INTERNATIONAL STANDARD

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AMENDMENT 1
2023-02

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

Part 4: Light scattering airborne particle counter for clean spaces

AMENDMENT 1

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

AMENDEMENT 1

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

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

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

Part 4: Light scattering airborne particle counter for clean spaces

AMENDMENT 1

3.2

Add the following text at the end of the definition: "or ratio of the particle number measured by an LSAPC to that introduced to the LSAPC for a given sampling time", so that the entry reads:

3.2
counting efficiency
ratio of the number concentration measured by a light scattering airborne particle counter (LSAPC) (3.4) to that measured by a reference instrument for the same test aerosol, or ratio of the particle number measured by an LSAPC to that introduced to the LSAPC for a given sampling time

6.2

Add the following paragraph at the end of the subclause:

> It can be appropriate to evaluate counting efficiency for some applications at sizes larger than twice the minimum detectable size. It is recognized that the counting efficiency range of 0,90 to 1,10 [(100 ± 10) %] specified above does not remain relevant at all larger sizes due to particle losses within the LSAPC; depending on the application requirements, a tolerance of ±10 to ±30 % is recommended at a nominal particle diameter of 5 µm.

7.2

Add the following subclause heading above the first paragraph:

> **7.2.1 Parallel comparison method**

Add the following subclause at the end of subclause 7.2.1:

> **7.2.2 Generator method**
>
> Clause A.2 describes the generator method for evaluating the counting efficiency of LSAPC. Generator method uses monodisperse particles whose sizes are defined as the volume equivalent diameter. The method uses an inkjet aerosol generator (IAG) as a monodisperse particle number standard. In this method, the counting efficiency, η, is evaluated according to Formulae (3) and (4).

$$\eta = \frac{N_1}{N_0} \tag{3}$$

$$N_0 = t \cdot L_0 \tag{4}$$

where

N_1 is the number of particles measured by an LSAPC under test;

N_0 is the number of particles introduced to the LSAPC;

t is the sampling time set to the LSAPC;

L_0 is the particle generation rate of the IAG.

The counting efficiencies of Formula (2) is equivalent to Formula (3) when N_0 is evaluated by

$$N_0 = V \cdot C_0$$

where V is the volume of test aerosol sampled by the LSAPC.

Renumber subsequent Formulae (3) to (7) as (5) to (8).

Annex A

Replace Annex A with the following:

Annex A
(informative)

Counting efficiency

A.1 Introduction

This annex introduces the parallel comparison method and the generator method. The parallel comparison method is the general method, and the generator method is the alternative method. Table A.1 summarizes the characteristics of these two methods.

Table A.1 — Characteristics of the parallel comparison method and the generator method

Parallel comparison method	Generator method
Liquid or solid particles nebulised from solutions/particle suspensions or dispersed from dry powder; PSL spheres can be used as test particles.	Monodisperse solid or liquid particles are generated from aqueous solutions; PSL spheres cannot be used as test particles.
Particle size range: typically from 100 nm PSL optical diameter.	Particle size range: typically from 0,5 µm.
Since the method can select the particle size by using classification devices such as DEMC or AAC, the cut-off region of the counting efficiency curve can be evaluated.	Not an appropriate method to evaluate the lower cut-off diameter of the counting efficiency curve.
SI-traceability of the PSL geometric diameter can be established.	SI-traceability of the particle volume equivalent diameter can be established.
The number of particles delivered to a DUT-LSAPC must be measured with a reference instrument (e.g. a reference LSAPC or CPC).	The number of particles delivered to a DUT- LSAPC is accurately and precisely known.

A.2 Parallel comparison method

A.2.1 Principle

Figures A.1 and A.2 show the test system for counting efficiency. The particle generator generates an aerosol that consists of dry monodisperse PSL particles (100 nm to 10 µm) suspended in clean air.

PSL particles in the range of 100 nm to 5 µm can be generated by nebulizing aqueous suspensions. After nebulization of a PSL suspension, the aerosol typically contains residue particles which can bias the measurement of the counting efficiency. Measurement errors should be minimized by:

— separating the PSL particles from surfactants, for example, in several mixing/settling separation steps in ultrapure water before preparing the suspension for the aerosol generator;

— using a PSL suspension in the aerosol generator with very low concentration of impurities in the liquid phase, for example, traces of salt in ultrapure water, to a) achieve a low enough background of residue particles and b) avoid growth of PSL particles due to coating of impurities after evaporation of the suspension liquid droplet;

— optimising the concentration of PSL particles in the suspension to avoid measurement bias due to doublet PSL particles (two PSL particles were contained in a droplet);

— drying the aerosol to remove all suspension liquid from the surface of the PSL particles and to avoid condensation of suspension liquid vapour on the PSL particles.

After drying the aerosol, size classifying the PSL particles with a DEMC (compare ISO 15900 and ISO 27891; commercial DEMCs can be used for particles up to about 1 µm) or an aerodynamic aerosol classifier[11] (AAC), applicable up to 5 µm, can be applied if the background of residue particles needs to be further reduced. This can especially be necessary if the requirements in Clause 7 (see Figure 3) cannot be fulfilled.

Since PSL aerosol generated from a suspension is electrostatically charged and since DEMC-classified PSL particles are unipolarly charged, a bipolar diffusion charge conditioner (as known as aerosol neutralizer) further increases the accuracy of the measurement of the counting efficiency by minimizing particle losses in both the particle counter to be inspected and the reference particle counter.

After generation and conditioning, the PSL aerosol is fed to the particle counter to be inspected and the reference particle counter via a device (e.g. a distributing box, see Figures A.1 and A.2) which shall be designed in such a way that the particle number concentration at the inlet of both particle counters is as close as possible. The uncertainty associated with the inhomogeneity in the particle number concentration should be evaluated according to the procedure given in Clause E.2 [1].

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.

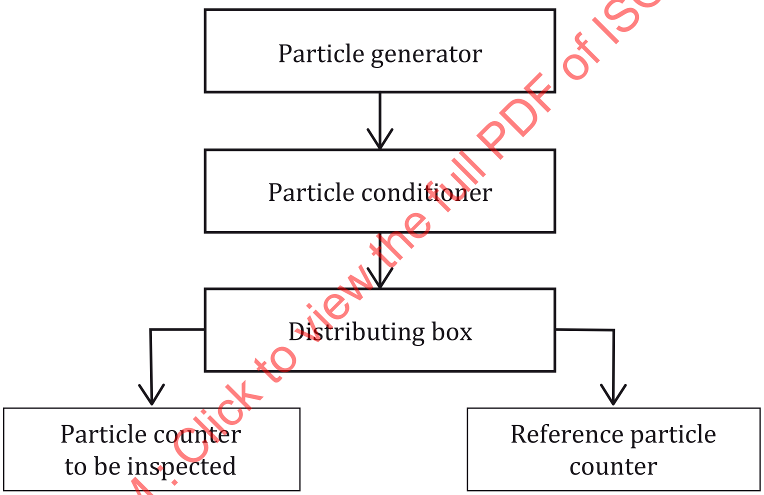
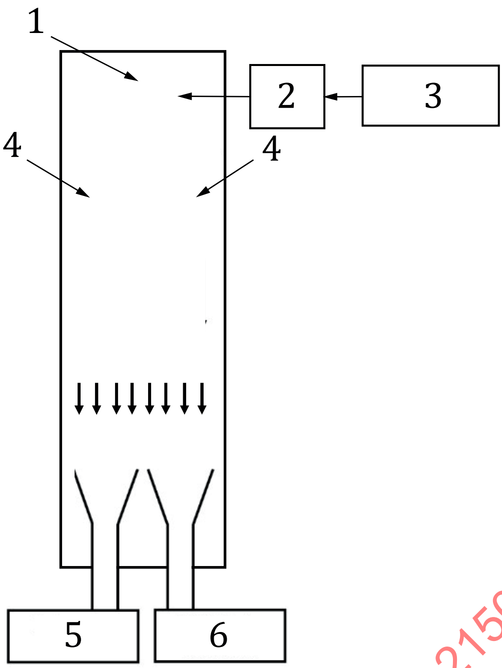


Figure A.1 — Example of counting efficiency test system



Key

1 filtered dilution air

2 DEMC or AAC

3 wet or dry PSL dispersion

4 turbulent airjets

5 reference LSAPC

6 LSAPC under test

Figure A.2 — Example of counting efficiency test system

As mentioned before, the method described above is most useful for PSL particles smaller than approximately 5 µm. If calibration with larger particles (e.g. 10 µm) is required, dry PSL particles generated with a dry powder dispenser are better suited. The counting efficiency of the LSAPC under test can decrease considerably for particles with a diameter larger than 1 µm. The monodisperse, dry PSL powder needs to be free of surfactants to avoid errors during the calibration. Homogenization of large particles (larger than about 0,5 µm) can require mixing by turbulent airjets as shown in Figure A.2. Moreover, distributing the aerosol between the reference particle counter and the particle counter to be inspected in Figure A.1 requires special attention for larger particles since particle losses due to inertial impaction and gravitational settling become important. To minimize errors, it is recommended to:

— use a distribution tube in Figure A.2 instead of a distribution box in Figure A.1;

— use isokinetic and isoaxial probes to extract the calibration aerosol for both particle counters;

— use vertical tubing to connect the distribution tube with the particle counters;

— use a large radius of curvature (radius larger than 10 times the inner diameter of the tube), if bends in the connection tubing cannot be avoided;

— use metallic, grounded tubing with polished inner surface for connection;

— avoid changes in tubing diameter; in particular avoid step changes.

A.2.2 Traceability

A sample traceability chart is shown in Figure A.3. Traceability is provided by calibrating LSAPCs against a reference LSAPC at a National Metrology Institute (NMI). An example of how to put the recommendations of this document into practice is provided in Reference [12]. The reference LSAPC is custom made. The sampled aerosol flow, measured with a traceably calibrated mass flow meter, is typically set to 60 ml min^{-1} to avoid coincidence losses. The sampled aerosol enters the detection chamber through a nozzle with an orifice of 0,2 mm and is surrounded by a sheath-air flow, which prevents the particle beam from diverging. A laser beam is generated by a continuous-wave laser (5 W) at a wavelength of 532 nm and focused at the point of intersection with the aerosol stream using a cylindrical lens. This results in a laser beam with a width of 0,7 mm. Particles cross the laser beam scatter light, which is detected by a photomultiplier tube placed at a 90° angle. The peak detection algorithm has been traceably validated using a pulse generator coupled to a traceable frequency standard.

The reference LSAPC at NMI is further validated through international inter-comparisons.

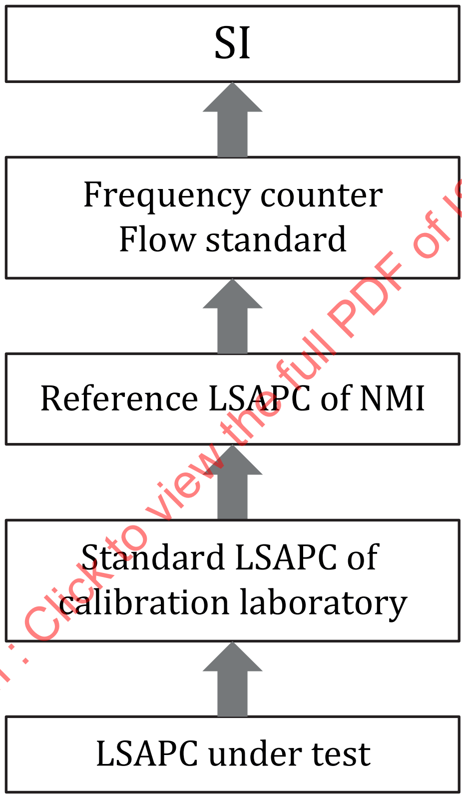


Figure A.3 — Metrological traceability of the particle concentration measured by an LSAPC under test

Traceably calibrated CPCs can also be used as reference counters for particle diameters up to 1 µm.

A.3 Generator method

A.3.1 Principle

The generator method is an alternative method that can be applied over the size range from 0,5 µm to 10 µm. The particle number concentration of the test aerosol depends on the sampling flowrate of an LSAPC since the particle generation rate of the test aerosol is set at the constant value. For example, when the particle generation rate is set to 30 s^{-1} and the flowrate is 2,83 l min^{-1} and 28,3 l min^{-1}, the particle number concentration at the inlet of LSAPC is 0,64 cm^{-3} and 0,064 cm^{-3}, respectively.

Figure A.4 shows the schematic diagram of the generator method. The inkjet aerosol generator (IAG) [13], consists of key items 1 to 5 of Figure A.4. The inkjet head is filled with an aqueous solution of a particle material. The inkjet head generates droplets of the solution at a constant rate. Subsequently, the droplets are electrically neutralized. Droplets are dried as they flow through an evaporation tube which is heated from its outside. Solvent content of the droplets evaporates, and the solute becomes aerosol particles. When an LSAPC is under test samples, the aerosol particles exit the IAG. The particle generation rate of the IAG, L_0, should be set between 10 to 100 particles s^{-1} to minimize the deposition of test particles inside the LSAPC. The LSAPC may be purged with a clean gas from the inlet to blow out potentially deposited particles after the calibration.

Typical sampling flowrate of LSAPC for cleanroom is larger than the aerosol flowrate of the IAG; therefore, filtered air needs to be added to make up for the rest of the sampling flowrate. Care needs to be taken to minimize the flow instability induced by the filtered air. Additional tubular wall may be placed around the sampling probe if needed. It is recommended that the flow Reynolds number inside a sampling chamber is less than 500 to supress turbulence inside the chamber.

The generator method can serve for two purposes. The first purpose is to evaluate whether there is any temporal shift in the intrinsic performance of the optical system of an LSAPC. In this evaluation, the vertical axis of the IAG and sampling probe of the LSAPC are aligned to deliver aerosol particles to the optical system without causing any transport losses. Formulae (3) and (4) are used to calculate the counting efficiency.

The second purpose is to evaluate the counting efficiency of an LSAPC including the transport efficiency through the sampling probe. In this evaluation, test particles are delivered to various spots over the inlet plane of the sampling probe to simulate the sampling of aerosol in which the particles are uniformly dispersed in space (see References [14] and [15]). LSAPC is placed on a platform with a motorized XY-stage to let the tip of the IAG exit tube travel through multiple injection points.

It is important to properly setup injection locations to simulate the sampling of aerosol in which particles are uniformly dispersed in space. Reference [15] explains how to setup the injection points. An overview of the procedure is given here. The particle flux is the number of particles passing through a unit area per unit time, injection points and the method simulates the particle flux being constant anywhere at the inlet plane. Since the test particles continuously pass through the inlet plane as the tip of the IAG exit tube travels through one point to another, the injection points are non-uniformly distributed toward radial direction to simulate the uniform particle flux.

The recommended moving speed of XY-stage, v, in millimetre per second is calculated by $v = 2R/10$ where the unit of R is in millimetres. The sampling time t in Formula (4) is set to a value greater than 180 s. The measurements are repeated to evaluate the average particle counts, $\bar{N}_1$.

$$\bar{N}_1 = \frac{1}{n}\sum_{i=1}^{n} N_\mathrm{i} \tag{A.1}$$

where

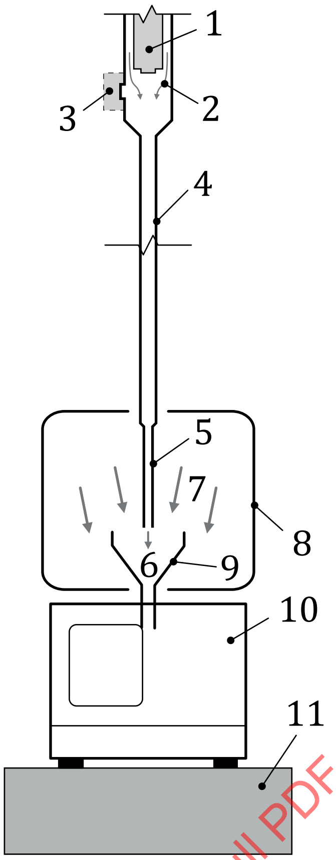
n is the number of measurements;

N_i is the particle counts at i-th measurement.

Then, the counting efficiency is evaluated by

$$\eta = \frac{\bar{N}_1}{N_0} \tag{A.2}$$

where N_0 is the number of particles introduced to the LSAPC.



Key

1 inkjet head
2 filtered carrier gas
3 bipolar ion source
4 evaporation tube
5 exit tube
6 aerosol flow
7 filtered sheath air
8 sheath flow chamber
9 sampling probe of LSAPC
10 LSAPC
11 platform with a XY-stage

Figure A.4 — Schematic description of the generator method

A.3.2 Particle diameter

The primary definition of the particle diameter is the volume equivalent diameter, d_v, when an IAG is used to generate test particles. d_v is defined as a volume equivalent sphere whose density is assumed to be the density of the particle material. The particle mass, m_p, in kg, is calculated from Formula (A.3):

$$m_p = C_m \cdot m_d \quad \text{(A.3)}$$

where

m_d is the average mass of inkjet droplets, in kg;

C_m is the mass concentration of solute in the inkjet solution, in kg solute/kg solution.

