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

Part 1:
Light scattering aerosol spectrometer

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

Partie 1: Spectromètre d'aérosol en lumière dispersée

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Foreword

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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 21501-1 was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

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ISO 21501 consists of the following parts, under the general title *Determination of particle size distribution — Single particle light interaction methods*:

- *Part 1: Light scattering aerosol spectrometer*
- *Part 2: Light scattering liquid-borne particle counter*
- *Part 3: Light extinction liquid-borne particle counter*
- *Part 4: Light scattering airborne particle counter for clean spaces*

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Introduction

Monitoring particle size distributions and particle number concentrations is required in various fields, e.g. in filter manufacturing, in the electronic industry, in the pharmaceutical industry, in the chemical industry, in the manufacture of precision machines and in medical operations. The aerosol spectrometer is a useful instrument for the determination of the size distribution and number concentration of particles suspended in a gas. The purpose of this part of ISO 21501 is to provide the calibration procedure and the validation method for aerosol spectrometers, so as to improve the accuracy of the measurement result by aerosol spectrometers in general, and to minimize the difference in the results measured by different instruments.

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

Part 1:
Light scattering aerosol spectrometer

1 Scope

This part of ISO 21501 specifies characteristics of a light scattering aerosol spectrometer (LSAS) which is used for measuring the size, number concentration and number/size distribution of particles suspended in a gas. The light scattering technique described in this part of ISO 21501 is based upon single particle measurements. The size range of particles measured by this method is between approximately 0,06 µm to 45 µm in diameter.

Instruments that conform to this part pf ISO 21501 are used for the determination of the particle size distribution and particle number concentration at relatively high concentrations of up to 10^{11} particles/m^3.

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Application fields include:

- characterization of metered dose inhalers (MDI), dry powder inhalers (DPI) and nebulizers in pharmacy;
- production control of active agents;
- cut-off determination: impactors, cyclones and impingers;
- atmospheric aerosols: bio-aerosols, stables/composting facilities, nebulized droplets, measurements in street tunnels;
- fractional separation efficiency determination of filters.

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For the above-mentioned applications, aerosol spectrometers should determine the particle size distribution, particle number concentration, size resolution and sizing accuracy as accurately as possible. These aerosol spectrometers are not suitable for the classification of clean rooms.

2 Terms and definitions

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

2.1
particle
discrete element of the material regardless of size

[ISO 2395:1990]

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2.2
aerosol

suspension in a gaseous medium of solid particles, liquid particles or solid and liquid particles having a negligible falling velocity

[ISO 4225:1994]

NOTE In general, one divides the atmospheric aerosol into three size categories: the superfine range $x < 0{,}1$ µm, the fine range 0,1 µm $< x <$ 1 µm and the coarse range $x >$ 1 µm, where x is the particle diameter.

2.3
particle size

size of a sphere having the same physical properties in the method of analysis as the particle being described

NOTE 1 See also equivalent particle diameter (2.4).

NOTE 2 There is no single definition of particle size. Different methods of analysis are based on the measurement of different physical properties. The physical property to which the equivalent diameter refers shall be indicated using a suitable subscript or reference to the documentary measurement standard according to which the particle size was measured.

In ISO 9276 the symbol x is used to denote the particle size or the diameter of a sphere. However, it is recognized there that the symbol d is also widely used to designate these values. Therefore the symbol x may be replaced by d where it appears.

2.4
equivalent particle diameter

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diameter of the sphere with defined characteristics which behaves under defined conditions in exactly the same way as the particle being described

2.5
light scattering equivalent particle diameter

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x_{sca}

equivalent diameter of a homogeneous sphere of a reference substance (e.g. latex) which scatters defined incident light with the same radiation efficiency into a defined solid angle element

2.6
number concentration density distribution

density (frequency) distribution of the particle number concentration represented as a function of the particle size

2.7
particle concentration

indication of, e.g. particle numbers, particle mass, particle surface related to the unit volume of the carrier gas

NOTE For the exact concentration indication, information on the gaseous condition (temperature and pressure) or the reference to a standard volume indication is necessary.

2.8
coincidence error

probability of the presence of more than one particle inside the sensing zone simultaneously

NOTE Coincidence error is related to particle number concentration and size of sensing zone.

2.9
counting efficiency

relation of the concentration determined from the counting rate of the measuring instrument and the real concentration of the aerosol at the inlet of the instrument

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2.10
border zone error
particle sizing error that occurs when particles pass through the optical border of the sensing zone

3 Requirements

3.1 Size range

The measuring size range is defined by the lower and upper size limit of quantification.

3.2 Counting efficiency

3.2.1 General

The counting efficiency is the relation of the particle number concentration $C_{N,\text{measured}}$ — determined from the counting rate of a device and corrected for possible coincidence errors — to the real particle number concentration $C_{N,\text{actual}}$ of the aerosol at the inlet of the device. The counting efficiency [$\eta(x)$] is a function of the particle size and is expressed as the ratio:

$$\eta(x) = \frac{C_{N,\text{measured}}(x)}{C_{N,\text{actual}}(x)} \tag{1}$$

The counting efficiency is also a function of signal processing, the homogeneous illumination of the measuring volume and the extent to which the particles enter the measuring volume and flow rate.

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Figure 1 shows a graphical representation of counting efficiency. In an ideal case, the counting efficiency in the middle of the measuring range — as represented here — has the value one. If an experimental examination results in a value deviating from one, then this is to be accounted for as a correction to the measurement result. Usually, one defines the lower and/or upper size limit of the measuring range for the particle size with the particle diameters for which the counting efficiency shows the value 0,5.

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For a proper evaluation of a measuring instrument, it is useful to determine the complete counting efficiency curve, or to indicate the particle diameters corresponding to values of the counting efficiency (e.g. at 0,1 and 0,9) besides those corresponding to a counting efficiency value of 0,5. The counting efficiency can be determined according to 4.5.

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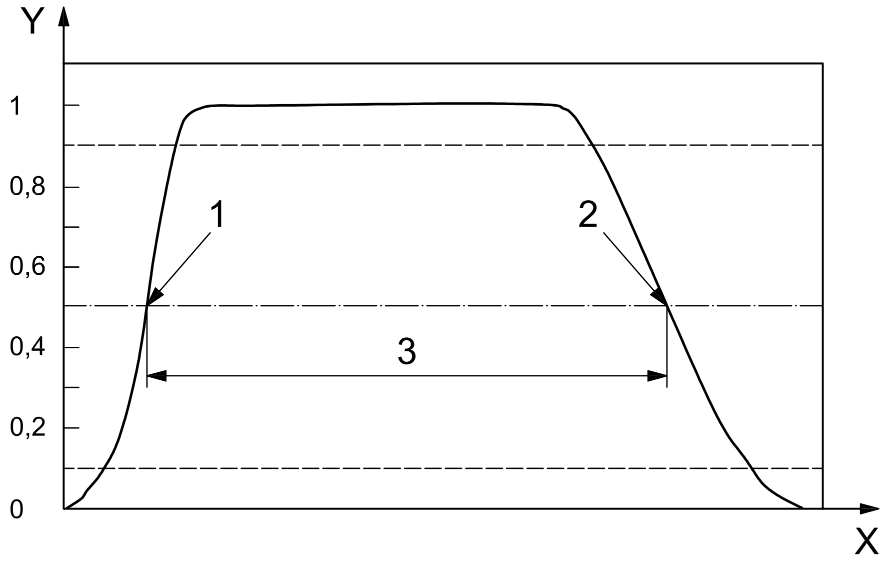

Key

X particle size

Y counting efficiency $\eta\,(x)$

1 lower size limit

2 upper size limit

3 size range

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Figure 1 — Graphical representation of the counting efficiency [5]

3.2.2 Lower size limit

The lower size limit for the particle size is defined by convention to be the smallest diameter with which the counting efficiency shall be 0,5 ± 0,15 (50 % ± 15 %; lower size limit of the measuring range).

3.2.3 Upper size limit

The upper size limit for the particle size is defined by convention to be the largest diameter with which the counting efficiency shall be 0,5 ± 0,15 (50 % ± 15 %; upper size limit of measuring range). This is of particular interest if the aerosol inlet is situated horizontally in the LSAS, as particle losses can occur in the LSAS through impact and sedimentation.

3.3 Size resolution

The size resolution indicates which neighbouring particle sizes a particle measuring instrument can still differentiate between and record separately. Aerosol spectrometers should determine the particle size distribution and the particle number concentration as accurately as possible with high size resolution and good sizing accuracy. The size resolution depends on particle size.

Almost all measuring instruments determine the particle number concentration in a limited number of size classes which are firmly specified by the instrument design (e.g. instrument geometry, evaluation electronics, evaluation software). In practical operation, the size resolution of an LSAS cannot be better than the width of its size classes.

The size resolution can be determined according to 4.4.

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3.4 Sizing accuracy

The sizing accuracy depends on the particle size. Therefore, the sizing accuracy can be evaluated for any particle size as follows

$$\varepsilon(x) = \frac{x_\mathrm{s} - x_\mathrm{r}}{x_\mathrm{r}} \times 100 \qquad (2)$$

where

$\varepsilon(x)$ is the ratio of particle size difference, in %;

x_r is the particle size of the certified reference material, in µm;

x_s is the particle size indicated by the LSAS, in µm.

The sizing accuracy of an LSAS describes the difference between the actual calibration particle size and the particle size indicated by the instrument. The correlation between the particle size and size class stated by the manufacturer (channel number) is normally based upon a calibration of the instrument with a known test aerosol [mostly polymer latex (PL) particles]. Refer to Annex E.

3.5 Sampling flow rate

The sampling flow rate is the volumetric flow rate through the LSAS. Any error in the volume flow will affect the reported particle number concentration with a proportional relationship. The error in the sampling flow rate shall be within 5 %. Typical sampling flow rate is about 0,5 l/min to 5 l/min.

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3.6 Effective detection flow rate

The effective detection flow rate is the volumetric flow rate through the optically and/or aerodynamically limited sensing zone. For particle number concentration measurements, the effective detection flow rate shall be evaluated. If the geometrical dimensions of the measuring volume are exactly known, the effective detection flow rate can be determined by measuring the transit time of the particles through the measuring volume. Otherwise, the effective detection flow rate has to be determined by a calibration experiment according to 4.2.

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3.7 Maximum particle number concentration

The LSAS must be able to measure in high particle number concentrations up to 10^{11} particles/m^3 and the maximum particle number concentration shall be specified by the manufacturer. According to 4.3, the coincidence loss at the maximum particle number concentration of an LSAS shall be equal to or less than 10 %.

4 Test method

4.1 Size calibration

The light scattered by the individual particles is detected and transformed into a voltage pulse. These pulses are classified according to their height in a multi-channel analyser (MCA). The result is a count rate histogram. This histogram is transferred into a particle size distribution by applying the calibration curve. This calibration curve, which is instrument specific, shall be determined either experimentally or theoretically. Normally, aerosol spectrometers are calibrated with monodisperse test aerosols of known (traceable) size and refractive index.

For the production of monodisperse test aerosols, several generation principles can be used. The size distribution of test aerosols with small variances are obtained if aqueous suspensions of PL particles are nebulized, dried and drawn through the instrument. Dry powder monodisperse PL particles are also useful for calibration.

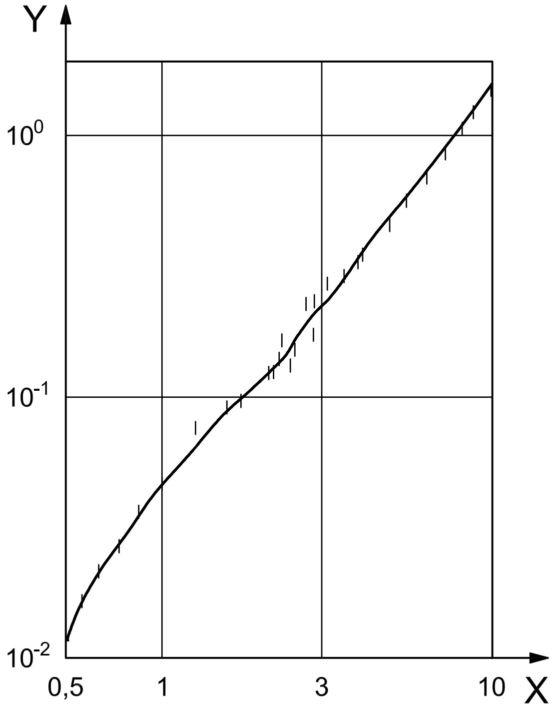

Figure 2 — Comparison of a theoretical calibration curve (line) and experimental data with monodisperse aerosol particles (vertical bars) [14]

Other techniques of providing narrow distributed aerosols are the vibrating-orifice generator or passing a polydisperse aerosol through a differential mobility analyser, which extracts particles within a narrow size range according to their electrical mobility.

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4.2 Effective detection flow rate

The effective detection flow rate is obtained by relating the instrument particle count rate to a reference particle number concentration measurement using an instrument with a higher particle number concentration range than the instrument to be calibrated and that is traceable to internationally accepted standards.

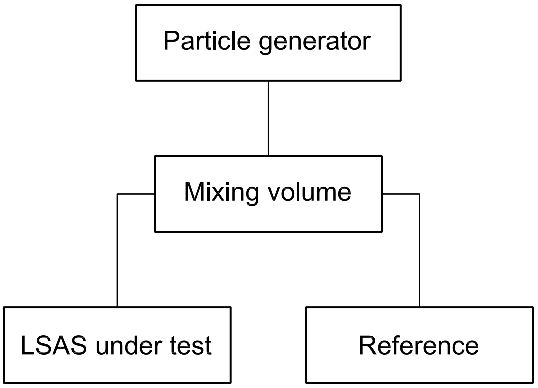

Figure 3 — Example for effective detection flow rate and counting efficiency test set-up