ambient atmospheres, the particle content of the gas is no more than a few hundred micrograms per cubic metre of gas. Where the gas being measured is the environment in which critical production occurs, the particle concentration is defined in terms of number of particles per unit volume of gas. In some production areas, as for semiconductor device, pharmaceutical product or critical optical system production, the maximum allowable number concentrations for particles  $\ge 0.1$  µm are in the range from 1 to 10<sup>5</sup> per cubic metre. Particle suspensions in these concentration ranges are also measured to evaluate the performance of fluid handling or fluid cleaning systems.

The development of new materials for semiconductors, optical components, powder metallurgy materials, and electronic materials is frequently based upon the use of finely divided powders. These materials are produced with well-defined particle size distribution, including close control to minimize over- and under-size particles. The use of a light-scattering measurement of single-particles has been found effective in verifying product quality when a few outlier particles may cause excessive yield loss.

Procedures for producing acceptably dispersed powder suspensions are not discussed in this part of ISO 13323. However, these and other applications require that the instruments used for these measurements provide consistent and accurate data. Understanding the requirements for good instrument design, performance and operation will aid in using these devices to provide valid data.

This part of ISO 13323 provides information on the measurement of particle size distribution and concentration of particle suspensions in gas or in liquid. This information is used to define the size and concentration characteristics of the particles in the fluid or the extent of particulate contamination in that fluid. Both areas are of concern in many technological and industrial applications. Data on particle size or particle size distribution in terms of particle population are provided. Particle-volume information can be derived under some circumstances; e.g., when measuring particles with known and constant physical properties. Measurements are made to verify product or process quality, to satisfy contractual or other specifications, or to carry out research studies.

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<sup>2)</sup> To be published.

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