Calibration of optical particle counters

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Abstract

The progress in research and development, the miniaturisation of high-tech processes and furthermore the strict safety regulations in comestible sectors, in health sectors and in the pharmaceutical industry lead towards more and more processes in clean rooms. An accurate monitoring of the clean rooms air quality is therefore mandatory. To fulfill air quality regulations in clean rooms, calibrated and traceable devices as optical particle counters are required.
As worldwide first metrology institute METAS is providing calibrations of optical particle counters in the range of 0.1 µm to 10.0 µm, traceable to SI-units.

Clean rooms and their classifications

According ISO 14644-1, the air quality in clean rooms and similar environments is defined by the number of smallest particles per cubic meter of air.
The clean rooms are categorized into nine classes based on the particle number concentration and particle size.
Very low particle number concentrations are crucial in clean rooms. The system provided by METAS is able to simulate clean rooms and allows comparisons with the national primary standard for particle counting, called LAPAZ.

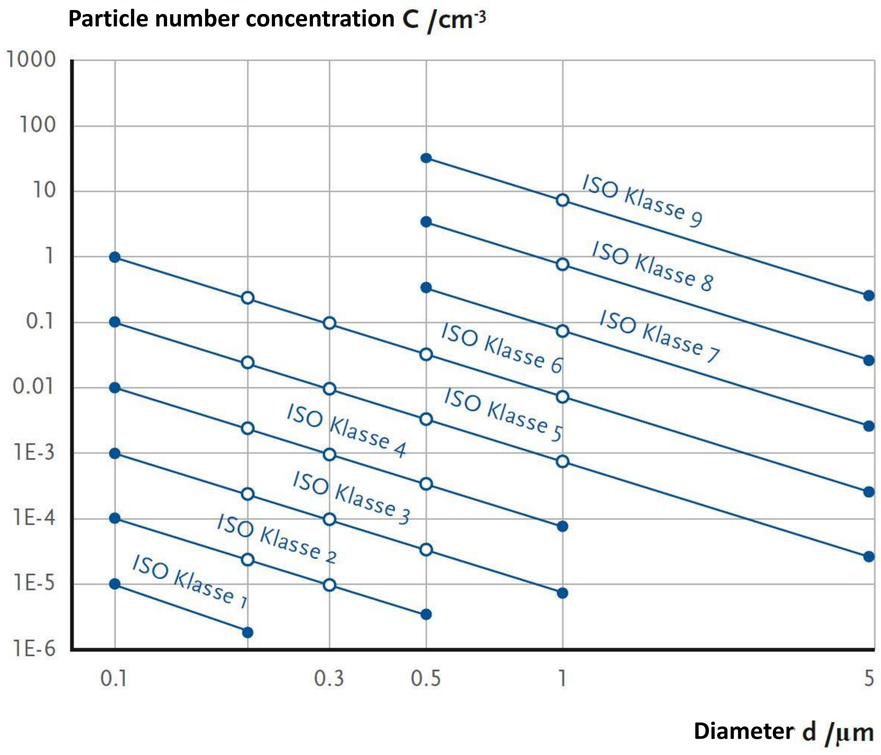

Fig. 1: Classification of clean rooms following the maximum allowed number concentration dependent on particle size according to ISO 14644-1.
Example: The particle number concentration of 35 cm^{-3} (respectively 8 cm^{-3}) of the size 0.5 µm (respectively 1 µm) must not be exceed for a class 9 room.

Requirements of ISO 21501-4

An optical particle counter complies with the requirements of ISO 21501-4 by meeting the following aspects, among others :

- The error of determining the particle size shall be less than 10 %.
- The counting efficiency at the minimum detectable size shall be (50 ± 20) %, and (100 ± 10) % at 1.5 to 2 times larger sizes.
- The device shall be calibrated once per year.

Particle Number Concentration Measurement Results

d	[nm]	:	certified diameter of PSL
C_{DUT}	[1/cm³]	:	average particle concentration of the DUT within the size range (SR) (uncorrected instrument reading at above measurement conditions)
s	[1/cm³]	:	empirical standard deviation of C_{DUT}
C_{LAPAZ}	[1/cm³]	:	average particle concentration of LAPAZ (results refer to above measurement conditions)
SR	[µm]	:	considered size range(s) of the DUT. All results are given within specified diameter limits. Possible contributions to the measurements beyond these limits are neglected.

$d \pm U$	$C_{DUT} \pm s$	$C_{LAPAZ} \pm U$	$C_{DUT} / C_{LAPAZ} \pm U$	SR	C_{SR1}/C_{SR2}
102.0 ± 3.4	0.482 ± 0.003	0.95 ± 0.08	0.51 ± 0.04	0.1…0.2	-
102.0 ± 3.2	1.197 ± 0.049	2.47 ± 0.19	0.48 ± 0.04	0.1…0.2	-
152 ± 6	0.971 ± 0.010	0.94 ± 0.08	1.03 ± 0.08	0.1…0.2 & 0.2…0.3	9.69 ± 0.08
199 ± 7	0.872 ± 0.012	0.84 ± 0.07	1.04 ± 0.08	0.1…0.2 & 0.2…0.3	0.95 ± 0.01
199 ± 7	2.068 ± 0.006	2.00 ± 0.14	1.03 ± 0.07	0.1…0.2 & 0.2…0.3	0.98 ± 0.00
300 ± 7	1.106 ± 0.032	1.11 ± 0.09	1.00 ± 0.08	0.2…0.3 & 0.3…0.5	0.94 ± 0.00
300 ± 7	2.533 ± 0.021	2.63 ± 0.14	0.96 ± 0.05	0.2…0.3 & 0.3…0.5	0.95 ± 0.00
498 ± 6	1.009 ± 0.016	0.97 ± 0.07	1.04 ± 0.07	0.3…0.5 & 0.5…1.0	1.02 ± 0.01
498 ± 6	2.345 ± 0.026	2.24 ± 0.13	1.05 ± 0.06	0.3…0.5 & 0.5…1.0	1.00 ± 0.00
1005 ± 22	1.46 ± 0.06	1.55 ± 0.14	0.94 ± 0.09	0.5…1.0 & 1.0…5.0	0.98 ± 0.00
1005 ± 22	3.653 ± 0.026	3.84 ± 0.21	0.95 ± 0.05	0.5…1.0 & 1.0…5.0	0.95 ± 0.00
5300 ± 330	1.14 ± 0.09	1.10 ± 0.11	1.04 ± 0.13	0.5…1.0 & 1.0…5.0	0.75 ± 0.00
5300 ± 320	3.14 ± 0.09	3.02 ± 0.19	1.04 ± 0.07	0.5…1.0 & 1.0…5.0	0.74 ± 0.01

Efficiency of DUT

Uncertainty of Measurement

The reported uncertainty of measurement is stated as the combined standard uncertainty multiplied by a coverage factor $k = 2$. The measured value (y) and the associated expanded uncertainty (U) represent the interval ($y \pm U$) which contains the value of the measured quantity with a probability of approximately 95 %. The uncertainty was estimated following the guidelines of the ISO (GUM:1995).

The measurement uncertainty contains contributions originating from the measurement standard, from the calibration method, from the environmental conditions and from the object being calibrated. The long-term characteristic of the object being calibrated is not included

Fig. 3: Extract of a calibration certificate

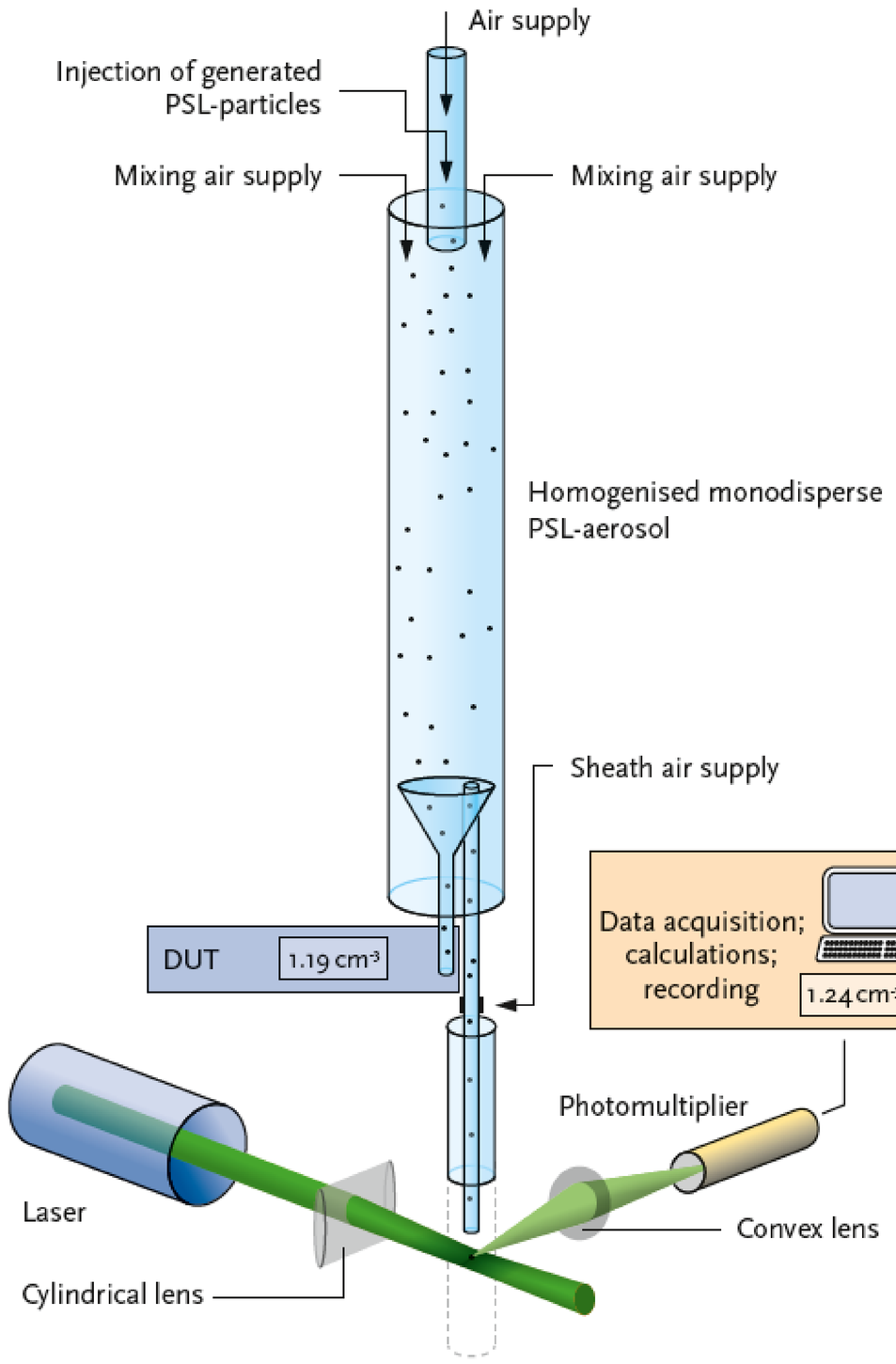

Fig. 2: The vertical assembly of the homogeniser with the sampling for the national primary standard and the device under test (DUT).

The Federal Institute of Metrology METAS

The Federal Institute of Metrology (METAS) serves as the federal center of competence for all issues related to measurement and for measuring equipment and measuring procedures.

METAS is the Swiss national metrology institute. As such, its mandate is to ensure the availability of measurement and testing facilities with the degree of accuracy needed to meet the requirements of the economy, research and administration in Switzerland.

METAS stands at the cutting edge of measurement accuracy in Switzerland. It develops the national measurement base, that is to say it looks after the physical implementation, mutual comparison and thus the international recognition of measurement units. For this purpose, it operates the necessary laboratories and conducts the necessary research and development. It implements the Metrology Act; its other tasks are defined in the Federal Act on the Swiss Federal Institute of Metrology.

Calibration of particle size…

According to ISO 21501-4, the measured particle size shall be calibrated by using Polystyrene Latex Particles (PSL).
The diameter of the PSL-particles is traceable to national length standards by means of atomic force microscopy calibration at METAS.
The expanded relative uncertainty of measurement of the particle diameter is between 2 % and 5 %.

…and number concentration

The revision of the measuring devices usually includes the adjustment of the size channels only.
The calibration of the counting efficiency is often skipped or high concentrated aerosols are used for the calibration, therefore its practical application for clean rooms is not sufficient.
According to ISO 21501-4, traceable measurements of the reference particle number concentration shall be done with a calibrated condensation particle counter (CPC) or a calibrated optical particle counter.
CPCs need to be calibrated with an electrometer according to ISO 27891. As electrometers only allow calibrations at particle number concentrations > 1000 cm^{-3}, again its practical application for clean rooms is not sufficient.
The national primary standard at METAS is directly traceable to SI-units and stands therefore at the cutting edge of measurement pyramid. The expanded relative uncertainty of measurement of the particle number concentration is between 5 % and 10 %.

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