INTERNATIONAL STANDARD

ISO 27891

First edition
2015-03-01

Aerosol particle number concentration — Calibration of condensation particle counters

Densité de particules d'aérosol — Étalonnage de compteurs de particules d'aérosol à condensation

Reference number
ISO 27891:2015(E)

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Foreword

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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).

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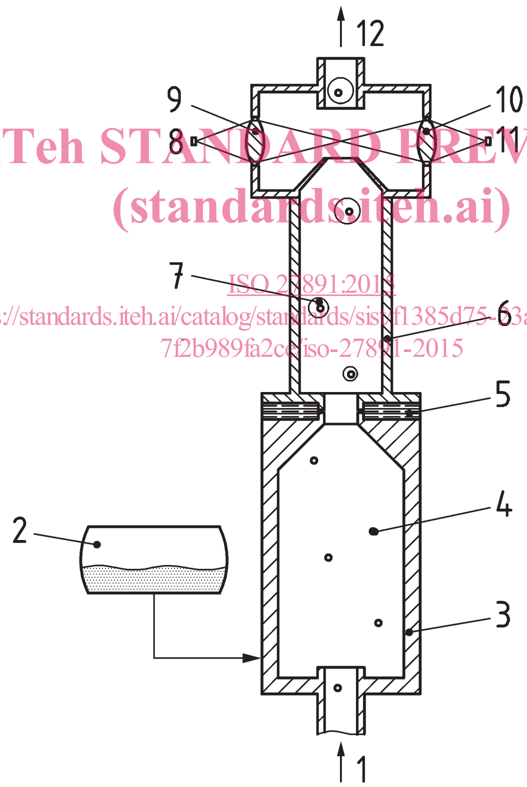
For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

Introduction

A condensation particle counter (CPC) is a measuring device for the number concentration of small aerosol particles. The common principle of all different CPC types is that condensation of supersaturated vapours is used to grow ultra-fine and nanoparticles to droplets of sizes that can be detected optically.[44] The counting of the droplets is performed via optical light scattering. The droplet passes through a detection area where it is illuminated by a focused light beam and a portion of the scattered light is detected with a photodetector. The frequency of this event leads, with the known volume of sampled air, to the particle number concentration. At low concentrations, the CPC counts individual particles and allows an absolute determination of particle number concentration.

Commercially available CPCs employ different working fluids to generate the vapour, e.g. 1-butanol, 2-propanol, or water. Moreover, different principles are in use to achieve the needed supersaturation in the sample air. The most common CPC uses laminar flow and diffusional heat transfer. The diffusion constant of the working fluid determines the needed heating or cooling steps to initiate condensation and hence, the principle design of a laminar flow CPC. Less common are turbulent mixing CPCs: in these CPCs, the supersaturation is achieved by turbulently mixing the sample air with a particle free gas flow saturated with the working fluid. Figure 1 shows a schematic of the probably most common CPC type with a laminar flow through a heated saturator and a cooled condenser.



Key

1	aerosol inlet	7	droplet
2	working fluid reservoir	8	light source
3	heated saturator	9	illumination optics
4	nanoparticle	10	receiving optics
5	thermoelectric cooling and heating device	11	photodetector
6	condenser	12	aerosol outlet

Figure 1 — Principle of a laminar flow CPC

The accuracy of CPC measurements, however, depends on various influences. For example, if the flow rate had an error, the concentration would have an error. Coincidence error at very high concentration, inefficient activation of particle growth at very small sizes, and losses of particles during transport from the inlet to the detection section are other possible sources of errors. For accurate measurement, the CPC shall be calibrated.

“Calibration” of the CPC is usually done using a Faraday-cup aerosol electrometer (FCAE) as reference instrument.[33][36] In many cases, the purpose of the “calibration” is to determine the limit of particle detection at very small size. The FCAE has been used as the reference since the detection efficiency of the FCAE was considered to be unity at any size. The detection efficiency of a CPC is determined as the ratio of the concentration indicated by the CPC under calibration to that by the FCAE, while aerosols of singly charged, size-classified particles of the same number concentration are supplied simultaneously to both instruments.

This International Standard sets out two distinct methods of CPC calibration: the characterization of a CPC by comparison with an FCAE, which is the same as the traditional approach described above; and by comparison with a reference CPC. An FCAE that has a reputable calibration certificate, covering the relevant particle number concentrations, sizes, and composition, can be used. In the latter case, the reference CPC is one that has a reputable calibration certificate, again covering the relevant particle number concentrations, sizes, and composition. A reputable calibration certificate shall mean either one that has been produced by a laboratory accredited to ISO/IEC 17025 or an equivalent standard, where the type and range of calibration is within the laboratory’s accredited scope, or a European Designated Institute or a National Metrology Institute that offers the relevant calibration service and whose measurements fulfil the requirements of ISO/IEC 17025.

Two major sources of errors are known in CPC calibration: the presence of multiply charged particles and the bias of the particle concentrations between the inlet of the CPC under calibration and that of the reference instrument. Evaluation of these factors and corrections for them shall be included in the calibration procedure, the methods of which are specified in this International Standard.

This International Standard is aimed at

— users of CPCs (e.g. for environmental or vehicle emissions purposes) who have internal calibration programmes,

— CPC manufacturers who certify and recertify the performance of their instruments, and

— technical laboratories who offer the calibration of CPCs as a service, which can include National Metrology Institutes who are setting up national facilities to support number concentration measurements.

Aerosol particle number concentration — Calibration of condensation particle counters

1 Scope

This International Standard describes methods to determine the detection efficiency of condensation particle counters (CPCs) at particle number concentrations ranging between 1 cm^{-3} and 10^5 cm^{-3}, together with the associated measurement uncertainty. In general, the detection efficiency will depend on the particle number concentration, the particle size, and the particle composition. The particle sizes covered by the methods described in this International Standard range from approximately 5 nm to 1 000 nm.

The methods can therefore be used both to determine a CPC calibration factor to be applied across the range of larger particle sizes where the detection efficiency is relatively constant (the plateau efficiency), and to characterize the drop in CPC detection efficiency at small particle sizes, near the lower detection limit. These parameters are described in more detail in Annex A.

The methods are suitable for CPCs whose inlet flows are between approximately 0,1 l/min and 5 l/min.

This International Standard describes a method for estimating the uncertainty of a CPC calibration performed according to this International Standard.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 15900, *Determination of particle size distribution — Differential electrical mobility analysis for aerosol particles*

3 Terms and definitions

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

3.1
aerosol
system of solid or liquid particles suspended in gas

3.2
bipolar charger
particle charge conditioner to attain the equilibrium, known size-dependent charge distribution by exposing aerosol particles to both positive and negative ions within the device

Note 1 to entry: Exposing aerosol particles to an electrically neutral cloud of positive and negative gas charges with sufficiently high charge concentration and for a sufficiently long period of time leads to an equilibrium with the net charge of the aerosol nearly zero (also known as charge neutralization).

3.3
calibration
operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

Note 1 to entry: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Note 2 to entry: Calibration should not be confused with adjustment of a measuring system, often mistakenly called "self-calibration", nor with verification of calibration.

Note 3 to entry: Often, the first step alone in the above definition is perceived as being calibration.

[SOURCE: ISO/IEC Guide 99]

3.4
calibration aerosol
charge conditioned and size classified primary aerosol with particle number concentration adjusted for the calibration measurement, as delivered by the flow splitter

3.5
calibration particle material
material of the particles of the calibration aerosol

3.6
charge concentration
concentration of the net electrical charges per unit volume

Note 1 to entry: Charge concentration is the measurand of the FCAE.

Note 2 to entry: FCAE measurement can be displayed as charge concentration, C_Q, (e.g. in fC/cm^3), charge number concentration, C_N^*, (e.g. in cm^{-3}) or electrical current, I_{FCAE}, (e.g. in fA). Using the elementary charge, e, and the volumetric FCAE inlet flow rate, q_{FCAE}, these displayed values are related as follows:

$$C_N^* = C_Q / e = I_{\mathrm{FCAE}} / (q_{\mathrm{FCAE}} \times e)$$

EXAMPLE A charge concentration of 1 fC/cm^3 corresponds to a charge number concentration of 6241 cm^{-3}. When the volumetric FCAE inlet flow rate is 1 l/min, the resulting electrical current is 16,67 fA.

3.7
charge conditioning
process that establishes a steady state charge distribution on the sampled aerosol

3.8
coefficient of variation
CV
ratio of the standard deviation to the arithmetic mean value

3.9
coincidence error
probability of the presence of more than one particles inside the sensing zone simultaneously

Note 1 to entry: Coincidence error is related to particle number concentration, flow velocity through the sensing zone and size of sensing zone.

3.10
condensation particle counter
CPC
instrument that measures the particle number concentration of an aerosol

Note 1 to entry: The sizes of particles detected are usually smaller than several hundred nanometres and larger than a few nanometres.

Note 2 to entry: In some cases, a CPC may be called a condensation nucleus counter (CNC).

Note 3 to entry: The CPC used as the reference instrument is called the "reference CPC" throughout this International Standard.

Note 4 to entry: The CPC under calibration is called the "test CPC" throughout this International Standard.

[SOURCE: ISO 15900:2009, modified]

3.11
detection efficiency
η
ratio of the concentration reported by an instrument to the actual concentration at the inlet of the instrument

3.12
differential electrical mobility classifier
DEMC
classifier that is able to select aerosol particles according to their electrical mobility and pass them to its exit

Note 1 to entry: A DEMC classifies aerosol particles by balancing the electrical force on each particle with its aerodynamic drag force in an electrical field. Classified particles are in a narrow range of electrical mobility determined by the operating conditions and physical dimensions of the DEMC. Classified particles can have different sizes due to difference in the number of charges that they have.

[SOURCE: ISO 15900:2009, modified]

3.13
differential mobility analyzing system
DMAS
system to measure the size distribution of submicrometre aerosol particles consisting of a pre-conditioner, particle charge conditioner, DEMC, flow meters, a particle detector, interconnecting plumbing, a computer, and software suitable for size-distribution calculation

[SOURCE: ISO 15900:2009, modified]

3.14
diffusion loss
reduction of particle number concentration due to thermal (or Brownian) and turbulent diffusion transport (e.g. to the walls of a transport tube)

3.15
electrometer
device that measures electrical current of about 1 femtoampere (fA) and higher

[SOURCE: ISO 15900:2009, modified]

3.16
equivalent particle diameter
d
equivalent diameter of the sphere with defined characteristics which behaves under defined conditions in exactly the same way as the particle being described

Note 1 to entry: Particle diameter (or simply diameter) used throughout this International Standard always refers to the electrical mobility equivalent diameter, which defines the size of charged particles with the same electrical mobility or the same terminal migration velocity in still air under the influence of a constant electrical field.

3.17
Faraday-cup aerosol electrometer
FCAE
electrometer designed for the measurement of electrical charge concentration carried by an aerosol

Note 1 to entry: An FCAE consists of an electrically conducting and electrically grounded cup as a guard to cover the sensing element that includes aerosol filtering media to capture charged aerosol particles, an electrical connection between the sensing element and an electrometer circuit, and a flow meter.

[SOURCE: ISO 15900:2009, modified]

3.18
flow rate
quantity (volume or mass to be specified) of a fluid crossing the transverse plane of a flow path per unit time

Note 1 to entry: For the exact flow rate indication of gases, information on the gaseous condition (temperature and pressure) or the reference to a standard volume indication is necessary.

3.19
GSD
acronym used in this International Standard for geometric standard deviation

3.20
laminar flow
gas flow with no temporally or spatially irregular activity or turbulent eddy flow

3.21
lower limit of the plateau efficiency
$d_{min,ref}$
lower size limit for which a reference CPC can be applied for the calibration of a test CPC

Note 1 to entry: This size limit depends on the CPC itself, but also to some extent on experimental conditions and on the particle type.

3.22
monodisperse aerosol
aerosol with a narrow particle size distribution

Note 1 to entry: Monodispersity can be quantified by the geometric standard deviation (GSD) of the size distribution.

Note 2 to entry: In this International Standard, the term “monodisperse” is used for the GSD less than or equal to 1,15.

3.23
particle
piece of matter with defined physical boundary

Note 1 to entry: The phase of a particle can be solid, liquid, or between solid and liquid and a mixture of any of the phases.

3.24
particle charge conditioner
device used for charge conditioning

3.25
particle number concentration
C
number of particles related to the unit volume of the carrier gas

Note 1 to entry: For the exact particle number concentration indication, information on the gaseous condition (temperature and pressure) or the reference to a standard volume indication is necessary.

3.26
particle type
several particle properties like chemical composition of the particle material (especially chemical surface composition), physical particle shape and morphology (e.g. an agglomerate or aggregate)

Note 1 to entry: The CPC detection efficiency at low particle sizes will depend on the chemical affinity between the particle and the working fluid (see Annex B).

Note 2 to entry: Much of the underlying theory assumes that the particles are solid spheres. Non-sphericity can affect the size selection by the DEMC, the fraction of multiply charged particles, and the condensation of working fluid on the particle surface.

3.27
plateau efficiency
mean detection efficiency of a CPC in the size range which is not biased by particle size

3.28
primary aerosol
aerosol generated and conditioned in the primary aerosol source section of the calibration setup

3.29
single particle counting mode
measurement mode of a particle number or number concentration measurement device (e.g. a CPC) in which every detected particle is counted to obtain the measurement result

3.30
size distribution
distribution of particle concentration as a function of particle size

Note 1 to entry: In this International Standard, this term is used in the sense "particle number concentration represented as function of the particle diameter".

Note 2 to entry: ISO 9276-1 can be applied for the representation of results of particle size distribution analysis.

3.31
turbulent flow
gas flow with temporally or spatially irregular activity or turbulent eddy flow

3.32
unipolar charger
particle charge conditioner that expose aerosol particles to either positive or negative ions within the device

4 Symbols

For the purpose of this International Standard, the following symbols and abbreviated terms apply. Units are in accordance with Reference [15].

Symbol	Quantity	Unit	Used in
C_N	total number concentration of particles out of the DEMC	cm^{-3}	6.3.5 e) 7.3.5 c)
$C_N(d_p)$	number concentration of particles out of the DEMC of equivalent particle diameter *d* and with *p* charges	cm^{-3}	6.3.3 c) 7.3.3 c)
C_N^*	charge number concentration	cm^{-3}	3.6 NOTE 2
$C_{N,\mathrm{CPC}}$	indicated particle number concentration measured by the test CPC	cm^{-3}	5.1
$C_{N,\mathrm{CPC},i}$	*i*-th indicated number concentration measured by the test CPC when measuring particles	cm^{-3}	6.3.5 b) 7.3.5 a)

Symbol	Quantity	Unit	Used in
$C_{N,\text{CPC,ref},i}$	*i*-th indicated number concentration measured by the reference CPC when measuring particles	cm^{-3}	7.3.5 a)
$C_{N,\text{FCAE},i}$	*i*-th calculated number concentration of the calibration aerosol	cm^{-3}	6.3.5 d)
$C_{N,\text{ref}}$	indicated particle number concentration measured by the reference instrument	cm^{-3}	5.1
C_Q	indicated charge concentration measured by FCAE when measuring particles	$C\ cm^{-3}$	3.6 NOTE 2
$C_{Q,i}$	*i*-th indicated charge concentration measured by FCAE when measuring particles	$C\ cm^{-3}$	6.3.5 a), c)
$C_{Q,0,i}$	*i*-th indicated charge concentration measured by FCAE when the DEMC voltage set at zero	$C\ cm^{-3}$	6.3.5 a), c)
d	equivalent particle diameter	nm	3.16
$d_{\text{min,ref}}$	lower size limit for which a reference CPC can be applied for the calibration of a test CPC	nm	3.21 5.4.6 b) 5.5 a)
e	elementary charge = 1,602 177 × 10^{-19} C	C	3.6 NOTE 2
I_{FCAE}	electrical current	fA	3.6 NOTE 2
k	coverage factor	(dimensionless)	6.4.3 7.4.3
N_{ambient}	number of particle counts over 1 min without a HEPA filter	(dimensionless)	6.2.5 a) 2)
N_{FCAE}	number of particle counts over 1 min through the FCAE filter	(dimensionless)	6.2.5 a) 5)
N_{HEPA}	number of particle counts over 1 min with a HEPA filter	(dimensionless)	6.2.5 a) 1)
N_{leak}	$= N_{\text{FCAE}} - N_{\text{HEPA}}$	(dimensionless)	6.2.5 a) 6)
p	number of net charges on a particle	(dimensionless)	5.1
$q_{\text{CPC,amb}}$	inlet flow rate indicated by the test CPC or the nominal inlet flow rate of the test CPC	$l\ min^{-1}$	6.2.6 c) 7.2.6 e)
$q_{\text{CPC,cal,amb}}$	inlet flow rate of the test CPC measured with a calibrated flow meter	$l\ min^{-1}$	6.2.6 c) 7.2.6 e)
$q_{\text{CPC,ref}}$	inlet flow rate indicated by the reference CPC or the nominal inlet flow rate of the reference CPC	$l\ min^{-1}$	7.2.7 b)
$q_{\text{CPC,ref,cal}}$	inlet flow rate of the reference CPC measured with a calibrated flow meter	$l\ min^{-1}$	7.2.7 b)
$q_{\text{CPC,ref,cal,amb}}$	inlet flow rate of the reference CPC measured with a calibrated flow meter	$l\ min^{-1}$	7.2.5 c)
$q_{\text{CPC,ref,amb}}$	inlet flow rate indicated by the reference CPC or the nominal inlet flow rate of the reference CPC	$l\ min^{-1}$	7.2.5 c)
$q_{\text{CPC,ref,cert}}$	inlet flow rate of the reference CPC in the calibration certificate	$l\ min^{-1}$	7.2.5 b)
q_{FCAE}	inlet flow rate indicated by the FCAE or the nominal inlet flow rate of the FCAE	$l\ min^{-1}$	3.6 NOTE 2 6.2.7 b)
$q_{\text{FCAE,amb}}$	inlet flow rate indicated by the FCAE or the nominal inlet flow rate of the FCAE	$l\ min^{-1}$	6.2.5 c)
$q_{\text{FCAE,cal}}$	inlet flow rate of the FCAE measured with a calibrated flow meter	$l\ min^{-1}$	6.2.7 b)
$q_{\text{FCAE,cal,amb}}$	inlet flow rate of the FCAE measured with a calibrated flow meter	$l\ min^{-1}$	6.2.5 c)
$q_{\text{FCAE,cert}}$	inlet flow rate of the FCAE in the calibration certificate	$l\ min^{-1}$	6.2.5 c)

Symbol	Quantity	Unit	Used in
R_{FCAE}	$= N_{leak}/N_{ambient}$	(dimensionless)	6.2.5 a) 7)
$r_{q,CPC,ref}$	accuracy of the inlet flow rate of the reference CPC specified by the manufacturer	$l\ min^{-1}$	7.2.5 c)
$r_{q,FCAE}$	accuracy of the inlet flow rate of the FCAE specified by the manufacturer	$l\ min^{-1}$	6.2.5 c)
$U(\eta)$	expanded uncertainty for η	(dimensionless)	6.4.3 7.4.3
$U_r(\eta)$	relative expanded uncertainty for η	(dimensionless)	6.4.3 7.4.3
$u_r(q_{cal,cert})$	relative standard uncertainty of the flow meter	(dimensionless)	6.2.5 c) 7.2.5 e)
$u_r(q_{CPC,ref})$	relative standard uncertainty for the inlet flow of the reference CPC	(dimensionless)	7.2.7 b) 7.4.3
$u_r(q_{CPC,ref,cert})$	relative standard uncertainty for the inlet flow of the reference CPC in the calibration certificate	(dimensionless)	7.2.5 c)
$u_r(q_{FCAE})$	relative standard uncertainty for the inlet flow of the FCAE	(dimensionless)	6.2.7 b) 6.4.3
$u_r(q_{FCAE,cert})$	relative standard uncertainty for the inlet flow of the FCAE in the calibration certificate	(dimensionless)	6.2.5 c)
$u(1)$	standard uncertainty for ϕ_1	(dimensionless)	6.4.3
$u(2)$	standard uncertainty for ϕ_2	(dimensionless)	6.4.3
$u(3)$	standard uncertainty for ϕ_3	(dimensionless)	6.4.3
$u_{c,r}(\eta)$	relative combined standard uncertainty for η	(dimensionless)	6.4.3 7.4.3
$u_r(FCAE)$	relative standard uncertainty for the FCAE detection efficiency	(dimensionless)	6.4.3
$u_r(MCC)$	relative standard uncertainty for multiple-charge correction	(dimensionless)	6.4.3
$u_r(RCPC)$	relative standard uncertainty for the reference CPC detection efficiency	(dimensionless)	7.4.3
$u_r(\beta)$	relative standard uncertainty for β	(dimensionless)	6.4.3 7.4.3
$u_r(\eta_{rep})$	relative standard uncertainty for repeatability	(dimensionless)	6.4.3 7.4.3
β	concentration bias correction factor for flow splitter	(dimensionless)	5.1 6.3.4 7.3.4
η	detection efficiency	(dimensionless)	3.11
η_{CPC}	detection efficiency of the test CPC	(dimensionless)	5.1
η'_{CPC}	estimated plateau efficiency of the test CPC	(dimensionless)	6.3.5 e) 7.3.5 e)
$\eta_{CPC,i}$	*i*-th detection efficiency of the test CPC	(dimensionless)	6.3.5 e) 7.3.5 e)
$\eta_{CPC,ref}$	detection efficiency of the reference CPC	(dimensionless)	7.3.5 c)
$\bar{\eta}_{CPC}$	arithmetic mean detection efficiency of the test CPC	(dimensionless)	6.3.5 f) 7.3.5 d)
η_{FCAE}	detection efficiency of the FCAE	(dimensionless)	6.3.5 e)
η_{ref}	detection efficiency of the reference instrument	(dimensionless)	5.1