INTERNATIONAL STANDARD

ISO 21501-4

First edition
2007-05-15

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

Part 4:
Light scattering airborne particle counter for clean spaces

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

Partie 4: Compteur de particules en suspension dans l'air en lumière dispersée pour espaces propres

Reference number
ISO 21501-4:2007(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

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

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

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

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

ISO 21501-4 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

This first edition of ISO 21501-4, together with ISO 21501-2 and ISO 21501-3, cancels and replaces ISO 13323-1:2000, which has been technically revised.

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

— *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*

The following part is under preparation:

— *Part 1: Light scattering aerosol spectrometer*

Introduction

Monitoring particle contamination levels is required in various fields, e.g. in the electronic industry, in the pharmaceutical industry, in the manufacturing of precision machines and in medical operations. Particle counters are useful instruments for monitoring particle contamination in air. The purpose of this part of ISO 21501 is to provide a calibration procedure and verification method for particle counters, so as to minimize the inaccuracy in the measurement result by a counter, as well as the differences in the results measured by different instruments.

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

Part 4: Light scattering airborne particle counter for clean spaces

1 Scope

This part of ISO 21501 describes a calibration and verification method for a light scattering airborne particle counter (LSAPC), which is used to measure the size and particle number concentration of particles suspended in air. The light scattering method described in this part of ISO 21501 is based on single particle measurements. The typical size range of particles measured by this method is between 0,1 µm and 10 µm in particle size.

Instruments that conform to this part of ISO 21501 are used for the classification of air cleanliness in cleanrooms and associated controlled environments in accordance with ISO 14644-1, as well as the measurement of number and size distribution of particles in various environments.

The following are within the scope of this part of ISO 21501:

— size calibration;

— verification of size setting;

— counting efficiency;

— size resolution;

— false count rate;

— maximum particle number concentration;

— sampling flow rate;

— sampling time;

— sampling volume;

— calibration interval;

— test report.

2 Terms and definitions

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

2.1
calibration particles
mono-disperse spherical particle with a known mean particle size, e.g. polystyrene latex (PSL) particle, that is traceable to an international standard of length, and where the standard uncertainty of the mean particle size is equal to or less than ± 2,5 %

NOTE The refractive index of calibration particles is close to 1,59 at a wavelength of 589 nm (sodium D line).

2.2
counting efficiency
ratio of the measured result of a light scattering airborne particle counter (LSAPC) to that of a reference instrument using the same sample

2.3
particle counter
instrument that counts the number of particles and measures their size using the light scattering method or the light extinction method

2.4
pulse height analyser
PHA
instrument that analyses the distribution of pulse heights

2.5
size resolution
measure of the ability of an instrument to distinguish between particles of different sizes

3 Requirements

3.1 Size calibration

The recommended procedure for size calibration is described in 4.1.

3.2 Verification of size setting

The error in the detectable minimum particle size and other sizes specified by the manufacturer of an LSAPC shall be equal to or less than ± 10 % when the test is carried out by the method described in 4.2.

3.3 Counting efficiency

The counting efficiency shall be (50 ± 20) % for calibration particles with a size close to the minimum detectable size, and it shall be (100 ± 10) % for calibration particles with a size of 1,5 times to 2 times larger than the minimum detectable particle size.

3.4 Size resolution

The size resolution shall be equal to or less than 15 % for calibration particles of a size specified by the manufacturer.

3.5 False count rate

The false count rate is determined by measuring the particle number concentration in the unit of counts per cubic meter at the minimum reported size range when sampling clean air.

3.6 Maximum particle number concentration

The maximum measurable particle number concentration shall be specified by the manufacturer. The coincidence loss at the maximum particle number concentration of an LSAPC shall be equal to or less than 10 %.

NOTE When the particle number concentration is higher than the maximum particle number concentration, the number of uncounted particles increases because of an enhanced probability of multiple particles existing in the sensing volume (coincidence error) and/or saturation of the electronic system.

3.7 Sampling flow rate

The standard uncertainty of volumetric flow rate shall be equal to or less than ± 5 %.

If the LSAPC does not have a flow rate control system this subclause does not apply, however the manufacturer shall specify the allowable limit of its flow rate of the LSAPC.

3.8 Sampling time

The standard uncertainty in the duration of sampling time shall be equal to or less than ± 1 % of the preset value.

If the LSAPC does not have a sampling time control system, this subclause does not apply.

3.9 Response rate

The response rate of the LSAPC obtained according to the test method given in 4.9 shall be equal to or less than 0,5 %.

3.10 Calibration interval

It is recommended that the calibration interval of an LSAPC be one year or less.

3.11 Test report

The following minimum information shall be recorded:

a) date of calibration;

b) calibration particle sizes;

c) flow rate;

d) size resolution (with the particle size used);

e) counting efficiency;

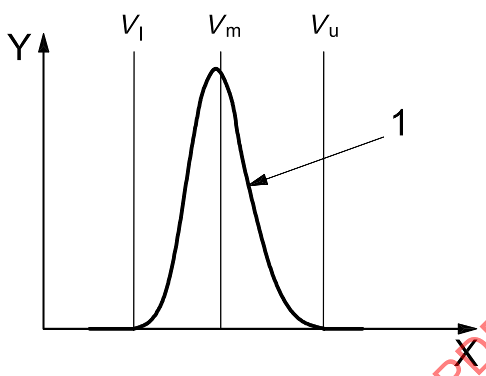
f) false count rate;

g) voltage limit or channel of an internal pulse height analyser (PHA).

4 Test method

4.1 Size calibration

When calibrating an LSAPC with calibration particles of known size, the median voltage (or internal PHA channel), corresponds to the particle size (see Figure 1). The median voltage (or internal PHA channel) should be determined by using a particle counter with variable voltage limit (or internal PHA channel) settings. The median voltage (or internal PHA channel) is the voltage (or internal PHA channel) that equally divides the total number of pulses counted. When a particle counter with variable voltage limit settings is not available, a PHA can be used in place of the counter.



Key

X pulse height voltage (or channel)

Y density

1 pulse height distribution with PSL particles

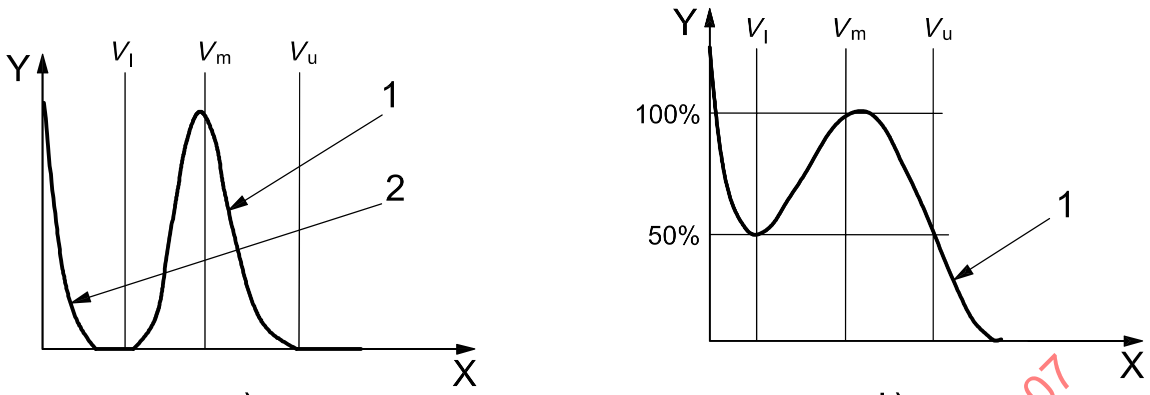
V_l lower voltage limit

V_m median voltage

V_u upper voltage limit

Figure 1 — Pulse height distribution of PSL particle signals

When noise signals appear as if there are many small particles in the sample, the median voltage (or internal PHA channel) shall be determined by discarding the pulses due to “false particles” [see Figure 2 a)]. The discarding should only be done when the density at the peak due to real particles is more than twice the density at the valley that separates it from the pulses due to “false particles” [see Figure 2 b)]. In this case, V_u is the voltage greater than the median voltage, V_m, where the density is the same as V_l. The median is calculated using only the population between the voltage limits V_l and V_u.



a) b)

Key

X pulse height voltage (or channel)

Y density

1 pulse height distribution with PSL particles

2 noise (false particles, small particles and/or optical, electrical noise)

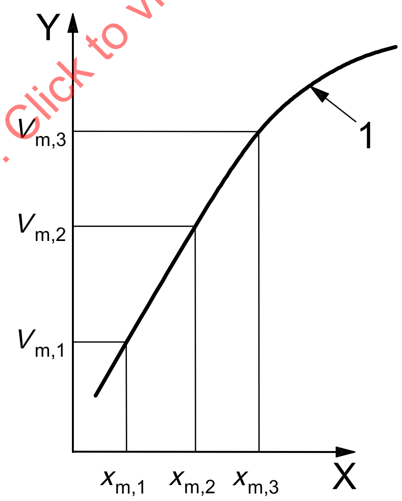
V_l lower voltage limit

V_m median voltage

V_u upper voltage limit

Figure 2 — Pulse height distribution of PSL particle signals with noise

The voltages of channels corresponding to particle size should be determined in accordance with the calibration curve provided by the manufacturer (see Figure 3).



Key

X particle size

Y median value of calibration particles

1 calibration curve

$V_{m,1}$ median voltage corresponding to particle size $x_{m,1}$

$V_{m,2}$ median voltage corresponding to particle size $x_{m,2}$

$V_{m,3}$ median voltage corresponding to particle size $x_{m,3}$

Figure 3 — Calibration curve

NOTE When the median voltage is determined by using an external PHA, the uncertainty in the voltage of PHA and the voltage uncertainty of the LSAPC are included in setting the voltage limits of the LSAPC (see Annex A).

4.2 Verification of size setting

Obtain response voltages (or internal PHA channel) in accordance with the test method given in 4.1, using at least three kinds of calibration particles that span most of the reported size range, x_r, of the LSAPC. Determine the calibration curve from these response voltages (or internal PHA channel) and the calibration particle sizes. Calculate the corresponding particle size, x_s, from the voltage (or internal PHA channel) setting of the LSAPC using the calibration curve. Obtain the size setting error, ε, by means of Equation (1) below, and examine whether it satisfies the requirement given in 3.2.

NOTE It is desirable that the manufacturer defines the method used to draw the calibration curve.

$$\varepsilon(\%) = \frac{x_s - x_r}{x_r} \times 100\ \% \quad (1)$$

where

ε is the size setting error, in %;

x_r is the reported size range, in µm;

x_s is the calculated particle size, in µm.

4.3 Counting efficiency

To test the counting efficiency of the LSAPC, use calibration particles with two sizes: one that is close to the minimum detectable reported size range, and another that is 1,5 times to 2 times larger than the minimum detectable size.

Measure the particle number concentration of both particles with the LSAPC under test and either a condensation particle counter (CPC) or a calibrated LSAPC as a reference instrument (see Annex B).

NOTE A condensation particle counter is also referred to as a condensation nucleus counter (CNC).

The counting efficiency is the ratio of the particle number concentration measured by the LSAPC under test and the particle number concentration measured by the reference instrument.

For these measurements, the particle number concentration of the test sample should be equal to or less than 25 % of the maximum particle number concentration of both the LSAPC under test and the reference instrument.

4.4 Size resolution

The manufacturer's recommended calibration particle size should be used for this test. The standard deviation of the calibration particles should be a known quantity, σ_P. Determine the median voltage (or channel), V_m, using calibration particles, as shown in Figure 4.

The lower voltage limit, V_l, and upper voltage limit, V_u, are defined as those corresponding to a density of 61 %. Using the calibration curve, determine the particle sizes corresponding to V_l and V_u. Calculate the absolute value of the differences in particle size between PSL particle size and particle size corresponding to V_l and V_u The greater of these is the observed standard deviation, σ. Calculate the percentage of size resolution, R, of the LSAPC by Equation (2) below (see also Annex C).

$$R(\%) = \frac{\sqrt{\sigma^2 - \sigma_P{}^2}}{x_P} \times 100\ \% \quad (2)$$

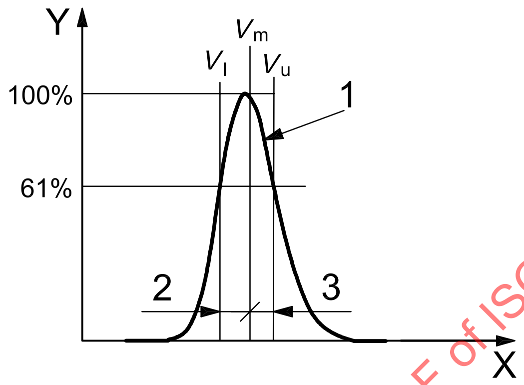
where

R is the size resolution, in %;

σ is the observed standard deviation of LSAPC, in μm;

σ_P is the supplier's reported standard deviation of calibration particles, in μm;

x_P is the particle size of the calibration particle, in μm.



Key

X pulse height voltage (or channel)
Y density

1 pulse height distribution with PSL particles
2 lower side resolution
3 upper side resolution

V_l lower voltage limit
V_m median voltage
V_u upper voltage limit

Figure 4 — Size resolution

4.5 False count rate

The false count rate is the measured particle number concentration (in particles per cubic meter) when the LSAPC is set to the minimum detectable size and particle free air flows to the LSAPC. The data should be statistically processed using the Poisson distribution with a 95 % upper confidence limit (see Annex D). The false count rate shall be described in units of particle number concentration (in particles per cubic meter).

4.6 Maximum particle number concentration

The coincidence loss is determined by the flow rate, the time required for particles to pass through the sensing zone and the electrical signal processing time. These values are determined by the design of the LSAPC. Coincidence loss is calculated as in Equation (3) below.

$$L(\%) = \left[1 - \exp\left(-q \cdot t \cdot C_{max}\right)\right] \times 100\ \% \quad (3)$$

where

L is the coincidence loss, in %;

q is the flow rate, in m^3/s;

t is the time of passing through the sensing region plus electrical processing time, in s;

C_{max} is the maximum particle number concentration, in particles per cubic metre.

NOTE If the particle number concentration becomes excessive, the coincidence error increases. This means several small particles are measured as one large particle.

4.7 Sampling flow rate

The volumetric flow rate should be measured using a calibrated soap bubble film flow meter, a wet gas meter, or another type of flow meter that has a low pressure drop. The flow rate measured is the volumetric flow rate. Examine whether the flow rate standard uncertainty satisfies the requirement given in 3.7. When using a mass flow meter, the flow rate should be converted to a volumetric flow rate. If the LSAPC does not have a flow rate control function, this subclause does not apply.

4.8 Sampling time

Sampling time is the time during which the LSAPC measures a sample (from the beginning of counting to the end of counting).

The sampling time tolerance is one minus the ratio of the measured sampling time, t, to the instrument's specified sampling time, t_0. This is shown as $1-\frac{t}{t_0}$.

Examine whether the sampling time tolerance satisfies the requirement given in 3.8. Calibrated instruments should be used for sampling time measurement.

If the LSAPC does not have a sampling time control function, this subclause does not apply.

4.9 Response rate

Set the reported size range of LSAPC to the minimum detectable particle size. Begin the test by sampling air containing PSL particles near the maximum rated particle number concentration for 10 min. The PSL particle size is near the minimum detectable size.

Measure the particle number concentration for a period of T s. Change over to clean air. After 10 s measure the particle number concentration for T s. Calculate the ratio of the particle number concentration obtained before and after the change of the air. Examine whether the ratio complies with the specification given in 3.9. The measurement duration, T, shall be 60 s or less, and the counts shall measure at least 1 000 counts (see Annex E).

4.10 Calibration

Calibration at the calibration interval (see 3.10) should include at least size calibration, size resolution, counting efficiency and sampling volume uncertainty. If the LSAPC does not have a flow rate control function, the standard uncertainty of sampling flow rate does not apply.

Annex A
(informative)

Uncertainty of particle size calibration

A.1 Size calibration using external and internal PHA

Figure A.1 shows the particle size calibration using an external PHA and a voltmeter. In this case, there are four sources of uncertainty:

— PSL particles,

— PHA,

— voltmeter, and

— offset voltage at the size setting circuit.

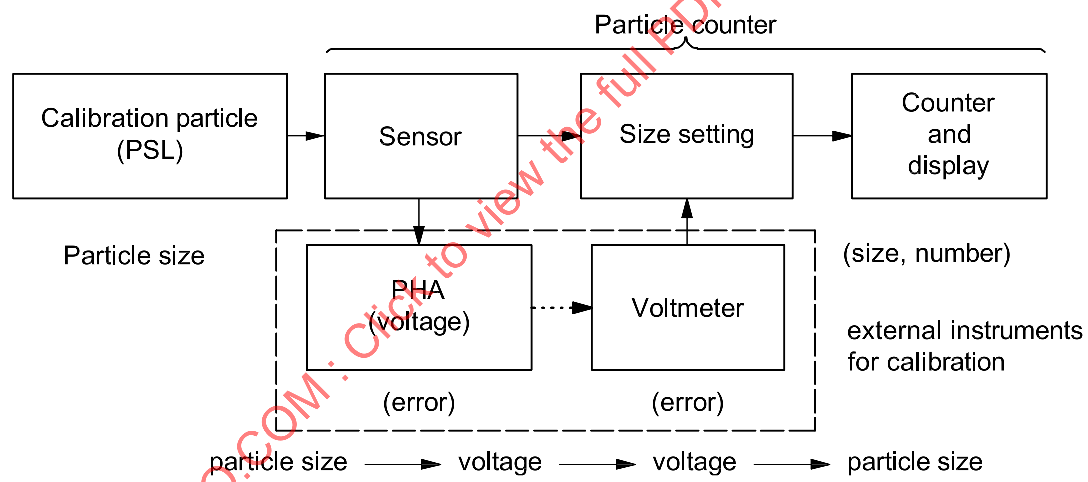


Figure A.1 — Particle size calibration using external instruments (PHA and voltmeter)

However, in Figure A.2, the uncertainty of particle size calibration depends only on the PSL particle size uncertainty.

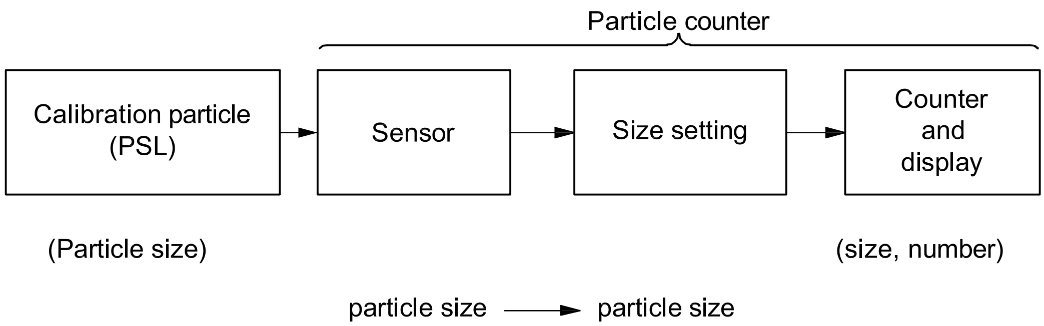


Figure A.2 — Particle size calibration using an internal PHA

A.2 Uncertainty of size calibration

Tables A.1 and A.2 show examples of uncertainty of size calibration. Table A.1 shows an example of combined standard uncertainty for size calibration using an external PHA and voltmeter. Table A.2 shows an example of combined standard uncertainty for size calibration using an internal PHA. The combined standard uncertainty for size calibration using an internal PHA is smaller than when using an external PHA.

Table A.1 — Relative standard uncertainty of size calibration using an external PHA (for example)

Items	Standard uncertainty %
PSL particles	2,5
PHA	2,5
Voltmeter	0,1
Offset voltage	0,5
Calibration curve	1,5
Combined standard uncertainty	3,9
Expanded uncertainty (k=2)	7,8
NOTE The standard uncertainty of the calibration curve is the uncertainty in the relationship between particle size and voltage limit or internal PHA channel.	

Table A.2 — Relative standard uncertainty of size calibration using an internal PHA (for example)

Items	Standard uncertainty %
PSL particles	2,5
Calibration curve	1,5
Combined standard uncertainty	2,9
Expanded uncertainty (k=2)	5,8
NOTE The standard uncertainty of the calibration curve is the uncertainty in the relationship between particle size and voltage limit or internal PHA channel.	

Annex B
(informative)

Counting efficiency

Figure B.1 shows the test system for counting efficiency. The particle generator generates air that contains PSL particles suspended in clean air. The counting efficiency of the reference particle counter at the minimum detectable particle size is known.

The counting efficiency is obtained by calculating the ratio of the particle number concentration measured by the particle counter under test and the particle number concentration measured by the reference particle counter. The particle number concentration of the sample should be less than 25 % of the maximum particle number concentration of both the reference particle counter and the particle counter under test. The counting efficiency of the reference particle counter should be confirmed to be 100 % by a condensation particle counter (CPC), or to be a known value.

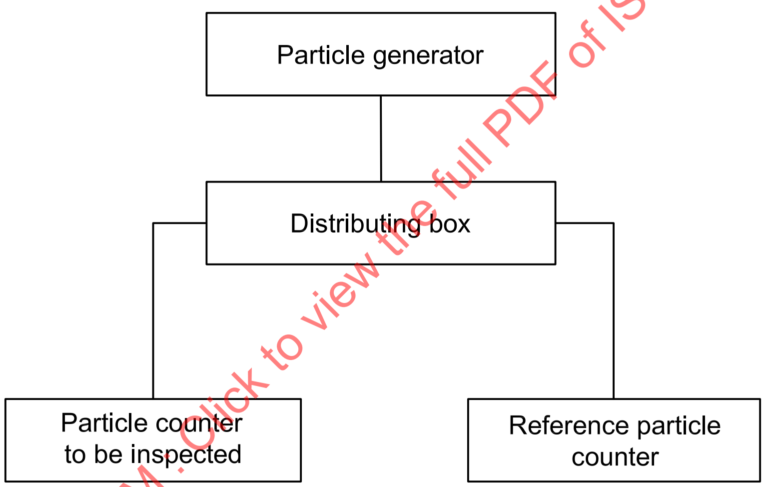


Figure B.1 — Example of a counting efficiency test system

Determine the counting efficiency of η by means of Equation B.1:

$$\eta = \frac{C_1}{C_0} \times 100\ \% \qquad \text{(B.1)}$$

where

η is the counting efficiency, in %;

C_0 is the particle number concentration measured by reference particle counter, in particles per cubic metre;

C_1 is the particle number concentration measured by particle counter under test, in particles per cubic metre.

Annex C
(informative)

Size resolution

Size resolution is defined as one standard deviation of the measured size distribution of monodisperse calibration particles, expressed as a percentage of the mean size of the monodisperse calibration particles.

If the distribution of calibration particles is assumed to be the Gaussian distribution,

$$f(x) = \frac{1}{\sqrt{2\pi}\sigma} \exp\left\{-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^2\right\} \quad \text{(C.1)}$$

where

$f(x)$ is the Gaussian function;

x is the particle size;

μ is the mean value;

σ is the standard deviation.

When $(x-\mu) = \pm\sigma$, the ratio of density to the maximum density is $\exp\left(-\frac{1}{2}\right) \approx 0{,}61$. This is the basis for the use of 61 % in the determination of size resolution.

Annex D
(informative)

False count rate

The probability of appearance of false counts is assumed to be defined by the Poisson distribution. The Poisson distribution is defined by Equation (D.1):

$$P(X:\lambda) = \frac{e^{-\lambda}\lambda^X}{X!} \qquad (D.1)$$

where

X is the number of false counts;

λ is the mean value of the population;

$P(X:\lambda)$ is the probability of observing value X from a population having a mean value of λ.

The lower confidence limit, λ_l, is defined by Equation (D.2):

$$\sum_{X=X}^{\infty} P(X;\lambda_l) = \frac{\varepsilon}{2} \qquad (D.2)$$

where

ε is the size setting error.

The upper confidence limit, λ_u, is defined by Equation (D.3):

$$\sum_{X=0}^{X} P(X;\lambda_u) = \frac{\varepsilon}{2} \qquad (D.3)$$

When the confidence limit is 95 %, ε is 0,05.

Table D.1 shows the observed count and the calculated upper and lower 95 % confidence limits. When the observed count is zero, it is possible to have up to three counts with a probability of 5 %. For example, if zero counts are observed in 15 min at the sample flow rate of 28,3 l/min, the false count rate is three counts in the volume sampled in 15 min with a 95 % confidence limit, i.e. the false count rate is 7 counts per cubic meter.

