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

Part 2:
Light scattering liquid-borne particle counter

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

Partie 2: Compteur de particules en suspension dans un liquide en lumière dispersée

Reference number
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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.

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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.

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ISO 21501-2 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 21501-2, together with ISO 21501-3 and ISO 21501-4, 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 liquid. 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 2:
Light scattering liquid-borne particle counter

1 Scope

This part of ISO 21501 describes a calibration and verification method for a light scattering liquid-borne particle counter (LSLPC), which is used to measure the size and particle number concentration of particles suspended in liquid. 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 evaluation of the cleanliness of pure water and chemicals, as well as the measurement of number and size distribution of particles in various liquids. The measured particle size using the LSLPC depends on the refractive index of particles and medium; therefore the measured particle size is equivalent to the calibration particles in pure water.

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 particle
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 liquid-borne particle counter (LSLPC) 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 the 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 LSLPC shall be equal to or less than ± 15 % when the test is carried out by the method described in 4.2.

3.3 Counting efficiency

The counting efficiency shall be (50 ± 30) % for calibration particles with a size close to the minimum detectable size, and it shall be (100 ± 30) % for calibration particles with the particle size of 1,5 times to 3 times larger than the minimum detectable particle size.

3.4 Size resolution

The size resolution shall be equal to or less than 10 % 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 litre at the minimum reported size range when sampling pure water.

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 LSLPC 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 the sampling flow rate shall be specified by the manufacturer. The user shall check that the sampling flow rate is within the range specified by the manufacturer.

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 LSLPC does not have a sampling time control system, this subclause does not apply.

3.9 Sampling volume

The standard uncertainty of sampling volume shall be equal to or less than ± 5 % of the preset value.

This subclause does not apply when the LSLPC is not equipped with a sampling system.

3.10 Calibration interval

It is recommended that the calibration interval of an LSLPC 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);

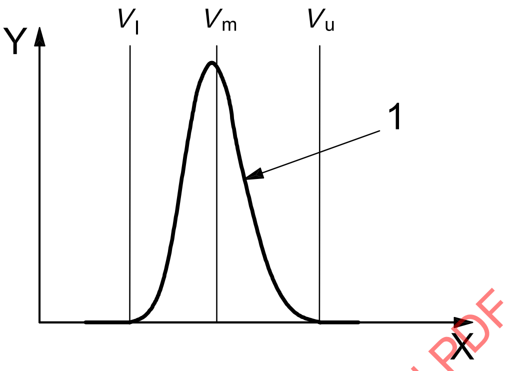
e) counting efficiency;

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

4 Test method

4.1 Size calibration

When calibrating an LSLPC 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

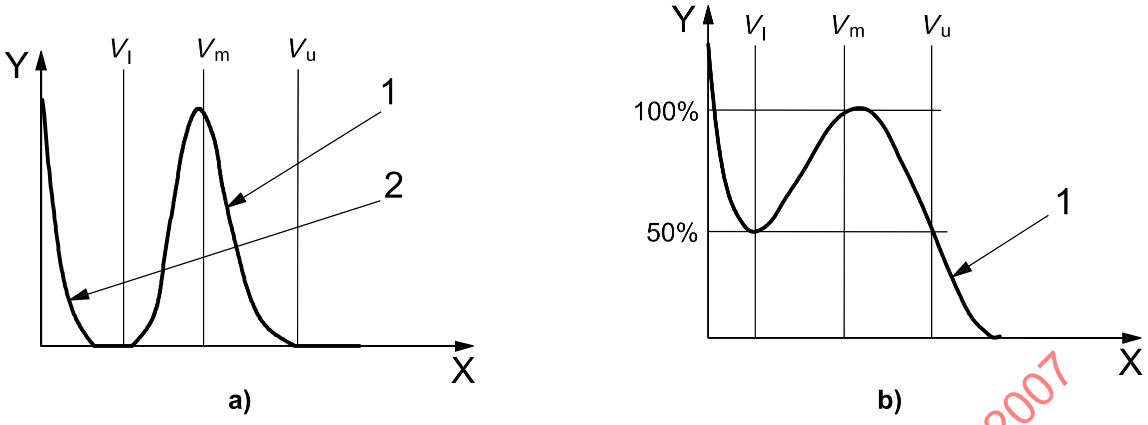
X pulse height voltage (or channel)
Y density

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.



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)

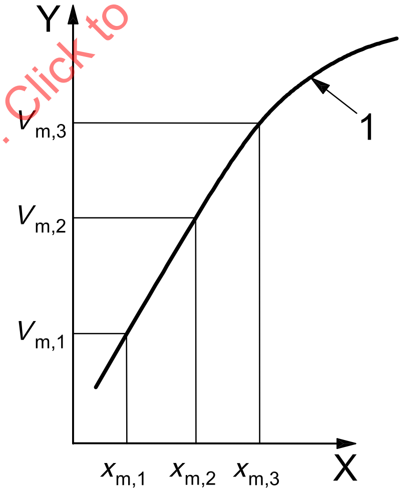
V_l lower voltage limit

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 LSLPC are included in setting the voltage limits of the LSLPC (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 LSLPC. 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 LSLPC 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.

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

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 LSLPC, 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 3 times larger than the minimum detectable size.

Measure the particle number concentration of both particles with the LSLPC under test and either a microscopic method or a calibrated LSLPC as a reference instrument.

The counting efficiency is the ratio of the particle number concentration measured by the LSLPC under test and the particle number concentration measured by the reference instrument (see Annex B).

4.4 Size resolution

Determine the median voltage (or channel), V_m, using monodisperse 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 LSLPC by Equation (2) below (see also Annex C).

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

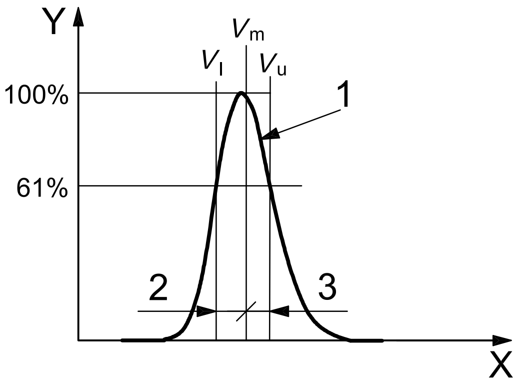
where

R is the size resolution, in %;

σ is the observed standard deviation of LSLPC, 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 litre) when the LSLPC is set to the minimum detectable size and particle free liquid flows to the LSLPC. 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 counts per litre).

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 LSLPC. Coincidence loss is calculated as in Equation (3) below.

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

where

L is the coincidence loss, in %;

q is the flow rate, in cm^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 centimetre.

4.7 Sampling flow rate

Obtain a flow rate by the sampling volume (see 4.9) and the sampling time (see 4.8), or use a calibrated flow meter. If the LSLPC does not have a sampling function, this subclause does not apply.

4.8 Sampling time

Sampling time is the time during which the LSLPC 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.

This subclause does not apply when the LSLPC is not equipped with a sampling system.

This subclause does not apply when the LSLPC is equipped with a volumetric sampling system.

4.9 Sampling volume

Measure the sampling volume by weighing the pure water with the balance and converting to volume, or measure the volume by means of a calibrated graduated cylinder.

If the LSLPC does not have a sampling function, this subclause does not apply.

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 LSLPC does not have a 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.

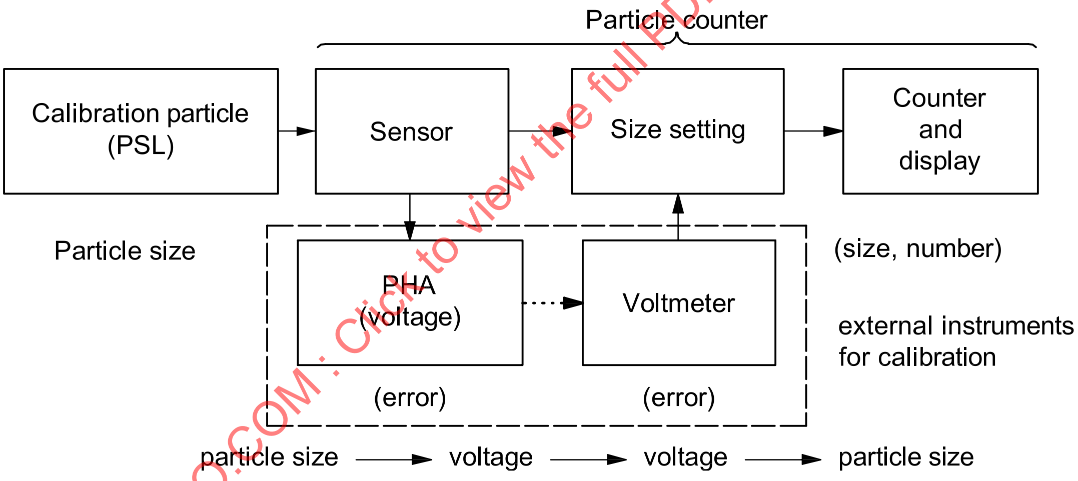


Figure A.1 — Particle size calibration using external instruments (PHA and voltmeter)

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

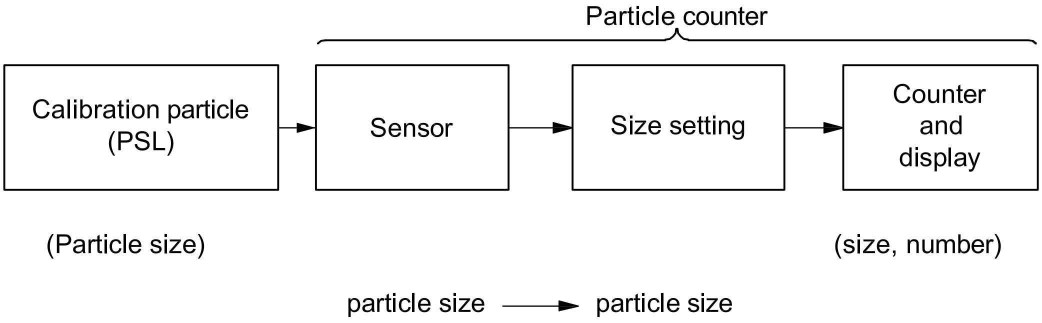


Figure A.2 — Particle size calibration using an internal PHA

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 sample contains calibration particles in pure water. The counting efficiency of the reference particle counter at the minimum detectable particle size of the particle counter under test shall be 100 %.

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 shall be established by a method of known uncertainty, such as the microscopic method (see method described in JIS B 9925 [5]).

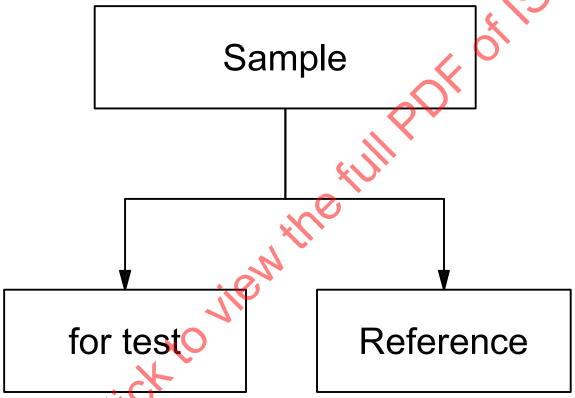


Figure B.1 — Example of a counting efficiency test system

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 litre;

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