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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

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 24., *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

This second edition cancels and replaces the first edition (ISO 21501-2:2007), which has been technically revised. The main changes from the previous edition are as follows:

— Clause 4 for “Principle” and Clause 5 for “Basic configuration” have been added;

— “size calibration” and “verification of size setting” have been combined as “size setting error” in the requirements (Clause 6);

— “Test report” (3.11 in the previous edition) has been changed to 6.10 on “Reporting of test and calibration results”;

— information about uncertainties has been enriched and is now the subject of Annex D.

A list of all parts in the ISO 21501 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 document 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 document 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 document 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.

The method is applicable to instruments 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 document:

- size setting error;
- counting efficiency;
- size resolution;
- false count;
- maximum particle number concentration;
- sampling flow rate error;
- sampling time error;
- sampling volume error;
- calibration interval;
- reporting results from test and calibration.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
calibration particles
monodisperse spherical particles with a known mean particle size, e.g. polystyrene latex (PSL) particles, where the certified size is traceable to the International System of Units (SI), a relative standard uncertainty equal to or less than 2,5 %, and a refractive index that is approximately 1,59 at the wavelength of 589 nm (sodium D line)

Note 1 to entry: For spherical particles, the particle size is equal to the diameter.

3.2
counting efficiency
ratio of the number concentration measured by a *light scattering liquid-borne particle counter* (3.4) to that measured by a reference instrument for the same sample

3.3
false count
apparent count per unit volume of sample liquid when a sample liquid containing no measurable particles is measured by the *light scattering liquid-borne particle counter* (3.4)

3.4
LSLPC
light scattering liquid-borne particle counter
instrument that measures liquid-borne particle numbers by counting the pulses as the particles pass through the sensing volume, as well as particle size by scattered light intensity

Note 1 to entry: The optical particle size measured by the LSLPC is the light scattering equivalent particle size and not the geometrical size.

3.5
PHA
pulse height analyser
instrument that analyses the distribution of pulse heights

3.6
size resolution
measure of the ability of an instrument to distinguish between particles of different sizes

3.7
coincidence loss
reduction of particle count caused by multiple particles passing simultaneously through the sensing volume and/or by the finite processing time of the electronic system

3.8
MPE
maximum permissible error
limit of error
extreme value of measurement error, with respect to a known reference quantity value, permitted by specifications for a given measurement, measuring instrument, or measuring system

Note 1 to entry: This document uses decimal numbers for the requirements to MPEs to avoid confusions that may arise when relative uncertainties of test results are reported in percent figures.

4 Principle

The measurement principle of the LSLPC is based on detection of light scattered by a particle when the particle passes through an incident light beam.

The particle size is determined from the intensity of the scattered light, and the number of particles from the number of light pulses scattered by individual particles.

More specifically, a sample liquid is drawn from the inlet of the LSLPC at a constant flow rate, and introduced to the sensing volume of the LSLPC where a light beam is irradiated. When a particle suspended in the sample liquid passes through the light beam, it scatters the light, emitting a light pulse. The light pulse is detected by a photo detector and converted to an electrical pulse. The electrical pulse height is proportional to the scattered light intensity, and depends on the optical system design, the electronic components used, and the light source. The intensity of the scattered light is dependent on the size, refractive index and shape of the particle. If the particle is spherical, the scattered light intensity is described by the Mie theory. In order to establish a relationship between the electrical pulse height and the particle size, calibration of each LSLPC with use of particles having a well-defined size, refractive index, and shape is required.

5 Basic configuration

An LSLPC is composed typically of a light source, a sample liquid supply/suction system, a sensing volume, a photoelectric conversion device, a pulse height analyser, and a display (see Figure 1). Some LSLPCs do not contain a sample liquid supply/suction system and/or a display.

To make the particle size calibration possible, the LSLPC should be constructed so that pulse height distributions for calibration particles can be measured.

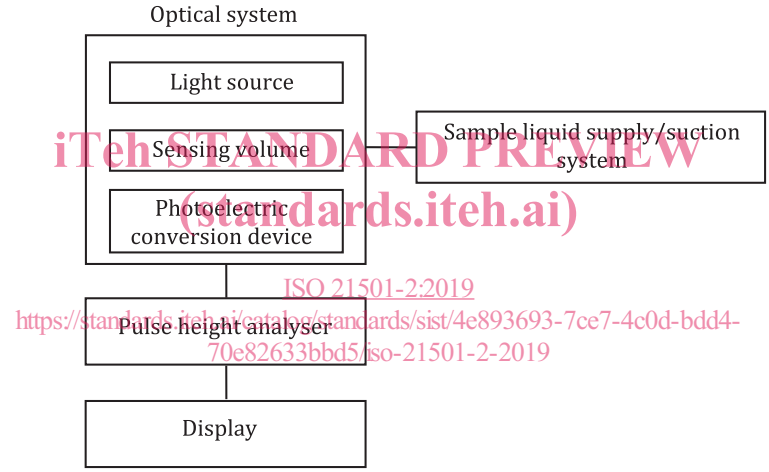

Figure 1 — Example of basic configuration of LSLPC

6 Requirements

6.1 Size setting error

The MPE for size setting in the minimum detectable particle size and other sizes specified by the manufacturer of an LSLPC is 0,15 (corresponding to 15 % of the specified size).

Size setting shall be conducted before the LSLPC is shipped from the manufacturer, and when the size setting error is found not fulfilled in a periodic calibration.

A recommended procedure for size setting is described in 7.1.2. If other methods are used, their uncertainty shall be evaluated and described.

6.2 Counting efficiency

The counting efficiency shall be within 0,20 to 0,80 [corresponding to (50 ± 30) %] for calibration particles with a size close to the minimum detectable size, and it shall be within 0,70 to 1,30 [(100 ± 30) %] for calibration particles with the particle size 1,5 to 3 times larger than the minimum detectable particle size.

When calibration particles with exactly the same size as the minimum detectable particle size are not available, particles whose size is within ±5 % of the minimum detectable particle size may be used and the diameter of the calibration particles shall be reported.

6.3 Size resolution

The size resolution shall be less than or equal to 0,10 (corresponding to 10 % of the specified particle size), when it is evaluated using calibration particles of a certified average size specified by the manufacturer.

A recommended procedure is described in 7.3. If other methods are used, their uncertainty shall be evaluated and described.

6.4 False count

The false count per volume in litre and its 95 % upper confidence limit (UCL) shall be determined according to 7.4. The 95 % UCL shall be less than or equal to the value specified and reported by the manufacturer of the LSLPC.

6.5 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 less than or equal to 0,1 (corresponding to 10 %).

NOTE The probability of occurrence of coincidence loss increases with increasing particle number concentration.

6.6 Sampling flow rate error

The MPE 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.

6.7 Sampling time error

The MPE in the duration of sampling time shall be 0,01 (corresponding to 1 %) of the preset value.

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

6.8 Sampling volume error

The MPE of sampling volume shall be 0,05 (corresponding to 5 %) of the preset value.

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

6.9 Calibration interval

The calibration of the LSLPC should be conducted at an interval equal to or shorter than one year. The requirements should be met during the calibration interval.

6.10 Reporting of test and calibration results

The report shall contain at least the following information:

a) date of test/calibration;

b) test/calibration particles used;

c) results for the parameters:

1) size setting error;

2) counting efficiency;

3) sampling flow rate error;

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

d) threshold voltage values or channel of the built-in PHA corresponding to the size settings;

e) reference of the test/calibration method used (i.e. ISO 21501-2).

f) report/certificate identification, test/calibration location, title and identification of test/calibration provider including signature and date;

g) identification of customer and device under test, including how output was obtained for counting efficiency (e.g. analogue, display or digital output).

A calibration certificate shall furthermore include:

h) identification and — if possible — statement of metrological traceability of all reference equipment and calibration particles used;

i) relevant environmental conditions (e.g. temperature, air pressure and humidity) under which the calibration was performed;

j) a stated uncertainty for each result for the parameters 1 to 2 with reference to the calculation method (e.g. ISO/IEC Guide 98-3) — Annex D gives a recommended procedure for evaluating the uncertainty of the results of the performance tests.

k) a stated false count at a 95 % confidence limit (see Annex C).

NOTE Calibration certificates issued by ISO/IEC 17025 accredited laboratories and covering all results for the parameters 1 to 2 are considered to comply with the requirements above.

7 Test and calibration procedures

7.1 Size setting

7.1.1 Evaluation of size setting error

Calculate the size setting error, ε, according to Formula (1).

$$\varepsilon = \frac{x_i' - x_i}{x_i} \tag{1}$$

where

x_i is the size setting specified for the LSLPC;

x_i' is the actual size setting corresponding to V_{ti} (see 7.1.2 for the meaning of V_{ti}).

7.1.2 Procedure of size setting

By use of a PHA connected to the output terminal for signal pulses of the LSLPC, or by use of a built-in PHA if one is contained as a part of the LSLPC, obtain a pulse height distribution for a sample liquid in which calibration particles are suspended. Let V_l and V_u denote the lower and upper voltage limits, respectively, of the range of pulse heights for the calibration particles (see Figure 2). The median voltage