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Aerosol particle number concentration — Calibration of condensation particle counters



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National foreword

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Aerosol particle number concentration — Calibration of condensation particle counters

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





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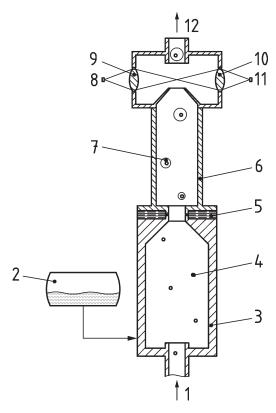
For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

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
- 2 working fluid reservoir
- 3 heated saturator
- 4 nanoparticle
- 5 thermoelectric cooling and heating device
- 6 condenser

- 7 droplet
- 8 light source
- 9 illumination optics
- 10 receiving optics
- 11 photodetector
- 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 $1 \, \text{cm}^{-3}$ and $10^5 \, \text{cm}^{-3}$, 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 $5 \, \text{nm}$ to $1 \, 000 \, \text{nm}$.

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/cm³), 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:

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

EXAMPLE A charge concentration of 1 fC/cm³ 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

$\mathbf{C}\mathbf{V}$

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 preconditioner, 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

7

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 <u>Annex B</u>).

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	<u>5.1</u>
$C_{N,\mathrm{CPC},i}$	<i>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
$C_{N, \mathrm{CPC}, \mathrm{ref}, i}$	<i>i</i> -th indicated number concentration measured by the reference CPC when measuring particles	cm ⁻³	<u>7.3.5</u> a)
$C_{N,\text{FCAE},i}$	<i>i</i> -th calculated number concentration of the calibration aerosol	cm ⁻³	6.3.5 d)
$C_{N,ref}$	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>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>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
$d_{ m min,ref}$	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)
е	elementary charge = 1,602 177 \times 10 ⁻¹⁹ C	С	3.6 NOTE 2
I_{FCAE}	electrical current	fA	3.6 NOTE 2
k	coverage factor	(dimensionless)	6.4.3 7.4.3
Nambient	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 _{FCAE} - N _{HEPA}	(dimensionless)	6.2.5 a) 6)
р	number of net charges on a particle	(dimensionless)	5.1
<i>q</i> 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)
<i>q</i> 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)
<i>q</i> 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)
<i>q</i> CPC,ref,cal	inlet flow rate of the reference CPC measured with a calibrated flow meter	l min ⁻¹	7.2.7 b)
<i>q</i> CPC,ref,-cal,amb	inlet flow rate of the reference CPC measured with a calibrated flow meter	l min ⁻¹	<u>7.2.5</u> c)
<i>q</i> CPC,ref,amb	inlet flow rate indicated by the reference CPC or the nominal inlet flow rate of the reference CPC	l min ⁻¹	<u>7.2.5</u> c)
<i>q</i> CPC,ref,cert	inlet flow rate of the reference CPC in the calibration certificate	l min ⁻¹	<u>7.2.5</u> b)
<i>q</i> 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)
<i>q</i> 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)
<i>q</i> FCAE,cal	inlet flow rate of the FCAE measured with a calibrated flow meter	l min ⁻¹	6.2.7 b)
<i>q</i> FCAE,cal,amb	inlet flow rate of the FCAE measured with a calibrated flow meter	l min ⁻¹	6.2.5 c)
<i>q</i> 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}	$= N_{\text{leak}}/N_{\text{ambient}}$	(dimensionless)	<u>6.2.5</u> a) 7)
$r_{q, \mathrm{CPC, ref}}$	accuracy of the inlet flow rate of the reference CPC specified by the manufacturer	l min ⁻¹	7.2.5 c)
$r_{q, ext{FCAE}}$	accuracy of the inlet flow rate of the FCAE specified by the manufacturer	l min ⁻¹	6.2.5 c)
<i>U</i> (η)	expanded uncertainty for η	(dimensionless)	6.4.3 7.4.3
$U_{ m r}(\eta)$	relative expanded uncertainty for η	(dimensionless)	6.4.3 7.4.3
$u_{\rm r}(q_{\rm cal,cert})$	relative standard uncertainty of the flow meter	(dimensionless)	6.2.5 c) 7.2.5 e)
$u_{\rm r}(q_{\rm CPC,ref})$	relative standard uncertainty for the inlet flow of the reference CPC	(dimensionless)	7.2.7 b) 7.4.3
$u_{ m r}(q_{ m CP}$ -C,ref,cert)	relative standard uncertainty for the inlet flow of the reference CPC in the calibration certificate	(dimensionless)	7.2.5 c)
$u_{\rm r}(q_{\rm FCAE})$	relative standard uncertainty for the inlet flow of the FCAE	(dimensionless)	6.2.7 b) 6.4.3
$u_{\rm r}(q_{\rm FCAE,cert})$	relative standard uncertainty for the inlet flow of the FCAE in the calibration certificate	(dimensionless)	<u>6.2.5</u> 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
$u_{\rm r}({\rm FCAE})$	relative standard uncertainty for the FCAE detection efficiency	(dimensionless)	6.4.3
$u_{\rm r}({ m MCC})$	relative standard uncertainty for multiple-charge correction	(dimensionless)	6.4.3
$u_{\rm r}({ m RCPC})$	relative standard uncertainty for the reference CPC detection efficiency	(dimensionless)	7.4.3
$u_{\rm r}(\beta)$	relative standard uncertainty for eta	(dimensionless)	6.4.3 7.4.3
$u_{ m r}(\eta_{ m rep})$	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
$\eta_{ ext{CPC}}$	detection efficiency of the test CPC	(dimensionless)	5.1
η΄СРС	estimated plateau efficiency of the test CPC	(dimensionless)	6.3.5 e) 7.3.5 e)
$\eta_{\mathrm{CPC},i}$	<i>i</i> -th detection efficiency of the test CPC	(dimensionless)	6.3.5 e) 7.3.5 e)
$\eta_{ ext{CPC,ref}}$	detection efficiency of the reference CPC	(dimensionless)	<u>7.3.5</u> c)
$ar{\eta}_{ ext{CPC}}$	arithmetic mean detection efficiency of the test CPC	(dimensionless)	6.3.5 f) 7.3.5 d)
$\eta_{ extsf{FCAE}}$	detection efficiency of the FCAE	(dimensionless)	<u>6.3.5</u> e)
$\eta_{ m ref}$	detection efficiency of the reference instrument	(dimensionless)	5.1

Symbol	Quantity	Unit	Used in
$\sigma(\eta_{ m rep})$	standard deviation for the repeated measurements of the detection efficiency of the test CPC	(dimensionless)	6.3.5 f) 7.3.5 d)
Φ	fraction of multiply charged particles	(dimensionless)	5.5 b) 6.3.3 c) 7.3.3 c)
ϕ_p	fraction of particles with p charges	(dimensionless)	5.1 6.3.3 c) 7.3.3 c)

5 Calibration using reference instruments — General principles

5.1 General principles

This Clause describes general aspects of CPC calibration using traceable reference instruments, while the subsequent <u>Clauses 6</u> and <u>7</u> refer to specific aspects of FCAE and CPC as reference instruments, respectively.

The reference instruments shall have an up-to-date reputable calibration certificate specifying the particle type, the particle sizes, and the particle number concentration range which was used for its calibration. Volumetric inlet flow rate, inlet pressure and inlet temperature at the time of calibration shall also be specified. 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. Examples of calibration certificates are given in Annex C.

The result of a calibration will be the particle detection efficiency for an individual CPC instrument with specified operating parameters, and for specific cases of

- particle size,
- particle type, and
- particle number concentration.

In CPC single particle counting mode, the detection efficiency is often expressed as a single figure (with uncertainty) over a range of concentrations, i.e. a single factor applies. In other modes, or over wider concentration ranges, more complicated relationships between detection efficiency and particle number concentration may be appropriate (Annex A). The calculations of the detection efficiency and its uncertainty are described in Clauses 6 and 7 and follow the general formula

$$\eta_{\text{CPC}} = \frac{C_{N,\text{CPC}}}{C_{N,\text{ref}}} \cdot \eta_{\text{ref}} \cdot \beta \cdot \sum_{p} \phi_{p} \cdot p \tag{1}$$

where

 $C_{N,CPC}$ is the indicated concentration of the test CPC (i.e. the CPC being calibrated);

 $C_{N,ref}$ is the concentration of the reference instrument;

 $\eta_{\rm ref}$ is the efficiency of the reference instrument; and

 β is the concentration bias from the flow splitter.

The summing term in Formula (1) is only used if the reference instrument is an FCAE. ϕ_p is the fraction of particles having p charges [see also Formula (6)].

5.2 Objectives for the calibration aerosol

The role of many of the components described in subsequent clauses is to modify the output of a primary aerosol into a form suitable for the calibration. The calibration aerosol should have the following:

- a narrow size distribution, so that the size of the particles is well defined (typically GSD <1,1 for the primary peak in the size distribution), to minimize uncertainty in size and efficiency;
- stable mode diameter, and stable number concentration in relation to the required time for calibration (typically 10 min), so that the calibration can take place in essentially constant conditions;
- a small fraction of multiply charged particles (typically <5 %), because these become a significant component of the uncertainty for FCAE calibrations, and in both cases they form extra populations of particles at unwanted larger sizes (see <u>Annex D</u>);
- a low vapour content (from water, other dispersion media and/or solvents), to minimize the growth
 of particles within the system;
- a stable and reproducible gas phase and particle type.

A CPC calibration certificate is only applicable for the calibration aerosol that is described in the calibration certificate, especially at low particle size.

5.3 Setup overview

A primary aerosol source and a DEMC are used to deliver monodisperse calibration aerosols of known size, electrostatic charge and composition. A traceable reference instrument and the test CPC sample this aerosol in parallel downstream of the DEMC. Either an FCAE or a reference CPC is used as traceable reference instrument. Figure 2 shows the schematic setup of the components necessary. A temperature controlled box and heat exchangers for all important air flows may be used in this setup as an option to stabilize all temperatures.

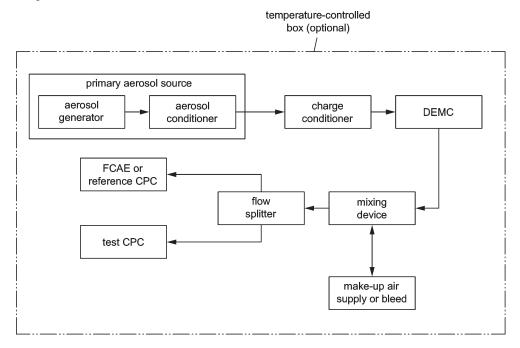


Figure 2 — Schematic calibration setup

In order that results of CPC calibration that is performed according to this International Standard are regarded traceable to national standards, instruments used in the calibration, including the FCAE and reference CPC, shall be calibrated with metrological traceability to international or national standards.

The figures in Annex E illustrate the metrological traceability chains for quantities that are influential to results of calibration of a CPC.

5.4 Components and their requirements

5.4.1 Primary aerosol source

5.4.1.1 General

The primary aerosol source consists of an aerosol generator and an aerosol conditioner to provide calibration particles in a stable particle type and adequate number concentration.

The stability of flow rate, number concentration and size distribution shall be such that the requirements for the calibration aerosol (see 5.2) are fulfilled.

A narrow primary aerosol source particle size distribution is recommended for particles larger than 20 nm since this minimizes larger, multiply charged particles in the calibration aerosol; it is less important for smaller particles due to their lower probability of multiple charging. This recommendation is valid for both reference instruments, FCAE and CPC.

5.4.1.2 Aerosol generator

The suitability of a generator type depends on the required calibration particle material. Examples for combinations of aerosol generators and calibration particle materials are:

- a) Arc-plasma generator for metal, metal oxide or carbon particles; [6] [7]
- b) Electrospray aerosol generator for oil droplets, poly-alpha-olefin (PAO) droplets, or sucrose particles;[16]
- c) Evaporation condensation aerosol generator[5][10][51][53] for metallic particles like Ag, Au and salt particles like NaCl, KCl, ammonium nitrate, etc.;
- d) Quenched flame aerosol generator for flame soot particles;[32][56]
- e) Spray atomizer generators for solutions and dispersions; [19][43]
- f) Glowing wire generator for uniformly sized metal or metal oxide particles.[49]

In addition, the single charged aerosol reference (SCAR) is used for generating singly charged particles over a wide size range. [60]

5.4.1.3 Aerosol conditioner

The aerosol conditioner is used to control the state of the calibration particle material. Which of the following conditioning steps are necessary depends on the chosen aerosol generation method and calibration particle type.

- a) Adapting the primary aerosol number concentration and aerosol flow rate to an appropriate level for reliable charge conditioning (Annex K). For aerosol dilution setups see Annex F.
- b) Pre-classifying the primary aerosol particles using an additional charge conditioner and DEMC for example if the particle size distribution of the primary aerosol source has too large a fraction of multiply charged particles.[57]
- c) The vapour contents (from water, other dispersion media and/or solvents) in the primary aerosol shall be less than 40 % of the saturated value. High vapour contents in the primary aerosol can lead to condensational growth of the calibration particles, change the equilibrium charge distribution after the bipolar charger, and build-up vapour in the sheath flow loop of the DEMC. This can be achieved by dilution with dry air or vapour adsorption (e.g. with silica gel, zeolites or calcium chloride).

5.4.2 Charge conditioner

In order to achieve a stable, repeatable and reproducible calibration aerosol after the electrostatic size classification in the DEMC, the conditioned primary aerosol entering the DEMC shall have a stable, repeatable and reproducible charge distribution. Unipolar and bipolar chargers produce the ion concentration required to stabilize the charge distribution of the primary aerosol (see e.g. ISO 15900).

Sources with alpha or beta radiation can be used as bipolar chargers. If under appropriate operating conditions the equilibrium charge level is established, the charge distribution according to ISO 15900 shall be applied. Other bipolar chargers may be used if the equivalence to radioactive sources has been proven or the charge distribution has been fully characterized.

For primary aerosol with a mode size larger than 20 nm or with a non-monodisperse size distribution bipolar chargers shall be used. In these cases the equilibrium charge distribution has (compared to a unipolar charger) a significantly lower fraction of multiply charged particles.

If the primary aerosol is already monodisperse or if the primary aerosol does not contain particles larger than 20 nm, all calibration aerosol particles leaving the DEMC are singly charged no matter which type of charge conditioner is used upstream the DEMC. Therefore, in this case, unipolar (e.g. corona discharge device) or bipolar chargers may be used.

NOTE The SCAR[60] is an exception because it already generates singly charged particles over a wide size range.

5.4.3 **DEMC**

The DEMC classifies the conditioned primary aerosol particles based on their electrical mobility. It delivers calibration aerosols in a narrow mobility band, either positively or negatively charged. If classified particles carry more than one electrical charge, each charge level corresponds to a different particle size.

The DEMC shall be set up, operated, and calibrated according to ISO 15900.

Ideally, the primary aerosol fed into the DEMC is conditioned in such a way that only singly charged particles leave the DEMC to be used as calibration aerosol. In this case, the calibration aerosol is monodisperse.

If, due to the nature of the conditioned primary aerosol, the calibration aerosol also contains larger, multiply charged particles, corrections shall be applied and the measurement uncertainty may increase. Details about the necessary corrections are given in <u>Annex D</u>.

5.4.4 Make-up or bleed air

Additional make-up air is necessary if the calibration aerosol flow from the DEMC is lower than the sum of the flow rates required by the test CPC and the reference instrument.

The make-up air shall be practically particle free; the particle number concentration should be less than 0.1 cm^{-3} . This can be achieved with a HEPA filter with 99,995 % efficiency (or better).

The relative humidity of the make-up air shall be less than 40 %.

To avoid excessive variations in the number concentration of the calibration aerosol the make-up air flow shall be kept sufficiently stable.

If the calibration aerosol flow from the DEMC is higher than the sum of the flow rates required by the test CPC and the reference instrument, the excess air should be vented off as bleed flow. In this case, the operator should be protected from particle exposure by an exhaust particle filter.

5.4.5 Mixing device, flow splitter and connection tubing

The mixing device, flow splitter, and connecting tubing deliver the calibration aerosol to the test CPC and the reference instrument. The aerosol should have identical size distribution and number concentration when it reaches both instruments.

Concentration bias resulting from poor mixing is a major source of error in CPC calibration. Baffle plates, mixing chambers, and mixing orifices are examples for proven devices to avoid this bias.

The flow splitter divides the calibration aerosol flow coming from the mixing device into two flows, one to the test CPC and one to the reference instrument. Ideally, the flow splitter and the connection tubing are designed in such a way that the particle size dependent transport losses from the inlet of the flow splitter to both instruments are equal.

If the inlet flow rate of both instruments is equal, exchanging the position of the test CPC and the reference instrument can be used to demonstrate the equivalence of both sampling positions (see Annex G). The differences from each position shall be less than 5 %. The bias correction factor β represents the particle-loss compensated calibration result.

If the inlet flow rate of both instruments cannot be operated at the same flow rate, the length of the connection tubing shall be used to compensate the difference in the transfer losses. The ratio of the different inlet flow rates shall not be larger than 5 or smaller than 0,2. A calibration setup with such different inlet flows does not allow evaluating experimentally transport losses by exchanging the instrument positions. Therefore the uncertainty of measurement is increased by the uncertainty of each flow rate.

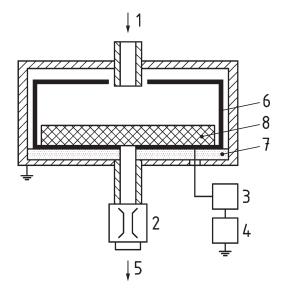
The design of the mixing device, the flow splitter, and the connection tubing shall follow good engineering practices, such as avoiding bends, avoiding sudden change of tubing diameters. Use conductive tubing and provide sufficient electrical grounding for all connections, especially for flexible tubing if it cannot be avoided.

5.4.6 Reference instrument: FCAE or CPC

a) Design and operation of an FCAE

The 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 aerosol particles, an electrical connection between the sensing element and an electrometer circuit, and a flow meter, as shown in Figure 3.

NOTE The efficiencies for an FCAE are expected to be greater than 95 % for particles covered by this International Standard (>5 nm) and FCAE flow rates greater than 1 l/min.



Key

- 1 charged particles
- 2 flow meter
- 3 preamplifier
- 4 electrometer
- 5 particle-free air
- 6 Faraday cup to collect electrical charge from charged collected particles and to reduce externally-induced electro-magnetic noise
- 7 very-high-resistance electrical insulator to isolate the filter from electrical ground
- 8 high-efficiency particulate air (HEPA) filter to trap airborne charged particles

Figure 3 — Schematic diagram of an FCAE (Adapted from ISO 15900)

b) Design and operation of a reference CPC

The reference CPC shall have a design such that particles in the entire aerosol flow entering the inlet are counted. That is, the aerosol flow sampled through the inlet shall not be diluted or filtered and the total inlet flow shall be led to the optics.

The concentration range for single particle counting mode shall be established from the manufacturer's specifications. The reference CPC shall not be used in photometric mode.

The lower size limit $d_{\min, ref}$ for the application of the reference CPC for calibration of test CPCs is the smallest value of at least three diameters, the two larger diameters are at least 2 and 3 times $d_{\min, ref}$. The three respective efficiency values may not differ more than 5 % from the largest of the three efficiency values. The detection efficiency of the reference CPC shall be documented for the relevant particle type in such a way that its $d_{\min, ref}$ is determined or can be derived from the documented detection efficiencies.

c) Certificates for FCAE and reference CPC

The reference instrument shall have a current, reputable calibration certificate as defined in $\underline{5.1}$. Examples of calibration certificates are given in $\underline{\text{Annex C}}$.

The calibration certificate for the FCAE shall specify the aerosol flow rate measurement (volumetric flow rate, inlet pressure and inlet temperature) and the electrical charge concentration measurement. The product of charge concentration times aerosol flow rate is typically in the range from 1 fC/s to 10 fC/s.

The calibration certificate for the reference CPC can be the result of either a calibration against a traceable FCAE, or a calibration with another CPC used as a reference instrument. It shall specify the particle type, the particle sizes, the particle number concentrations for which the calibration is valid, and the maximum concentration of the reference CPC in single counting mode. Volumetric inlet flow rate, inlet pressure and inlet temperature at the time of calibration shall also be specified.

The reference instrument detection efficiency can change when internal surfaces become loaded with particles. In addition to the certificate being within its specified time period, the reference instrument shall have had limited accumulated exposure to such particles since calibration. The use of the reference instrument shall therefore be logged, and internal procedures shall be set up to ensure that the accumulated exposure to particles since calibration does not significantly affect the performance of the reference instrument.

The reference instrument shall be recertified

- after maintenance or repair,
- after detection of significant drift in the reference instrument, through QA/QC procedures set up by the user,
- after accumulated exposure to particles reaches a predefined level, to be established by the user, or
- if a period of 3 years has passed since the last certification.

5.4.7 Other tools

The following additional sensors are used for CPC calibration and shall be calibrated using reference instruments that deliver results that are traceable to internationally accepted standards:

- low pressure drop flow meter for checking and setting instrument and calibration flows;
- pressure sensor for measuring the pressure of the calibration aerosol;
- gas temperature sensors for measuring several temperatures;
- humidity sensor is necessary to measure the relative humidity of the primary aerosol.

5.5 Differences between FCAE and CPC as a reference instrument

As a reference instrument FCAE and CPC have different capabilities and demand different requirements. This subclause describes the differences in order to choose the appropriate method for the specific purpose.

a) Lower size detection limits

In general an FCAE can detect charged particles smaller than the particles detected by a CPC. Furthermore, the efficiency curve of the CPC depends to some extent on the details of experimental conditions and particle type. Therefore, a reference CPC shall only be used for the calibration with monodisperse particles with diameters larger than $d_{\min,\text{ref}}$ of the reference CPC. For calibration with particles with broader size distributions, it shall be avoided that significant numbers of the test particles have a size similar to or smaller than $d_{\min,\text{ref}}$ of the reference CPC. Therefore, the median diameter of a polydisperse test aerosol shall be larger than or equal to $d_{\min,\text{ref}}$ times the geometric standard deviation of the size distribution of the test aerosol.

b) Particle charging

For an FCAE comparison, the calibration aerosol needs to consist of singly charged particles, or at least have a known charge distribution with a small fraction of multiply charged particles. This requirement also applies for a reference CPC comparison at the lower size detection limit of a test CPC, since particles with multiple charges have a larger diameter and are counted with a higher efficiency. For a reference CPC comparison at larger particle diameters in the plateau region of the test CPC, this particular

requirement does not apply. Therefore, the use of a reference CPC for calibration with larger particles can result in smaller uncertainties, as their higher probability of multiple charging is less important.

c) Low calibration aerosol particle number concentration

For an FCAE comparison, there is a minimum requirement for the number concentration of charged particles exiting the DEMC (typically about $10^3 \, \mathrm{cm}^{-3}$) to provide sufficient charge concentration to the electrometer. This is not needed for a reference CPC comparison if the linearity of the reference CPC is proven.[44] A method demonstrating validation at lower concentrations than those on the reference instrument certificate is described in Annex H.

d) High calibration aerosol particle number concentration

An FCAE will cover its specified range of particle concentrations using one principle — determining the electrical charge concentration. A CPC can have two or more fundamentally different measurement modes, starting with a simple optical counting mode (single particle counting mode) at low concentrations, with coincidence correction at higher concentrations, and a light intensity-based particle concentration mode at the highest concentrations (sometimes known as photometric mode). The photometric mode is more liable to change in sensitivity due to contamination of the optics, for example. Reference CPCs in this International Standard are therefore constrained to operate in their single particle counting modes (with or without coincidence correction), and the upper number concentration limit will generally be lower than for the FCAE method.

Calibration with a reference CPC therefore has disadvantages compared with an FCAE at low particle size or high particle number concentration. It has the advantages with larger particles, fewer constraints on the calibration aerosol source, and at lower particle concentrations. The differences between the calibration aerosol that can be used with the FCAE and reference CPC are summarized in Table 1.

	Particle	Typical particle	Calibration aerosol parti	cle charge requirements
Reference instrument	diameter range [nm]	concentration range [cm ⁻³]	Test CPC calibrated at lower detection limit particle diameters Test CPC calibrated in plateau range of particle diameters	
FCAE	5 – 1 000	$10^3 \text{ to} > 10^5$	φ < 0,1	<i>φ</i> < 0,1
CPC	d _{min,ref} - 1 000	~1 to > 10 ⁴	φ < 0,1	no restriction

Table 1 — Calibration aerosol requirements for the FCAE and reference CPC

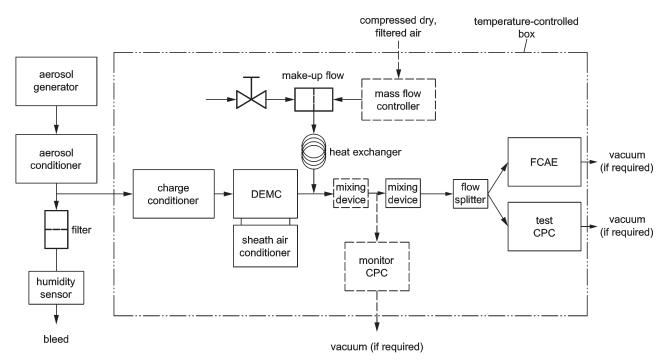
6 Calibration using an FCAE as reference instrument

6.1 Overview of the setup and calibration procedure

A schematic for a typical calibration setup using an FCAE as reference instrument is given in Figure 4. All parts drawn with solid lines are necessary components (see discussion in Clause 5). These include the aerosol generator, the aerosol conditioner, the humidity sensor to measure the humidity of the aerosol that enters the DEMC, the charge conditioner, the DEMC, the make-up flow, the mixing device, the flow splitter, the FCAE, and the test CPC. Although not shown in the figure a relative humidity sensor shall be used to measure the relative humidity of the make-up air at the beginning and end of the experiments. A pressure sensor might also be necessary for the determination of the volumetric flow rate of the instruments.

In case that the calibration aerosol flow from the DEMC is higher than the sum of the flow rates required by the instruments, the excess air shall be vented off as bleed flow. While it is not shown in the figure, a temperature sensor shall be used to monitor the temperature in the temperature-controlled box or room temperature.

The parts of Figure 4 with dashed lines are recommended but not required. For example the temperature-controlled box and heat exchangers for the DEMC sheath air flow and the make-up air flow can be used to stabilize all temperatures. A monitor CPC can be used to check the stability of the calibration aerosol. The make-up air flow can be controlled with a throttle valve or compressed air with a mass flow controller.



NOTE Dashed lines show equipment that is not required, but highly recommended.

 $Figure\ 4-Typical\ calibration\ setup\ with\ FCAE\ as\ reference\ instrument$

The calibration procedure can be seen in $\underline{\text{Table 2}}$.

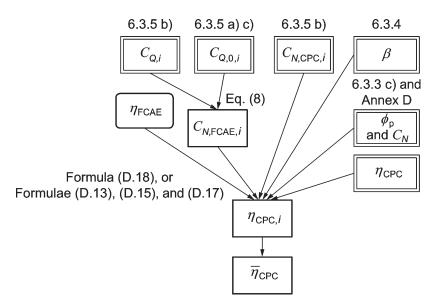
Table 2 — CPC calibration procedure using an FCAE as reference instrument

<u>6.2</u>	Preparation
6.2.2	Aerosol generator and conditioner (size distribution)
6.2.3	Other equipment (e.g. mass flow meters, etc.)
6.2.4	DEMC (according to ISO 15900) and sheath air conditioner
6.2.5	FCAE
	Absolute zero corrected value for charge concentration times inlet flow rate < 1 fC/s,
	standard deviation <2,5 fC/s (from 1-s averages of 15 min)
	Overall leak test
	Flow measurement and stability <2 % (from 5 measurements in 15 min)
6.2.6	Test CPC
	Zero arithmetic mean <0,1 cm ⁻³ (from 1-s average concentrations of at least 5 min)
	High response check
	Flow measurement and stability < 2 % (from 5 measurements in 5 min)
6.2.7	Connect the instruments and the aerosol generator/conditioner to the DEMC
	DEMC (voltage off), sheath to sample >7:1
	FCAE flow measurement
	FCAE absolute zero corrected value for charge concentration times inlet flow rate <1 fC/s,
	standard deviation <0,5 fC/s (from 30-s arithmetic means of 2 min)
	Test CPC zero arithmetic mean <1 cm ⁻³ (from 30-s arithmetic means of 2 min)
	Determine minimum level of FCAE charge concentration times inlet flow rate with Formula (5)

Table 2 (continued)

<u>6.3</u>	Detection efficiencies
6.3.2	DEMC diameter adjustment
6.3.3	Primary aerosol adjustment
	The concentration to be within the capability of the charge conditioner
	Multiply charged particles fraction < 10 %
	Concentration within the range of the FCAE
6.3.4	Splitter bias $oldsymbol{eta}$ measurement
<u>6.3.5</u>	Test CPC efficiency measurement
	Set DEMC voltage 0 (or off) for initial zero measurement
	 Record 1 min FCAE, test CPC, use the last 30 s for the calculations
	— FCAE absolute zero corrected arithmetic mean <1 fC/s
	— FCAE standard deviation <0,5 fC/s
	— CPC arithmetic mean <1 cm ⁻³
	At the specific diameter and concentration:
	 Record 1 min FCAE, test CPC, use the last 30 s for the calculations
	— FCAE (CPC) CV < 3 %, or standard deviation < 0,5 fC/s (0,5 cm $^{-3}$)
	Set DEMC voltage 0 (or off)
	 Record 1 min FCAE, test CPC, use the last 30 s for the calculations
	 FCAE absolute zero corrected arithmetic mean <1 fC/s
	— FCAE standard deviation <0,5 fC/s
	— CPC arithmetic mean <1 cm ⁻³
	Calculate detection efficiency $\eta_{ ext{CPC},i}$
	Repeat another 4 times
	Calculate the arithmetic mean detection efficiency $\bar{\eta}_{\mathrm{CPC}}$. All $\eta_{\mathrm{CPC},i}$ shall be within $\bar{\eta}_{\mathrm{CPC}}$ ± 0,02
6.3.6	Measurement of a different concentration (optionally)
	Go to <u>6.3.3</u> and then <u>6.3.5</u>
6.3.7	Measurement of a different size (optionally)
	Go to <u>6.3.2</u>
6.3.8	Repetition of first measurement
	If > 5 points have been tested (difference has to be within 0,025)
<u>6.3.9</u>	Fill in the calibration certificate

Figure 5 provides a graphical summary of the derivation of the detection efficiency.



NOTE Plain rectangles represent calculated values while double-lined and round-cornered rectangles are for measured values and values taken from certificates, respectively.

Figure 5 — A map of parameters and formulae needed to derive the detection efficiency in calibration with an FCAE

Annex I gives an example of a protocol for the calibration of a CPC using an FCAE as the reference instrument.

6.2 Preparation

6.2.1 General preparation

Check that all instruments operate properly according to the manufacturers' specifications (see 6.2.2 to 6.2.6), then prepare (according to Figure 4) and check the complete setup (6.2.7). Do not proceed to the calibration procedure of detection efficiency (6.3) unless all tests have been passed.

6.2.2 Primary aerosol

Start the operation of the primary aerosol source according to the manufacturer's recommendations. It is highly recommended to measure the generated size distribution after the aerosol conditioner with a DMAS (e.g. combine DEMC with FCAE) if it is not known. Ensure that the relative vapour contents (from water and/or solvents) in the primary aerosol is less than 40 %.

6.2.3 Other equipment

Switch on and allow all auxiliary necessary equipment to stabilize. Turn on the charge conditioner if it is off. Prepare the calibrated pressure sensor(s), the calibrated temperature sensor(s), and the calibrated flow meter for the measurement of the FCAE and test CPC flow rates and the humidity sensors.

Any other recommended instruments in the setup should be also turned on and prepared according to the manufacturers' manuals (e.g. a monitor CPC, mass flow meters, mass flow controllers, pressure and temperature sensors, etc.). If the whole setup (i.e. DEMC and instruments) is in a temperature-controlled box, set the desired temperature and leave the system time to stabilize.

6.2.4 DEMC

Turn on the instrument, check the DEMC according to ISO 15900, and set the desired flows. Prepare the conditioner of the sheath flow.

6.2.5 FCAE

Turn on the FCAE. Leave it running for at least 30 min. If switched on after transport or a longer period without use, it may require several hours of operation until it reaches the necessary stability.

NOTE Ideally the FCAE's electrometer electronics should remain turned on all time.

Check the zero level and flow rates of the FCAE at ambient conditions. All indicators (e.g. for temperatures, flows and pressures) shall show error-free operation of the instrument. The following checks ensure that the instrument is working properly before connecting it to the calibration setup.

a) Zero check

Zero the FCAE, following the advice in the FCAE's user manual or following an appropriate external zeroing method. Report the chosen method in the Calibration Certificate.

Attach a HEPA filter (>99,99 % efficiency) to the inlet of the FCAE. Record the FCAE's zero-corrected value for charge concentration times inlet flow rate ($C_{Q \times qFCAE}$) for at least 15 min with 1-s reading interval and 1-s averaging time. There should be no obvious decreasing or increasing tendency of the value. If there is such a tendency, allow more time for the FCAE to stabilize, repeat FCAE zeroing, and repeat this test. Do not proceed unless the FCAE reaches stable operation.

Calculate the arithmetic mean and standard deviation for the recorded values. As a result of a successful zero-correction the FCAE's zero-corrected absolute arithmetic mean value for $C_Q \times q_{\text{FCAE}}$ shall be < 1 fC/s. The standard deviation shall be less than 2,5 fC/s.

The FCAE requires attention from the manufacturer if the FCAE fails twice this zero test.

b) FCAE overall leak test

For this test, the ambient concentration should be at least 500 cm^{-3} . This test is not applicable for FCAEs with internal bypass flow, e.g. to allow operation conditions for a flow control valve.

- 1) Connect a HEPA filter to inlet of the test CPC. Let the test CPC zero for 1 min, then measure the number of counts over 1 min as $N_{\rm HEPA}$.
- 2) Disconnect the HEPA filter from the test CPC, then sample the room air and measure the number of counts over 1 min as N_{ambient} .
- 3) Disconnect the vacuum from the test CPC and connect the inlet of the test CPC to the outlet of the FCAE. Connect the HEPA filter to the inlet of the FCAE so that it will sample filtered room air. Connect a vacuum to the test CPC to pull filtered room air through the FCAE.
- 4) Wait 3 min to zero the system.
- 5) Measure and record the number of counts over 1 min as the test CPC pulls filtered ambient air through the FCAE. Record the number counted by the CPC as N_{FCAE} .
- 6) Calculate the value $N_{\text{leak}} = N_{\text{FCAE}} N_{\text{HEPA}}$. If this value is negative, then use $N_{\text{leak}} = 0$.
- 7) Calculate the ratio $R_{\text{FCAE}} = N_{\text{leak}}/N_{\text{ambient}}$. The value of R_{FCAE} shall be less than 0,000 1 to perform the calibration. If $R_{\text{FCAE}} > 0,000$ 1, check the FCAE plumbing for leaks, verify that the filter is adequate, and perform steps 1 to 7 again.
- c) Flow rate measurement

If the inlet flow rate of the FCAE is adjustable, set it to the nominal value specified in the calibration certificate for which the FCAE calibration is valid before the flow rate measurement.

Measure the nominal (see calibration certificate) volumetric inlet flow rate of the FCAE at ambient conditions with an appropriate low pressure drop, calibrated flow meter (Annex J). The flow shall be stable over time, i.e. the CV of at least 5 measurements uniformly spaced over 15 min shall be <2 %. There should be no obvious decreasing or increasing tendency of the flow. If not fulfilled, leave the

FCAE more time to stabilize, check the pump (or vacuum connection) of the FCAE and repeat. The FCAE requires attention from the manufacturer if the flow check fails twice.

Compare the (calculated) arithmetic mean of the measured inlet flow rates of the FCAE ($q_{\text{FCAE},\text{cal},\text{amb}}$) with the arithmetic mean value indicated by the FCAE for the same time intervals or with the nominal value of the FCAE ($q_{\text{FCAE},\text{amb}}$). The latter case applies if no flow rate is reported or when the nominal value is used by the FCAE for the charge concentration calculation. The difference should be within the FCAE manufacturer's specifications, indicated as an accuracy $r_{q,\text{FCAE}}$ in %. If not, the manufacturer should be contacted.

The flow rate $q_{\text{FCAE,cal,amb}}$ should also be compared with the flow rate of the FCAE in its calibration certificates ($q_{\text{FCAE,cert}}$) and the deviation shall be within:

$$\frac{\left|q_{\text{FCAE,cal,amb}} - q_{\text{FCAE,cert}}\right|}{q_{\text{FCAE,cert}}} < 2\sqrt{u_{\text{r}}^2(q_{\text{FCAE,cert}}) + u_{\text{r}}^2(q_{\text{cal,cert}}) + \frac{1}{3}r_{q,\text{FCAE}}^2}$$
(2)

where

 $u_r(q_{FCAE,cert})$ is the relative standard uncertainty for the inlet flow of the FCAE in its calibration certificate:

 $u_{\rm r}(q_{\rm cal,cert})$ is the relative standard uncertainty of the flow meter used to measure $q_{\rm FCAE,-}$ cal,amb.

Higher deviations might indicate issues with the flow control of the FCAE.

All flow rates shall refer to the same temperature and pressure. Depending on the flow control used in the FCAE, different corrections should apply (see <u>Annex J</u>).

6.2.6 Test CPC

When a CPC has been transported for calibration, it will generally have been drained of working fluid. In that case, switch the test CPC on, fill with the required working fluid to the specified level (observing manufacturer's precautions regarding moving the unit when full) and allow the saturator, condenser and optics to reach their specified temperatures. Leave it running for at least 1 h.

When a CPC is to be calibrated without being drained of the working fluid, manufacturer's precautions regarding moving the unit when full shall be observed. Switch on the test CPC, leave it running for at least 30 min, and allow the saturator, condenser and optics to reach their specified temperatures.

All indicators (e.g. for temperatures, flows and pressures) shall show error-free operation of the instrument.

a) Zero count check

For zero count check, attach at least one HEPA filter (>99,99 % efficiency) to the test CPC inlet (an additional HEPA filter in series with the first one may be necessary to achieve extremely low concentrations). Run the CPC for a minimum of 5 min and record the concentration values with 1-s reading interval and 1-s averaging time. After any leaks are eliminated, the arithmetic mean concentration shall be $<0,1~\rm cm^{-3}$. Contact the customer if this requirement is not met.

b) High response check

Perform a simple check to demonstrate that the test CPC can detect particles. This can, for example, be done by sampling room air if the number concentration of the room air is expected to be higher than 500 cm⁻³. The number concentration measured by the test CPC should be higher than 500 cm⁻³. Aerosols from other sources with sufficiently high number concentrations may also be used for this test. Or, follow the manufacturer's recommendations. Contact the customer if this requirement is not met.

c) Flow rate measurement

Measure the volumetric inlet flow rate of the test CPC at ambient conditions with an appropriate low pressure drop, calibrated flow meter (Annex I). The flow shall be stable over time, i.e. the CV of at least 5 measurements uniformly spaced over 5 min shall be <2 %. There should be no obvious decreasing or increasing tendency of the flow. If not fulfilled, leave the test CPC more time to stabilize, check the pump (or vacuum connection) of the test CPC and repeat. The test CPC requires maintenance if the flow check fails twice.

Compare the (calculated) arithmetic mean of the measured inlet flow rates of the test CPC ($q_{CPC,cal,amb}$) with the arithmetic mean value indicated by the test CPC for the same time intervals or the nominal value for the test CPC ($q_{CPC,amb}$). The latter case applies if no flow rate is reported or when the nominal value is used by the test CPC for the particle number concentration calculation. The difference should be within the test CPC manufacturer's specifications. If not, the customer should be contacted. Higher differences might indicate issues with the flow control of the test CPC.

All flow rates shall refer to the same temperature and pressure. Depending on the flow control used in the test CPC, different corrections should apply (see <u>Annex J</u>).

6.2.7 Check of the complete setup

Initially the make-up flow path is connected downstream of the DEMC (typically a HEPA filter, or a mass flow controller with a HEPA filter). Then connect the mixing device and pressure sensor. The test CPC and FCAE are connected to the flow splitter which is positioned after the mixing device. Make sure that there is at least one opening free (e.g. the inlet of the DEMC or the make-up flow path) in order to avoid overpressure or underpressure at the inlet of the test CPC and FCAE. If there is a monitor CPC, this should be connected before the mixing device with another mixing device.

Connect the aerosol generator and conditioner to the inlet of the DEMC, making sure that the excess flow is vented, or that filtered air is added if the DEMC flow rate is higher.

Check if the pressure at the inlet of the FCAE and test CPC has remained in the desired range (i.e. no extreme underpressure or overpressure, outside the manufacturers' specifications). If not, adjust accordingly the make-up air flow rate or the throttling valve.

a) DEMC flow rate

Set the desired DEMC sheath flow rate. If required, set the desired DEMC inlet flow rate by adjusting the make-up flow (or bleed air). The ratio of sheath to sample flow rates shall be \geq 7:1 to ensure narrow monodisperse distribution after the DEMC.

After setting these flow rates, it is recommended to not adjust them during the calibration procedure. If adjusted the volumetric flow rates of the FCAE and the test CPC have to be measured again (see steps b) and c) below).

b) FCAE flow measurement

Measure the volumetric flow rate of the FCAE by inserting the calibrated flow meter between the splitter and the inlet of the FCAE. Compare this measured value ($q_{FCAE,cal}$) with the value reported by the FCAE or its nominal value (q_{FCAE}). The latter case applies if no flow rate is reported or when the nominal value is used by the FCAE for the charge concentration calculation. The difference should be within the FCAE manufacturer's specifications, indicated as an accuracy $r_{q,FCAE}$ in %. If not the manufacturer should be contacted.

The flow rate $q_{\text{FCAE,cal}}$ should also be compared with the flow rate of the FCAE in its calibration certificate $(q_{\text{FCAE,cert}})$ and the deviation shall be within:

$$\frac{\left|q_{\text{FCAE,cal}} - q_{\text{FCAE,cert}}\right|}{q_{\text{FCAE,cert}}} < 2\sqrt{u_{\text{r}}^2(q_{\text{FCAE,cert}}) + u_{\text{r}}^2(q_{\text{cal,cert}}) + \frac{1}{3}r_{\text{q,FCAE}}^2} \tag{3}$$

where

 $u_r(q_{FCAE,cert})$ is the relative standard uncertainty for the inlet flow of the FCAE in its calibration certificate;

 $u_r(q_{cal,cert})$ is the relative standard uncertainty of the flow meter used to measure $q_{FCAE,cal}$.

Higher deviations might indicate issues with the flow control of the FCAE.

The relative standard uncertainty of q_{FCAE} is calculated as Formula (4):

$$u_{\rm r}(q_{\rm FCAE}) = \left| q_{\rm FCAE,cal} - q_{\rm FCAE,cert} \right| / \sqrt{3} \, q_{\rm FCAE,cert} \tag{4}$$

All flow rates shall refer to the same temperature and pressure. Depending on the flow control used in the FCAE, different corrections should apply (see <u>Annex J</u>).

NOTE 1 The flow rate is affected by the composition of the gas. For more details, see Annex J.

NOTE 2 The flow measurement is repeated here to account for flow rate changes due to changed FCAE inlet pressure.

c) Test CPC flow rate measurement

Measure the volumetric flow rate of the test CPC by inserting the calibrated flow meter between the splitter and the inlet of the test CPC. Compare this measured value with the value reported by the test CPC or its nominal value. The latter case applies if no flow rate is reported or when the nominal value is used by the test CPC for the particle number concentration calculation. The difference should be within the test CPC manufacturer's specifications. If not the customer should be contacted. Higher differences might indicate issues with the orifice or the pump of the test CPC. This value shall be reported in the calibration certificate, along with the test CPC reported or nominal value.

All flow rates shall refer to the same temperature and pressure. Depending on the flow control used in the test CPC, different corrections should apply (see <u>Annex I</u>).

NOTE 1 The flow rate is affected by the composition of the gas. For more details, see Annex J.

NOTE 2 The flow measurement is repeated here to account for flow rate changes due to changed test CPC inlet pressure.

d) Zero levels

Set the DEMC voltage to 0 V (or off). The FCAE and test CPC readings shall remain comparable to the zero levels measured before [6.2.5] a) and 6.2.6 a)]: measure for a minimum of 2 min, the absolute, zero corrected 30-s arithmetic mean value (with 1-s reading interval) of the FCAE shall be lower than $C_{Q \times q\text{FCAE}} = 1$ fC/s and the standard deviation shall be lower than 0,5 fC/s. The arithmetic mean value (with 1-s reading interval) of the CPC shall be lower than $C_{N,\text{CPC}} = 1$ cm⁻³. If not, check for leaks in the calibration setup. Other reasons for increased zero levels can be — for example — an excessively high concentration at the inlet of the DEMC, overloading or failure of the filters inside the DEMC.

e) Determine the FCAE minimum level

Determine the arithmetic mean and standard deviation of $C_{Q \times q\text{FCAE}}$ reported by the FCAE in 30 s (at 1-s reading interval). Multiply the standard deviation by 3 and add the value:

$$(C_{Q \times q_{\text{FCAE}}})_{\min} = (C_{Q \times q_{\text{FCAE}}})_{\max} + 3\sigma_{C_{Q \times q_{\text{FCAE}}}}$$
 (5)

Compare this value with the lowest value of $C_{Q \times q\text{FCAE}}$ in its calibration certificate. The greater one is the minimum $C_{Q \times q\text{FCAE}}$ at the inlet of the FCAE that may be used in the calibration (defined here as the "minimum $C_{Q \times q\text{FCAE}}$ level").

Write down all parameters: Readings of the FCAE and test CPC, flow rates, pressures, temperatures, make-up flow (if available), sheath, sample flow rates, humidity, etc. All this information will be reported in the calibration certificate (see <u>Clause 8</u> and <u>Annex C</u>).

6.3 Calibration procedure of detection efficiency

6.3.1 General

The following procedure describes the measurement of the detection efficiency of a test CPC at one given calibration particle size and particle number concentration.

6.3.2 DEMC diameter adjustment

Adjust the DEMC such that the particle size of singly charged calibration particles leaving the DEMC equals the desired calibration particle size.

NOTE It is highly recommended to begin with a big size (such that the detection efficiency of the test CPC is at the maximum, i.e. at least 3 times the size where the detection efficiency is expected to be 50 %) and check the linearity of the test CPC (i.e. different concentrations at the same size). Then the detection efficiency at the steep part of the detection efficiency curve may follow. This is because the tests at the steep part of the detection efficiency curve usually need adjustment of the size distribution produced by the generator and are more time consuming.

6.3.3 Primary aerosol adjustment

Adjust the aerosol conditioner in such a way that the concentration of the calibration aerosol equals the desired concentration for the measurement of the detection efficiency. However the following requirements shall be fulfilled.

a) Minimum level

The concentration shall be higher than the value $(C_{Q \times qFCAE})_{min}$ of the FCAE, as defined in Formula (5) in <u>6.2.7</u> e).

b) Maximum level

The concentration shall be lower than the maximum determined by the maximum charge concentration the FCAE may measure, as specified in its calibration certificate.

The total number concentration of the conditioned primary aerosol shall be low enough to reach charge equilibrium after passing through the charge conditioner and to avoid particle charge caused bias in the DEMC. A method to check this is described in $\underbrace{Annex\ K}$.

c) Fraction of multiply charged particles Φ

The value of multiply charged particles Φ shall be <0,1. Determine the fractions of particles of p charges $[(\phi_p, \text{Formula (6)}]]$ according to one of the methods described in Annex D, and calculate the fraction of multiply charged particles (Φ) with Formula (7). The criterion Φ < 0,1 is a pass/fail criterion; the calibration procedure shall not be continued until it is fulfilled. The Φ can be decreased by, e.g. adjusting the mode diameter or the geometric standard deviation of the size distribution of the primary aerosol.

The fraction of particles with p charges, ϕ_p , within the aerosol leaving the DEMC is calculated as

$$\phi_p = C_N(d_p) / \sum_{p \ge 1} C_N(d_p) \tag{6}$$

where

 $C_N(d_p)$ is the concentration of particles with p charges.

The fraction of multiply charged particles, Φ , is calculated as

$$\Phi = \sum_{p \ge 2} \phi_p \tag{7}$$

NOTE 1 Depending on the polarity of the voltage in the DEMC, the charges of the particles can be either positive or negative. In this International Standard, we define p as the absolute number of charges.

NOTE 2 It is highly recommended that the geometric mean size of the primary distribution is smaller than the size at which the detection efficiency of the test CPC will be measured.

NOTE 3 If a tandem DEMC setup is employed (i.e. two DEMCs in series with a charge conditioner in between), the fraction of multiply charged particles, ϕ , is reduced significantly.

NOTE 4 The fraction of multiply charged particles can be reduced by the method described in Reference [60].

6.3.4 Splitter bias β measurement

Perform the splitter bias measurement according to Annex G. If the obtained bias (β) is greater than 1,05 or less than 0,95, check for any inhomogeneity of the calibration aerosol.

6.3.5 Test CPC efficiency measurement

Determine the detection efficiency of the test CPC according to the following steps.

a) Initial reading at DEMC voltage 0 (or off)

Set the DEMC voltage to 0 V (or off) and record the FCAE charge concentration and the number concentration reported by the test CPC every second for 1 min. Calculate the arithmetic mean ($C_{Q,0,0}$) and standard deviation of the charge concentrations reported by the FCAE and the arithmetic mean of the number concentrations reported by the test CPC for the last 30 s of the 1-min measurement interval.

The absolute, zero corrected arithmetic mean value and the standard deviation of the FCAE charge concentration times its inlet flow rate shall be less than 1 fC/s and 0,5 fC/s, respectively. The arithmetic mean of the number concentration of the test CPC shall be $<1~cm^{-3}$. If not, the measurement is not valid. Check the generator or other sources of instability and repeat.

b) Recordings at specific size and concentration

Record the FCAE charge concentration and the number concentration reported by the test CPC every second for 1 min. Calculate the arithmetic mean ($C_{Q,1}$) and standard deviation of the charge concentrations reported by the FCAE and the arithmetic mean ($C_{N,CPC,1}$) and standard deviation of the number concentrations reported by the test CPC for the last 30 s of the 1-min measurement interval (Annex L).

The CV of the FCAE charge concentration times its inlet flow rate shall be <3 % or the standard deviation <0.5 fC/s. The CV of the number concentration of the test CPC shall be <3 % or the standard deviation <0.5 cm⁻³. Either the CVs or the standard deviations shall respectively fulfil the criterion easiest to comply with. If not, the measurement is not valid. Check the generator or other sources of instability and repeat.

c) Recordings at DEMC voltage 0 (or off)

Set the DEMC voltage to 0 V (or off) and record the FCAE charge concentration and the number concentration reported by the test CPC every second for 1 min. Calculate the arithmetic mean ($C_{Q,0,1}$) and standard deviation of the charge concentrations reported by the FCAE and the arithmetic mean of the number concentrations reported by the test CPC for the last 30 s of the 1-min measurement interval.

The absolute, zero corrected arithmetic mean value and the standard deviation of the FCAE charge concentration times its inlet flow rate shall be less than 1 fC/s and 0,5 fC/s, respectively. The arithmetic mean of the number concentration of the test CPC shall be <1 cm $^{-3}$. If not, the measurement is not valid. Check the generator or other sources of instability and repeat.

d) Calculation of FCAE number concentration assuming singly charged particles

Calculate the number concentration by FCAE ($C_{N,FCAE,1}$) according to Formula (8). From the measured charge concentration (corrected for zero) and assuming that particles each carry a single electrical charge, the number concentration for measurement i is given by:

$$C_{N,\text{FCAE},i} = \frac{C_{Q,i} - (C_{Q,0,i-1} + C_{Q,0,i})/2}{e}$$
(8)

where

 $C_{N,FC,AE,i}$ is the calculated number concentration of the calibration aerosol;

 $C_{O,i}$ is the indicated charge concentration measured by the FCAE when measuring particles;

 $C_{Q,0,i}$ is the indicated charge concentration measured by the FCAE with the DEMC voltage set at zero;

e is the elementary charge.

e) Calculation of the detection efficiency of the test CPC

If the primary aerosol cannot be conditioned in such a way that the calibration aerosol contains one elementary charge, corrections shall be applied ($\underline{Annex\ D}$).

Annex D also quantifies the effect of multiple charges on the determination of particle size.

Information from the FCAE certificate regarding any correction factor required by the FCAE shall be incorporated (Annex D).

Calculate the detection efficiency $\eta_{CPC,1}$ of the test CPC using formulae in Annex D.

- If the particle size is in the range where the detection efficiency of the test CPC is known to be constant against size, substitute $C_{N,\text{FCAE},1}$ [6.3.5 d)], $C_{N,\text{CPC},1}$ [6.3.5 b)], ϕ_p [6.3.3 c)], β (6.3.4) and η_{FCAE} (from the calibration certificate of the FCAE) into Formula (D.18).
- If the particle size is in the range where the detection efficiency of the test CPC is known to vary with size, substitute $C_{N,\text{FCAE},1}$ [6.3.5 d)], $C_{N,\text{CPC},1}$ [6.3.5 b)], ϕ_p (and C_N obtained in Annex D) [6.3.3 c)], β (6.3.4), η_{FCAE} (from the calibration certificate of the FCAE), and an estimated plateau efficiency η'_{CPC} of the test CPC into Formulae (D.13), (D.15) and (D.17).

Record in the calibration certificate which method of calculation was used.

f) Measurement repetition

Repeat steps b) to d) four more times (i.e. five in total). Calculate the arithmetic mean detection efficiency $\bar{\eta}_{CPC}$ and its standard deviation $\sigma(\eta_{rep})$ for the specific concentration and size using the five detection efficiencies $\eta_{CPC,i}$ (i=1-5). The calibration is valid only if all five $\eta_{CPC,i}$ values are within $\pm 0,02$ of the arithmetic mean detection efficiency $\bar{\eta}_{CPC}$

6.3.6 Measurement of different particle concentrations

If a different concentration (at the same size) has to be measured, adjust the concentration of the primary aerosol (go to 6.3.3 and then 6.3.5). The measurement of the fraction of multiply charged particles [6.3.3 c)] is not necessary to repeat. Note that the splitter bias (6.3.4) and the FCAE flow rate [6.2.7 b)] do not have to be tested. For concentrations lower than the maximum level, there is no need to re-check the charge conditioner [6.3.3 b)]. If the CPC is to be calibrated at concentrations below the lowest charge concentration at the nominal inlet flow rate for which the FCAE has been certified, the method described in Annex H can be followed.

If the DEMC flow rates have to be adjusted or if any change in the setup is made that can lead to a different pressure at the inlet of the FCAE and test CPC, measure the inlet flow rates of the FCAE and the test CPC (see <u>6.2.7</u>).

6.3.7 Measurement of different sizes

If a different particle size has to be tested, then go to 6.3.2. In this case, all following steps (6.3.3 to 6.3.6) have to be performed.

If the DEMC flow rates have to be adjusted or if any change in the setup are made that can lead to a different pressure at the inlet of the FCAE and test CPC, measure the inlet flow rates of the FCAE and the test CPC (see <u>6.2.7</u>).

NOTE 1 It is also highly recommended to conduct all detection efficiency tests on the same test CPC at various particle sizes at approximately the same particle number concentration. This would avoid any influence from a nonlinear CPC response.

NOTE 2 When planning the concentrations to be used, attention shall be paid to the concentrations at which the measurement mode of the test CPC changes (as stated in the manufacturer's manual), so that discontinuities can be anticipated.

6.3.8 Repetition of first measurement point

When several points are tested, or when a measurement instrument was switched off, or when a malfunction occurred, the detection efficiency measurement with the first set of aerosol parameters has to be repeated (i.e. concentration and size). This includes the determination of the fraction of multiply charged particles. The difference of the two detection efficiencies should be within $\pm 0,025$. In addition the flow rates of the FCAE and test CPC have to be within 5 % of their original measurements in <u>6.2.7</u>. If not, all tests conducted are not valid.

NOTE The repetition of the first point can be done e.g. after three tests, but also after more tests. However when more than five tests are conducted the risk of losing all measurements is higher (due to a higher than 0,025 difference between the first and last points).

6.3.9 Preparation of the calibration certificate

Fill in the calibration certificate (see <u>Clause 8</u> and <u>Annex C</u>). This completes the measurements for the determination of the detection efficiency of the test CPC.

6.4 Measurement uncertainty

6.4.1 General

The result of the CPC calibration takes the form of a detection efficiency, η , at a certain particle size, a certain particle number concentration, for a certain type of generated particles. The principal quantities requiring well-defined measurement uncertainties are the particle size and the detection efficiency, covered in <u>6.4.2</u> and <u>6.4.3</u>. The particle number concentration is less critical, and its uncertainty is largely covered by that of the detection efficiency. It is covered in <u>6.4.4</u>.

6.4.2 Particle size

The particle size associated with the calibration is determined by the DEMC supplying the calibration aerosol to both instruments. The particle size and its measurement uncertainty shall be determined according to ISO 15900.

When there is a significant fraction of multiply charged particles in the calibration aerosol, significant numbers of particles will have much larger sizes than the selected size. The fractions ϕ_p , which have been determined according to Annex D, shall be noted on the calibration certificate. When the fraction has not been determined a note shall be made on the certificate describing the scale of any relevant effects expected from the system used.

6.4.3 Detection efficiency

The uncertainty in the result for detection efficiency is principally determined by components from:

- the FCAE (as described on its certificate);
- the multiple-charge correction;
- differences in particle concentrations sampled by the FCAE and the test CPC (the splitter bias correction factor);
- the accuracy and variations of the FCAE input flow measurements;
- the repeatability of the detection efficiency calibration;
- the effect of uncertainty in the particle size determination (when the particle size is such that the CPC detection efficiency is significantly affected by the particle size).

As stated in <u>6.1</u>, the FCAE shall have an unexpired calibration certificate specifying the charge concentration or current and flow rate range for which the calibration is valid. The certificate will give an uncertainty for the charge concentration, or alternatively for the current and the flow rate that can be combined to form an uncertainty for the particle charge concentration. Note that this will often be expressed as expanded uncertainties (k = 2; approximately 95 % confidence) values, while uncertainties must be combined as standard uncertainties (k = 1) values.

Correction for multiply charged particles is made according to formulae in Annex D. As a first approximation, the uncertainty in the correction factor is best determined semi-empirically by repeated determination of the multiply charged fractions ϕ_p , and expressing the variation as a standard deviation associated with the multiple-charge correction. If the uncertainties for the fractions ϕ_1 , ϕ_2 , ϕ_3 are termed u(1), u(2), and u(3) respectively, the required relative uncertainty component $u_r(MCC)$ is given by Formula (9):

$$u_{r}(MCC) = \frac{100}{\sum_{p>1} \phi_{p} \cdot p} \cdot \sqrt{u^{2}(1) + 4u^{2}(2) + 9u^{2}(3)}$$
(9)

This uncertainty component applies in the detection efficiency plateau region (see <u>D.1</u>). The more complicated case away from the plateau region is not covered by this International Standard.

The procedure for calculating the bias correction factor β and its uncertainty are given in Annex G.

If the input flow to the FCAE is different at the time of the test CPC calibration to when the FCAE was calibrated, there will be a proportionate effect on the detection efficiency determination. The flows shall be measured with calibrated flow meters on each occasion, as described in 6.2.7 b), and shall agree within the specified tolerance. The uncertainty associated with the FCAE flow is set by this tolerance. A correction could be made to allow for different flows, with a consequent reduction in uncertainty, but this is not covered by this International Standard.

The five repeated measurements specified in $\underline{6.3.5}$ e) give an estimate of short-term repeatability of the detection efficiency measurement. The standard deviation of the repeated measurements is included in

the uncertainty calculation. The uncertainty calculation therefore applies only to individual batches of these five repeated measurements.

Guidance on cases where the uncertainty associated with the particle size is expected to have a significant effect on the uncertainty of the detection efficiency is given in Annex M.

The calculation of uncertainty in the detection efficiency is summarized in <u>Table 3</u>.

Table 3 — Relative uncertainty components for calibration with an FCAE

Component	Symbol	Reference	Comment
FCAE detection efficiency	$u_{\rm r}({\rm FCAE})$	Taken from FCAE certificate	Expressed as % of FCAE reading
Multiple charge correction	$u_{\rm r}({ m MCC})$	Formula (9)	Expressed as %
Splitter bias correction factor	$u_{\mathrm{r}}(eta)$	Annex G, Formulae (G.10) (Refer to Annex G for the case of unequal flows.)	Expressed as %, i.e. $u_r(\beta) = 100 \ u(\beta)/\beta$
FCAE flow rate deviation	$u_{\rm r}(q_{\rm FCAE})$	6.2.7 b)	Expressed as %
Repeatability	$u_{\mathrm{r}}(\eta_{\mathrm{rep}})$	6.3.5 f)	Expressed as %, i.e. $u_{\rm r}(\eta_{\rm rep}) = 100~\sigma~(\eta_{\rm rep})/\overline{\eta}_{\rm CPC}$

All components are to be in the form of relative standard uncertainties, corresponding to standard deviations.

The combined relative standard uncertainty is given by Formula (10):

$$u_{c,r}(\eta) = \sqrt{u_r^2(FCAE) + u_r^2(MCC) + u_r^2(\beta) + u_r^2(q_{FCAE}) + u_r^2(\eta_{rep})}$$
(10)

The relative expanded uncertainty $U_r(\eta)$ is obtained by multiplying the combined relative standard uncertainty by a coverage factor k: $U_r(\eta) = k u_{c,r}(\eta)$. Typically a value of k = 2 is used.

A worked example is given in **Annex I**.

6.4.4 Particle number concentration

The particle number concentration to be reported on the CPC calibration certificate provides information relevant when nonlinearity is expected in the response of the CPC, for example when changing between instrument measurement modes. The concentration to be reported on the certificate is the arithmetic mean concentration recorded by the FCAE, after corrections have been applied for multiple charge correction and FCAE flow. It is not necessary to estimate an uncertainty for this figure. The uncertainty would be expected to be slightly less than the uncertainty of the detection efficiency, as all of the components except the splitter bias correction factor would apply.

The uncertainty that can be assigned to measurements made with the test CPC after calibration is a more complicated topic, which is addressed in $\underline{\text{Annex N}}$.

7 Calibration using a CPC as reference instrument

7.1 Overview of the setup and calibration procedure

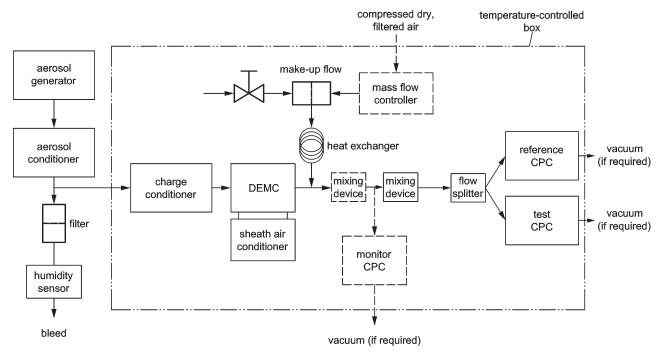
A schematic for a typical calibration setup with a reference CPC is given in Figure 6. All parts drawn with solid lines are necessary components (see discussion in Clause 5). These include the aerosol generator, the aerosol conditioner, the humidity sensor to measure the humidity of the aerosol that enters the DEMC, the charge conditioner, the DEMC, the make-up flow, the mixing device, the flow splitter, the reference and test CPCs. Although not shown in the figure, a relative humidity sensor shall be used to measure

the relative humidity of the make-up air at the beginning and end of the experiments. A pressure sensor might also be necessary for the determination of the volumetric flow rate of the instruments.

In case that the calibration aerosol flow from the DEMC is higher than the sum of the flow rates required by the instruments, the excess air shall be vented off as bleed flow. While it is not shown in the figure, a temperature sensor shall be used to monitor the temperature in the temperature-controlled box or room temperature.

The parts of Figure 6 with dashed lines are recommended but not required. For example, the temperature-controlled box and heat exchangers for the DEMC sheath air flow and the make-up air flow can be used to stabilize all temperatures. A monitor CPC can be used to check the stability of the calibration aerosol. The make-up air flow can be controlled with a throttle valve or compressed air with a mass flow controller.

NOTE Apart from the reference CPC, the components and the respective requirements are the same as those for the FCAE comparison ($\underline{\text{Clause 6}}$).



NOTE Dashed lines show equipment that is not required, but highly recommended.

Figure 6 — Typical calibration setup with CPC as reference instrument

The calibration procedure can be seen in Table 4.

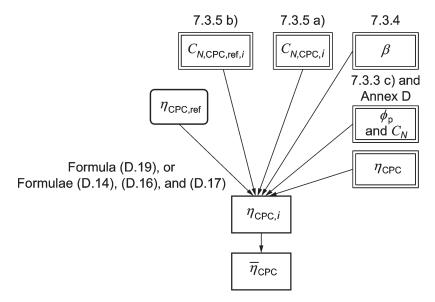
Table 4 — CPC calibration procedure using a CPC as reference instrument

<u>7.2</u>	Preparation
<u>7.2.2</u>	Aerosol generator and conditioner (size distribution)
<u>7.2.3</u>	Other equipment (e.g. mass flow meters, etc.)
7.2.4	DEMC (according to ISO 15900) and sheath air conditioner
<u>7.2.5</u>	Reference CPC
	Zero arithmetic mean <0,1 cm ⁻³ (from 1-s average concentrations of at least 5 min)
	High response check
	Flow measurement and stability <2 % (from 5 measurements in 15 min)

Table 4 (continued)

7.2.6 Test CPC Zero arithmetic mean <0,1 cm ⁻³ (from 1-s average of High response check) Flow measurement and stability <2 % (from 5 means) Connect the instruments and the aerosol generator of DEMC (voltage off), sheath to sample >7:1 Reference CPC flow measurement	surements in 5 min) //conditioner to the DEMC arithmetic means of 2 min); Ref. CPC zero arithme-
High response check Flow measurement and stability <2 % (from 5 measurement and stability <2 % (from 5 measurement) Connect the instruments and the aerosol generator DEMC (voltage off), sheath to sample >7:1	surements in 5 min) /conditioner to the DEMC arithmetic means of 2 min); Ref. CPC zero arithme-
Flow measurement and stability <2 % (from 5 measurement and stability <2 % (from 5 measurement and the aerosol generator DEMC (voltage off), sheath to sample >7:1	/conditioner to the DEMC
7.2.7 Connect the instruments and the aerosol generator DEMC (voltage off), sheath to sample >7:1	/conditioner to the DEMC
DEMC (voltage off), sheath to sample >7:1	arithmetic means of 2 min); Ref. CPC zero arithme-
Deference CDC flour measurement	
Reference CPC flow measurement	
Ref. CPC zero arithmetic mean <1 cm ⁻³ (from 30-s at tic mean < 0,1 cm ⁻³ if calibration is extended to low	or componer action (million 11)
Test CPC zero arithmetic mean <1 cm ⁻³ (from 30-s tic mean <0,1 cm ⁻³ if calibration is extended to low	arithmetic means of 2 min); test CPC zero arithmeter concentration ($\frac{Annex H}{A}$)
Determine minimum level of Ref. CPC with Formul	a (14)
7.3 Detection efficiencies	
7.3.2 DEMC diameter adjustment	
7.3.3 Primary aerosol adjustment	
The concentration to be within the capability of th	e charge conditioner
Multiply charged particles fraction <10 %	
Concentration within the range of the Ref. CPC	
7.3.4 Splitter bias β measurement	
7.3.5 Test CPC efficiency measurement	
At the specific diameter and concentration:	
Set DEMC voltage off (or 0)	
 Record 60 s CPC concentrations, use the last 30 	s for the calculations.
 Check each CPC's arithmetic mean <1 cm⁻³. 	
Set DEMC voltage for the specific diameter	
 Record 180 s CPC concentrations, use the last 5 	× 30 s for the calculations.
— Check each CPC's CV for each of the five 30 s in	ervals <3 %, or standard deviation <0,5 cm ⁻³ .
Calculate detection efficiency $\eta_{\mathrm{CPC},i}$ for each of the	five 30 s intervals.
Calculate the arithmetic mean detection efficiency	$\overline{\eta}_{\mathrm{CPC}}$. All $\eta_{\mathrm{CPC},i}$ shall be within $\overline{\eta}_{\mathrm{CPC}}\pm 0.02$.
7.3.6 Measurement of a different concentration (optional	lly)
Go to <u>7.3.3</u> and then <u>7.3.5</u>	
7.3.7 Measurement of a different size (optionally)	
Go to <u>7.3.2</u>	
7.3.8 Repetition of first measurement	
If >5 points have been tested (difference of $\overline{\eta}_{ ext{CPC}}$ h	as to be within 0,025)
7.3.9 Fill in the calibration certificate	

Figure 7 provides a graphical summary of the derivation of the detection efficiency.



NOTE Plain rectangles represent calculated values while double-lined and round-cornered rectangles are for measured values and values taken from certificates, respectively.

Figure 7 — A map of parameters and formulae needed to derive the detection efficiency in calibration with a reference CPC

7.2 Preparation

7.2.1 General preparation

Check that all instruments operate properly according to the manufacturers' specifications (see <u>7.2.2</u> to <u>7.2.6</u>), then prepare (according to <u>Figure 6</u>) and check the complete setup (<u>7.2.7</u>). Do not proceed to the calibration procedure of detection efficiency (<u>7.3</u>) unless all tests have been passed.

7.2.2 Primary aerosol

Start the operation of the primary aerosol source according to the manufacturer's recommendations. It is highly recommended to measure the generated size distribution after the aerosol conditioner with a DMAS (e.g. combine DEMC with reference CPC) if it is not known. Ensure that the relative vapour contents (from water and/or solvents) in the primary aerosol shall be less than 40 %.

7.2.3 Other equipment

Switch on and allow all auxiliary necessary equipment to stabilize. Turn on the charge conditioner if it is off. Prepare the calibrated pressure sensor(s), the calibrated temperature sensor(s) and the calibrated flow meter for the measurement of the reference CPC and test CPC flow rates and the humidity sensors.

Any other recommended instruments in the setup should be also turned on and prepared according to the manufacturers' manuals (e.g. a monitor CPC, mass flow meters, mass flow controllers, pressure and temperature sensors, etc.). If the whole setup (i.e. DEMC and instruments) is in a temperature-controlled box, set the desired temperature and leave the system time to stabilize.

7.2.4 **DEMC**

Turn on the instrument, check the DEMC according to ISO 15900, and set the desired flows. Prepare the conditioner of the sheath flow.

7.2.5 Reference CPC

Turn on the reference CPC and allow the saturator, condenser and optics to reach their specified temperatures. Leave it running for at least 30 min. If working fluid has been changed, leave it running for at least 1 h.

Check the zero level and flow rates of the reference CPC at ambient conditions. All indicators (e.g. for temperatures, flows and pressures) shall show error-free operation of the instrument. The following checks ensure that the instrument is working properly before connecting it to the calibration setup.

a) Zero count check

For zero count check, attach at least one HEPA filter (>99,99 % efficiency) to the reference CPC inlet (an additional HEPA filter in series with the first one may be necessary to achieve the required extremely low concentrations). Run the CPC for a minimum of 5 min and record the concentration values with 1-s reading interval and 1-s averaging time. After any leaks are eliminated, the arithmetic mean concentration shall be $<0.1 \text{ cm}^{-3}$.

Contact the manufacturer if these requirements are not met.

b) High response check

Perform a simple check to demonstrate that the reference CPC can detect particles. This can, for example, be done by sampling room air if the number concentration of the room air is expected to be higher than 500 cm⁻³. The number concentration measured by the reference CPC should be higher than 500 cm⁻³. Aerosols from other sources with sufficiently high number concentrations may also be used for this test. Or, follow the manufacturer's recommendations. Contact the manufacturer if this requirement is not met.

c) Flow rate measurement

Measure the nominal (see calibration certificate) volumetric inlet flow rate of the reference CPC at ambient conditions with an appropriate low pressure drop, calibrated flow meter (Annex J). The flow shall be stable over time, i.e. the CV of at least 5 measurements uniformly spaced over 15 min shall be <2 %. There should be no obvious decreasing or increasing tendency of the flow. If not fulfilled, leave the reference CPC more time to stabilize, check the pump (or vacuum connection) of the reference CPC, and repeat. The reference CPC requires attention from the manufacturer if the flow check fails twice.

Compare the (calculated) arithmetic mean of the measured inlet flow rates of the reference CPC ($q_{\text{CPC},\text{ref},\text{cal},\text{amb}}$) with the arithmetic mean value indicated by the reference CPC for the same time intervals or the nominal value of the reference CPC ($q_{\text{CPC},\text{ref},\text{amb}}$). The latter case applies if no flow rate is reported or when the nominal value is used by the reference CPC for the particle concentration calculation. The difference should be within the reference CPC manufacturer's specifications, indicated as an accuracy $r_{\text{q,CPC},\text{ref}}$ in %. If not, the manufacturer should be contacted.

The flow rate $q_{\text{CPC},\text{ref},\text{cal},\text{amb}}$ should also be compared with the flow rate of the reference CPC in its calibration certificate ($q_{\text{CPC},\text{ref},\text{cert}}$) and the deviation shall be within:

$$\frac{\left|q_{\text{CPC,ref,cal,amb}} - q_{\text{CPC,ref,cert}}\right|}{q_{\text{CPC,ref,cert}}} < 2\sqrt{u_{\text{r}}^2(q_{\text{CPC,ref,cert}}) + u_{\text{r}}^2(q_{\text{cal,cert}}) + \frac{1}{3}r_{\text{q,CPC,ref}}^2}$$
(11)

where

 $u_r(q_{CPC,ref,cert})$ is the relative standard uncertainty for the inlet flow of the reference CPC in its calibration certificate;

 $u_{\rm r}(q_{\rm cal,cert})$ is the relative standard uncertainty of the flow meter used to measure $q_{\rm CPC,ref,-}$ cal,amb·

Higher deviations might indicate issues with the flow control of the reference CPC.

All flow rates shall refer to the same temperature and pressure. Depending on the flow control used in the reference CPC, different corrections should apply (see <u>Annex I</u>).

7.2.6 Test CPC

When a CPC has been transported for calibration, it will generally have been drained of working fluid. In that case, switch the test CPC on, fill with the required working fluid to the specified level (observing manufacturer's precautions regarding moving the unit when full) and allow the saturator, condenser and optics to reach their specified temperatures. Leave it running for at least 1 h.

When a CPC is to be calibrated without being drained of the working fluid, manufacturer's precautions regarding moving the unit when full shall be observed. Switch on the test CPC, leave it running for at least 30 min, and allow the saturator, condenser and optics to reach their specified temperatures.

 $All indicators \ (e.g. for temperatures, flows and pressures) shall show error-free operation of the instrument.$

a) Zero count check

For zero count check, attach at least one HEPA filter (>99,99 % efficiency) to the test CPC inlet (an additional HEPA filter in series with the first one may be necessary to achieve extremely low concentrations). Run the CPC for a minimum of 5 min and record the concentration values with 1-s reading interval and 1-s averaging time. After any leaks are eliminated, the arithmetic mean concentration shall be <0,1 cm⁻³.

Contact the customer if these requirements are not met.

b) High response check

Perform a simple check to demonstrate that the test CPC can detect particles. This can, for example, be done by sampling room air if the number concentration of the room air is expected to be higher than 500 cm⁻³. The number concentration measured by the test CPC should be higher than 500 cm⁻³. Aerosols from other sources with sufficiently high number concentrations may also be used for this test. Or, follow the manufacturer's recommendations. Contact the customer if this requirement is not met.

c) Flow rate measurement

Measure the volumetric inlet flow rate of the test CPC at ambient conditions with an appropriate low pressure drop, calibrated flow meter (Annex I). The flow shall be stable over time, i.e. the CV of at least 5 measurements uniformly spaced over 5 min shall be <2 %. There should be no obvious decreasing or increasing tendency of the flow. If not fulfilled, leave the test CPC more time to stabilize, check the pump (or vacuum connection) of the test CPC and repeat. The test CPC requires maintenance if the flow check fails twice.

Compare the (calculated) arithmetic mean of the measured inlet flow rates of the test CPC ($q_{CPC,cal,amb}$) with the arithmetic mean value indicated by the test CPC for the same time intervals or the nominal value of the test CPC ($q_{CPC,amb}$). The latter case applies if no flow rate is reported or when the nominal value is used by the CPC for the particle number concentration calculation. The difference should be within the test CPC manufacturer's specifications. If not, the customer should be contacted. Higher differences might indicate issues with the flow control of the test CPC.

All flow rates shall refer to the same temperature and pressure. Depending on the flow control used in the test CPC, different corrections should apply (see Annex J).

7.2.7 Check of the complete setup

Initially, the make-up flow path is connected downstream of the DEMC (typically a HEPA filter, or a mass flow controller with a HEPA filter). Then connect the mixing device and pressure sensor. The test and reference CPCs are connected to the flow splitter which is positioned after the mixing device. Make sure that there is at least one opening free (e.g. the inlet of the DEMC or the make-up flow path) in order to avoid overpressure or underpressure at the inlet of the test and reference CPCs. If there is a monitor CPC, this should be connected before the mixing device with another mixing device.

Connect the aerosol generator and conditioner to the inlet of the DEMC, making sure that the excess flow is vented, or that filtered air is added if the DEMC flow rate is higher.

Check if the pressure at the inlet of the test and reference CPCs has remained in the desired range (i.e. no extreme underpressure or overpressure, outside the manufacturers' specifications). If not, adjust accordingly the make-up air flow or the throttling valve.

a) DEMC flow rate

Set the desired DEMC sheath flow rate. Set the desired DEMC inlet flow rate by adjusting the make-up flow (or bleed air). The ratio of sheath to sample flow rates shall be \geq 7:1 to ensure narrow monodisperse distribution after the DEMC.

After setting these flow rates, it is recommended to not adjust them during the calibration procedure. If adjusted, the volumetric flow rates of the reference and test CPCs have to be measured again [see steps b) and c) below].

b) Reference CPC flow rate measurement

Measure the volumetric flow rate of the reference CPC by inserting the calibrated flow meter between the splitter and the inlet of the reference CPC. Compare this measured value ($q_{\text{CPC,ref,cal}}$) with the value reported by the reference CPC or its nominal value ($q_{\text{CPC,ref}}$). The latter case applies if no flow rate is reported or when the nominal value is used by the reference CPC for the particle number concentration calculation. The difference should be within the reference CPC manufacturer's specifications, indicated as an accuracy $r_{\text{q,CPC,ref}}$ in %. If not the manufacturer should be contacted.

The flow rate $q_{\text{CPC},\text{ref},\text{cal}}$ should also be compared with the flow rate of the reference CPC in its calibration certificate ($q_{\text{CPC},\text{ref},\text{cert}}$) and the deviation shall be within:

$$\frac{\left|q_{\text{CPC,ref,cal}} - q_{\text{CPC,ref,cert}}\right|}{q_{\text{CPC,ref,cert}}} < 2\sqrt{u_{\text{r}}^2(q_{\text{CPC,ref,cert}}) + u_{\text{r}}^2(q_{\text{cal,cert}}) + \frac{1}{3}r_{\text{q,CPC,ref}}^2} \tag{12}$$

where

 $u_r(q_{CPC,ref,cert})$ is the relative standard uncertainty for the inlet flow of the reference CPC in its calibration certificate;

 $u_{\rm r}(q_{\rm cal,cert})$ is the relative standard uncertainty of the flow meter used to measure $q_{\rm CPC,ref,-}$

Higher deviations might indicate issues with the flow control of the reference CPC.

The relative standard uncertainty of $q_{CPC,ref}$ is calculated as shown in Formula (13):

$$u_{\rm r}(q_{\rm CPC,ref}) = \left| q_{\rm CPC,ref,cal} - q_{\rm CPC,ref,cert} \right| / \sqrt{3} q_{\rm CPC,ref,cert}$$
(13)

All flow rates shall refer to the same temperature and pressure. Depending on the flow control used in the reference CPC, different corrections should apply (see <u>Annex I</u>).

NOTE 1 The flow rate is affected by the composition of the gas. For more details, see Annex I.

NOTE 2 The flow measurement is repeated here to account for flow rate changes due to changed reference CPC inlet pressure.

c) Test CPC flow rate measurement

Measure the volumetric flow rate of the test CPC by inserting the calibrated flow meter between the splitter and the inlet of the test CPC. Compare this measured value with the value reported by the test CPC or its nominal value. The latter case applies if no flow rate is reported or when the nominal value is used by the test CPC for the particle number concentration calculation. The difference should be within the test CPC manufacturer's specifications. If not the customer should be contacted. Higher differences

might indicate issues with the orifice or the pump of the test CPC. This value shall be reported in the calibration certificate, along with the test CPC reported or nominal value.

All flow rates shall refer to the same temperature and pressure. Depending on the flow control used in the test CPC, different corrections should apply (see <u>Annex J</u>).

NOTE 1 The flow rate is affected by the composition of the gas. For more details, see Annex I.

NOTE 2 The flow measurement is repeated here to account for flow rate changes due to changed test CPC inlet pressure.

d) Zero levels

Set the DEMC voltage to 0 V (or off). The reference and test CPCs' concentration readings shall remain comparable to the zero levels measured before [7.2.5 a) and 7.2.6 a)]: measure for a minimum of 2 min, the 30-s arithmetic mean values (with 1-s reading intervals) shall be lower than 1 cm⁻³. If not, check for leaks in the calibration setup. Other reasons for increased zero levels can be — for example — an excessively high concentration at the inlet of the DEMC or overloading or failure of the filters inside the DEMC.

If the calibration of a test CPC is extended to low concentrations according to Annex H, the measured concentrations resulting from the zero count checks must be less than 0,1 cm⁻³ for both the reference CPC (which may serve as the dummy CPC according to Annex H) and the test CPC. An additional HEPA filter in series with the first one may be necessary to achieve these low concentrations.

e) Determine the reference CPC minimum level

Determine the arithmetic mean and standard deviation of the particle number concentrations reported by the reference CPC in 30 s (at 1-s reading interval). Multiply the standard deviation by 3 and add the arithmetic mean value:

$$(C_{N,\text{ref}})_{\min} = (C_{N,\text{ref}})_{\text{mean}} + 3\sigma_{C_{N,\text{ref}}}$$
(14)

Compare this value with the lowest concentration in its calibration certificate. The greater one is the minimum concentration at the inlet of the reference CPC that may be used in the calibration (defined here as the "minimum level").

NOTE A large standard deviation $[\sigma_{CN,ref} > (C_{N,ref})_{mean}]$ is typical for particle bursts, caused e.g. by particle re-entrainment from tubing walls. Cleaning the connection tubing and the DEMC is recommended in this case.

Write down all parameters: Readings of the reference and test CPCs, flow rates, pressures, temperatures, make-up flow (if available), sheath, sample flow rates, humidity, etc. All this information will be reported in the certificate of calibration (see <u>Clause 8</u> and <u>Annex C</u>).

7.3 Calibration procedure of detection efficiency

7.3.1 General

The following procedure describes the measurement of the detection efficiency of a test CPC at one given calibration particle size and particle number concentration.

7.3.2 DEMC diameter adjustment

Adjust the DEMC such that the particle size of singly charged calibration particles leaving the DEMC equals the desired calibration particle size.

NOTE It is highly recommended to begin with a big size (such that the detection efficiency of the test CPC is at the maximum, i.e. at least 3 times the size where the detection efficiency is expected to be 50 %) and check the linearity of the test CPC (i.e. different concentrations at the same size). Then the detection efficiency at the steep part of the detection efficiency curve may follow. This is because the tests at the steep part of the detection efficiency curve usually need adjustment of the size distribution produced by the generator and are more time consuming.

7.3.3 Primary aerosol adjustment

Adjust the aerosol conditioner in such a way that the concentration of the calibration aerosol equals the desired concentration for the measurement of the detection efficiency. However the following requirements shall be fulfilled.

a) Minimum level

The concentration shall be higher than the minimum level of the reference CPC, as defined in Formula (14) in 7.2.7 e).

b) Maximum level

The concentration shall be lower than the maximum concentration the reference CPC may measure in the single particle counting mode, as specified in its calibration certificate.

The total number concentration of the conditioned primary aerosol shall be low enough to reach charge equilibrium after passing through the charge conditioner and to avoid particle charge caused bias in the DEMC. A method to check this is described in Annex K.

c) Fraction of multiply charged particles Φ

When the test CPC has reached its detection efficiency plateau and a CPC is used as the reference instrument, there is no requirement to measure the fraction of multiply charged particles.

When the test CPC is being calibrated at particle diameters near the lower detection limit and a CPC is used as the reference instrument, the value of multiply charged particles Φ shall be < 0,1. Determine the fractions of particles of p charges $[\phi_p$, Formula (15)] according to one of the methods described in Annex D, and calculate the fraction of multiply charged particles (Φ) with Formula (16). The criterion Φ < 0,1 is a pass/fail criterion; the calibration procedure shall not be continued until it is fulfilled. The Φ can be decreased by, e.g. adjusting the mode diameter or the geometric standard deviation of the size distribution of the primary aerosol.

The fraction of particles with p charges, ϕ_p , within the aerosol leaving the DEMC is calculated as shown in Formula (15):

$$\phi_p = C_N(d_p) / \sum_{p \ge 1} C_N(d_p) \tag{15}$$

where

 $C_N(d_p)$ is the concentration of particles with p charges.

The fraction of multiply charged particles, Φ , is calculated as

$$\Phi = \sum_{p>2} \phi_p \tag{16}$$

NOTE 1 Depending on the polarity of the voltage in the DEMC, the charges of the particles can be either positive or negative. In this International Standard, we define p as the absolute number of charges.

NOTE 2 It is highly recommended that the geometric mean size of the primary distribution is smaller than the size at which the detection efficiency of the test CPC will be measured.

NOTE 3 If a tandem DEMC setup is employed (i.e. two DEMCs in series with a charge conditioner in between), the fraction of multiply charged particles, ϕ , is reduced significantly.

7.3.4 Splitter bias β measurement

Perform the splitter bias measurement according to $\underline{\text{Annex G}}$. If the observed splitter bias β is greater than 1,05 or less than 0,95, check for any inhomogeneity of the calibration aerosol.

7.3.5 Test CPC efficiency measurement

Determine the detection efficiency of the test CPC according to the following steps.

a) Recordings at DEMC voltage 0 (or off)

Set the DEMC voltage to 0 V (or off) and record the number concentrations reported by the reference and test CPCs with 1-s reading interval and 1-s averaging time for 1 min. Calculate the arithmetic means of the number concentrations reported by the reference and test CPCs for the last 30 s of the 1-min measurement interval.

The arithmetic means of the reference and test CPCs' number concentrations shall be <1 cm⁻³. If not, the measurement is not valid. Check the generator or other sources of instability and repeat.

b) Recordings at specific size and concentration

Record the number concentrations reported by the reference and test CPCs with 1-s reading interval and 1-s averaging time for at least 180 s. Separate the recorded data into six equal time intervals i = 1 to 6 (e.g. 6 × 30 s if total time is 180 s). Calculate the arithmetic means $C_{N,\text{CPC},\text{ref},i}$ and $C_{N,\text{CPC},i}$ and the standard deviations of the number concentrations reported by the reference and test CPCs, respectively, for each of the time intervals i = 2 to 6.

In each time interval, either the CV or the standard deviation of the reference and test CPCs' concentrations shall be <3% or <0.5 cm⁻³, respectively. If not, the measurement is not valid. Check the generator or other sources of instability and repeat.

For low number concentrations (nominally less than 10^3 cm⁻³), it is recommended that longer time intervals be used to reduce measurement uncertainty. For example, record the indicated number concentrations reported by the reference and test CPCs every second with 1-s averaging time for 12 min. Separate the recorded data into six time intervals i of 2 min and calculate the arithmetic means and standard deviations of the number concentrations for the last five of the six 2-min intervals.

c) Calculation of the detection efficiency of the test CPC

If the primary aerosol cannot be conditioned in such a way that the calibration aerosol contains one elementary charge, corrections shall be applied (Annex D).

Annex D also quantifies the effect of multiple charges on the determination of particle size.

Information from the reference CPC certificate regarding any correction factor required by the reference CPC with respect to its displayed concentration shall be incorporated (Annex D).

Calculate the detection efficiency $\eta_{CPC,i}$ of the test CPC using the arithmetic mean concentrations obtained in 7.3.5 b) and formulae in Annex D:

- If the particle size is in the range where the detection efficiency of the test CPC is known to be constant against size, substitute $C_{N,\text{CPC},\text{ref},i}$ [7.3.5 a)], $C_{N,\text{CPC},i}$ [7.3.5 a)], β (7.3.4), and $\eta_{\text{CPC},\text{ref}}$ (from the calibration certificate of the reference CPC) into Formula (D.19).
- If the particle size is in the range where the detection efficiency of the test CPC is known to vary with size, substitute $C_{N,CPC,ref,i}$ [7.3.5 a)], $C_{N,CPC,i}$ [7.3.5 a)], ϕ_p (and C_N obtained in Annex D) [7.3.3 c)], β (7.3.4), $\eta_{CPC,ref}$ (from the calibration certificate of the reference CPC) and an estimated plateau efficiency η'_{CPC} of the test CPC into Formulae (D.14), (D.16), and (D.17).

Record in the calibration certificate which method of calculation was used.

Calculate the arithmetic mean detection efficiency $\bar{\eta}_{\text{CPC}}$ and its standard deviation $\sigma(\eta_{\text{rep}})$ for the specific concentration and size using the five detection efficiencies, $\eta_{\text{CPC},i}$ (i=2-6). The calibration is valid only if all five $\eta_{\text{CPC},i}$ values are within $\pm 0,02$ of the arithmetic mean detection efficiency $\bar{\eta}_{\text{CPC}}$.

7.3.6 Measurement of different particle concentrations

If a different concentration (at the same size) has to be measured, adjust the concentration of the primary aerosol (go to 7.3.3 and then 7.3.5). The measurement of the fraction of multiply charged particles [7.3.3 c)] is not necessary to repeat. Note that the splitter bias (7.3.4) and the reference CPC flow rate [7.2.7 b)] do not have to be tested. For concentrations lower than the maximum level, there is no need to re-check the charge conditioner [7.3.3 b)]. If the CPC is to be calibrated at concentrations below the lowest concentration for which the reference CPC has been certified, the method described in Annex H can be followed.

If the DEMC flow rates have to be adjusted, measure the inlet flow rates of the reference and test CPCs (see 7.2.7).

7.3.7 Measurement of different sizes

If a different particle size has to be tested, then go to 7.3.2. In this case all following steps (7.3.3) to 7.3.6 have to be performed.

If the DEMC flow rates have to be adjusted, measure the inlet flow rates of the reference and test CPCs (see 7.2.7).

NOTE 1 If the detection efficiency of a size at the steep part of the detection efficiency curve is tested (i.e. no linearity check), it is recommended to set at a concentration that the coincidence correction of the test CPC is <1%.

NOTE 2 It is also highly recommended to conduct all detection efficiency tests on the same test CPC at various particle sizes at approximately the same particle number concentration. This would avoid any influence from a nonlinear CPC response.

NOTE 3 When planning the concentrations to be used, attention shall be paid to the concentrations at which the measurement mode of the test CPC changes (as stated in the manufacturer's manual), so that discontinuities can be anticipated.

7.3.8 Repetition of first measurement point

When several points are tested, or when a measurement instrument was switched off, or when a malfunction occurred, the detection efficiency measurement with the first set of aerosol parameters has to be repeated (i.e. concentration and size). This includes the determination of the fraction of multiply charged particles. The difference of the two detection efficiencies should be within \pm 0,025. In addition the flow rates of the reference and test CPCs have to be within 5 % of their original measurements in 7.2.7. If not, all tests conducted are not valid.

NOTE The repetition of the first point can be done e.g. after 3 tests, but also after more tests. However when more than 5 tests are conducted the risk of losing all measurements is higher (due to a higher than 0,025 difference between the first and last points).

7.3.9 Preparation of the calibration certificate

Fill in the calibration certificate (see <u>Clause 8</u> and <u>Annex C</u>). This completes the measurements for the determination of the detection efficiency of the test CPC.

7.4 Measurement uncertainty

7.4.1 General

The result of the CPC calibration takes the form of a detection efficiency η at a certain particle size, a certain particle number concentration, for a certain type of generated particles. The principal quantities requiring well-defined measurement uncertainties are the particle size and the detection efficiency, covered in 7.4.2 and 7.4.3. The particle number concentration is less critical, and its uncertainty is largely covered by that of the detection efficiency. It is covered in 7.4.4.

7.4.2 Particle size

The particle size associated with the calibration is determined by the DEMC supplying the calibration aerosol to both instruments. The particle size and its measurement uncertainty shall be determined according to ISO 15900.

When there is a significant fraction of multiply charged particles in the calibration aerosol, significant numbers of particles will have much larger sizes than the selected size. The fractions ϕ_p , which have been determined according to Annex D, shall be noted on the calibration certificate. When the fraction has not been determined a note shall be made on the certificate describing the scale of any relevant effects expected from the system used.

7.4.3 Detection efficiency

The uncertainty in the result for detection efficiency is principally determined by components from the following:

- the reference CPC (as described on its certificate);
- the multiple-charge correction;
- differences in particle concentrations sampled by the reference and test CPCs (the splitter bias correction factor);
- the accuracy and variations of the reference CPC input flow measurements;
- the repeatability of the detection efficiency calibration;
- the effect of uncertainty in the particle size determination (when the particle size is such that the CPC detection efficiency is significantly affected by the particle size).

As stated in 7.1, the reference CPC shall have an unexpired calibration certificate specifying the particle number concentration range for which the calibration is valid. The certificate will give an uncertainty for its particle number concentration measurements. Note that this will often be expressed as expanded uncertainties (k = 2; approximately 95 % confidence) values, while uncertainties must be combined as standard uncertainties (k = 1) values.

There is no multiple charge correction in the detection efficiency plateau region (see <u>D.1</u>), so the uncertainty component in this case is zero. The more complicated case away from the plateau region is not covered in this International Standard.

The procedure for calculating the splitter bias correction factor β and its uncertainty are given in Annex G.

If the input flow to the reference CPC is different at the time of the test CPC calibration to when the reference CPC was calibrated, there will be a proportionate effect on the detection efficiency determination. The flows shall be measured with calibrated flow meters on each occasion, as described in 7.2.7 b), and shall agree within the specified tolerance. The uncertainty associated with the reference CPC flow is set by this tolerance. A correction could be made to allow for different flows, with a consequent reduction in uncertainty, but this is not covered by this International Standard.

The five repeated measurements specified in 7.3.5 d) give an estimate of short-term repeatability of the detection efficiency measurement. The standard deviation of the repeated measurements is included in the uncertainty calculation.

Guidance on cases where the uncertainty associated with the particle size is expected to have a significant effect on the uncertainty of the detection efficiency is given in Annex M.

The calculation of uncertainty in the detection efficiency is summarized in Table 5.

Table 5 — Relative uncertainty components for calibration with a reference CPC

Component	Symbol	Reference	Comment
Reference CPC	$u_{\rm r}({ m RCPC})$	Taken from reference CPC certificate	Expressed as %
		Annex G, Formula (G.8)	Expressed as %, i.e.
Splitter bias correction factor	$u_{\rm r}(\beta)$	(Refer to Annex G for unequal flows.)	$u_{\rm r}(\beta) = 100 \ u(\beta)/\beta$
Reference CPC flow rate deviation	$u_{\rm r}(q_{\rm CPC,ref})$	7.2.7 b)	Expressed as %
			Expressed as %, i.e.
Repeatability	$u_{\rm r}(\eta_{\rm rep})$	7.3.5 d)	$u_{\rm r}(\eta_{\rm rep}) = 100 \sigma(\eta_{\rm rep}) / \overline{\eta}_{\rm CPC}$

All components are to be in the form of relative standard uncertainties, corresponding to standard deviations.

The combined relative standard uncertainty is given by Formula (17):

$$u_{c,r}(\eta) = \sqrt{u_r^2(RCPC) + u_r^2(\beta) + u_r^2(q_{CPC,ref}) + u_r^2(\eta_{rep})}$$
(17)

The relative expanded uncertainty $U_r(\eta)$ is obtained by multiplying the combined relative standard uncertainty by a coverage factor k: $U_r(\eta) = k u_{c,r}(\eta)$. Typically a value of k = 2 is used.

7.4.4 Particle number concentration

The particle number concentration to be reported on the CPC calibration certificate provides information relevant when non-linearity is expected in the response of the CPC, for example when changing between instrument measurement modes. The concentration to be reported on the certificate is the arithmetic mean concentration recorded by the reference CPC, after corrections have been applied for the reference CPC flow. It is not necessary to estimate an uncertainty for this figure. The uncertainty would be expected to be slightly less than the uncertainty of the detection efficiency, as all of the components except the splitter bias correction factor would apply.

The uncertainty that can be assigned to measurements made with the test CPC after calibration is a more complicated topic, which is addressed in $\underline{\text{Annex N}}$.

8 Reporting of results

The reporting of the calibration of a test CPC shall include the following information:

- a) description of the test CPC: manufacturer, model, serial number and internal parameter settings;
- b) description of the reference instrument (FCAE or CPC): manufacturer, model, serial number, internal parameter settings, calibration certificate reference and expiry date;
- c) description of the aerosol generation: particle type, gas composition, method, and all relevant parameters;
- d) corrections made to the reference instrument readings, e.g. if the measured flow differs from the certificate flow;
- e) test CPC flow rate during calibration, together with its measurement uncertainty;
- f) description and date of last calibration of the flow meter(s) used;
- g) temperature and pressure of the laboratory during calibration;
- h) pressure of the calibration aerosol;

- i) result of the zero check of the test CPC;
- j) polarity of the calibration aerosol and measured fractions of doubly and triply charged particles in the calibration aerosol;
- k) description of the method used to calculate the detection efficiency (this may refer to sections of this International Standard).
- l) calibration results: detection efficiency (together with its measurement uncertainty) at a given particle size (together with its measurement uncertainty) and particle concentration;
- m) suggested expiry date of the calibration certificate;
- n) other relevant information.

Suggested templates for reporting calibration results on certificates are given in Annex C.

Annex A

(informative)

CPC performance characteristics

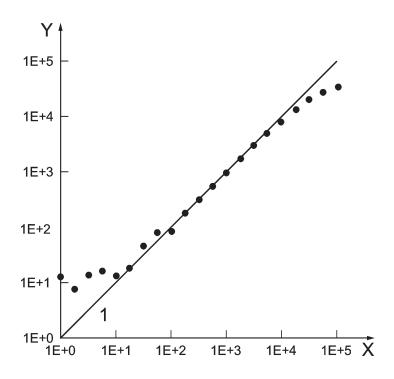
A.1 General

<u>Clauses 6</u> and <u>7</u> from this International Standard described the procedure to determine the detection efficiency of a test CPC at one or more sizes at different particle number concentrations. This Annex provides explanations for major characteristics that describe the performance of a CPC (<u>A.2</u>), and describes how calibration information provided at several particle concentrations and sizes can be used to characterize the CPC with a few parameters (i.e. the slope from the plateau and lower detection limit) (<u>A.3</u>). This information is only informative; the appropriate evaluation of the data are different in each case and only the user can decide what to do with the information that he has collected.

A.2 Major performance characteristics of a CPC

Figure A.1 describes a typical relationship between the actual concentration and the concentration reported by a CPC. In the concentration range in the middle of the graph, the relationship is very linear. This means the detection efficiency is constant. As the concentration decreases, the reported concentration starts to scatter, due to the particle counts becoming infrequent. At the lowest concentration limit, a CPC may report a concentration that is greater than the actual concentration (false count), which occurs due to reasons such as small leaks in the CPC or contamination of the optics. False counts may also occur with much higher concentration if the reasons are large leaks or homogenous nucleation. In the higher concentration range, on the other hand, the reported concentration underestimates the actual concentration due to coincidence count loss, if coincidence correction is not available or is turned off. The linear part can be extended to a higher concentration by implementing coincidence correction appropriately.

NOTE The relationship varies with particle size and particle type.



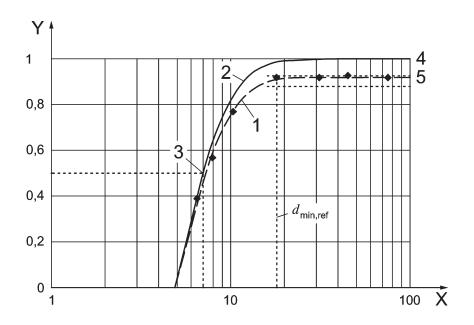
Key

- X actual concentration [cm⁻³]
- Y concentration [cm-3] reported by CPC under calibration
- 1 1:1 line

Figure A.1 — Relationship between the actual concentration and the concentration reported by a CPC in single particle counting mode illustrating false counts and coincidence losses with particles of a given size and type

Figure A.2 shows a typical size dependence of the detection efficiency of a CPC. At large sizes, the detection efficiency stays relatively constant (the plateau efficiency). As particle size decreases, the detection efficiency drops and falls to zero, because the vapour can no longer condense onto particles. The plateau efficiency is normally close to 100 %. The size at which the detection efficiency reaches 50 % of the plateau efficiency is called the lower detection limit or cut-off size. The lower limit of the plateau efficiency, $d_{\min, \text{ref}}$ (3.21), which applies to a reference CPC, is also shown in the figure.

NOTE The size dependence varies with particle concentration and particle type.



Key

- 1 efficiency curve
- 2 normalized curve
- 3 lower detection limit
- 4 normalized plateau
- 5 plateau

- X particle diameter [nm]
- Y detection efficiency [-]

Figure A.2 — Particle size dependence of the detection efficiency of a reference CPC with particles of a given concentration and particle type

A.3 Examples

The following sections will describe how to calculate the major characteristics of a CPC based on experimental detection efficiencies. $\underline{\text{Table A.1}}$ gives the measured detection efficiencies.

Table A.1 — Results of the comparison of a test CPC with a reference CPC

d [nm]	$C_{N,CPC,ref}$ [cm ⁻³]	<i>C_{N,CPC}</i> [cm ⁻³]	$C_{N,CPC}/C_{N,CPC,ref}$
90	11 944,7	11 184,9	0,94
90	10 422,6	9 827,8	0,94
90	8 267,9	7 809,3	0,94
90	4 448,8	4 191,1	0,94
90	609,2	5 71,4	0,94
90	13,3	12,4	0,93
90	5,9	5,5	0,93
90	2 305,0	2 176,2	0,94
90	10 104,6	9 510,0	0,94
75	8 380,2	7 807,8	0,93
55	4 073,9	3 671,3	0,90
55	5 157,7	4 634,6	0,90
41	5 422,9	4 533,6	0,84
23	2 521,8	1 074,6	0,43

CPC,ref

0.84

0,80

0,43

<i>d</i> [nm]	$C_{N,CPC,ref}$ [cm ⁻³]	$C_{N,CPC}$ [cm ⁻³]	$C_{N,CPC}/C_{N,C}$
15	778,1	1,4	0,00
28	2 565 4	2 2177	0.62

5 408.5

5 036,2

2 469,0

Table A.1 (continued)

4 531.1

4 029,7

1 051,4

A.3.1	Lino	arity
A.S.I	Lille	arity

41

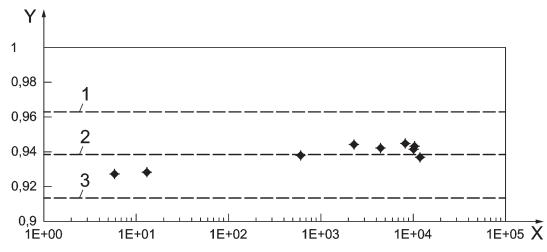
37

23

One important piece of information during the calibration is whether the response of the test CPC is linear or not. This is examined by comparing the test CPC with the reference instrument at different particle number concentrations using a size from the plateau. For example, for a calibration range between 0 and 10⁴ cm⁻³ the concentrations examined could be 2 000, 4 000, 6 000, 8 000, 10 000 cm⁻³ (using an FCAE as a reference instrument) or 10, 100, 1000, 5000, 10000 cm⁻³ (using a reference CPC as a reference instrument).

A.3.1.1 Residual plot

The first step is to plot the ratios of the number concentration of the test CPC to the reference instrument as a function of the reference instrument concentration. [22] The graph gives a correct visual assessment of the data behaviour and their relationship. From the plot it can be easily seen if and what kind of agreement (relationship) exists between the instruments, if there is any tendency for the amount of variation to change with the magnitude of the measurement, and if there are any outliers.[12] The bias of the test CPC is equal to the arithmetic mean of the ratio of the two instruments (test CPC/reference instrument) minus 1. If the differences are normally distributed then 95 % of the differences will lie between the arithmetic mean ± 2 s of the ratios. However a normal probability chart is necessary to assess the normality. What is typically done is that some limits are set for acceptable differences from the arithmetic mean. Figure A.3 shows this graph for the data for the example of Table A.1 with predefined limits of ± 2.5 % to assess the linearity. The limits can be different for each application.



Key

Acceptable variability:

 $C_{N,CPC,ref}$ [cm⁻³] $C_{N,CPC}/C_{N,CPC,ref}[-]$

1 mean + 0.025 = 0.9632 mean = 0.938

mean - 0.025 = 0.913

Figure A.3 — Ratios of the test CPC to the reference instrument as a function of the concentration by the reference instrument (data in Table A.1). The arithmetic mean is 0,938

It should be noted that to use this method, it must first be verified that the reference CPC is linear. Otherwise an apparent linearity of two CPCs may be an artefact of both being in error because of a generic bias in the instrument design. It should also be noted that, if the two instruments have similar uncertainties, it is mistaken to plot the difference against either method separately, as the difference will be related to each, a well-known statistical phenomenon. [11] However, if only the arithmetic mean will be calculated, there is no effect on the result. In addition, this effect is not important in this case where there is such a wide range in concentration and small difference between the two measurements.

Figure A.4 shows four typical cases.[20][24]

- When the two methods show excellent agreement, the ratio will be around 1 (small bias) and the scatter of the ratios will be narrow (good precision) (triangles in Figure A.4).
- When the ratio is different from 1 (and there is bias) but the precision is good, a correction factor usually will solve the problem (circles on Figure A.4). Typically this happens due to wrong initial calibration, drift of the flow controller system of the CPC or the saturator. [24]
- When there is an offset in one of the methods, the graph will look like the squares in Figure A.4. At very low concentrations, a CPC may report a concentration that is higher than the real (false count) due to e.g. small leaks in the CPC and contamination of the optics.
- Finally, when there is a difference at the high concentration range, the graph will look like the x in Figure A.4. This happens because two or more particles pass through the optics of the CPC and are counted as one particle. For this case, either the coincidence correction is turned off, or wrongly calibrated.

Higher scatter of the data shows a rather limited precision (no figure shown). Typically this happens at low concentrations, as the particle counts in the optic become less.

Note that the correlation in the middle is usually linear in all cases, the effect of offset or coincidence is minimal. For this reason it is highly recommended to calibrate a CPC in this range, whenever it is possible. Finally it should be noted that the above correlations vary with particle size and particle type (see Annex B).

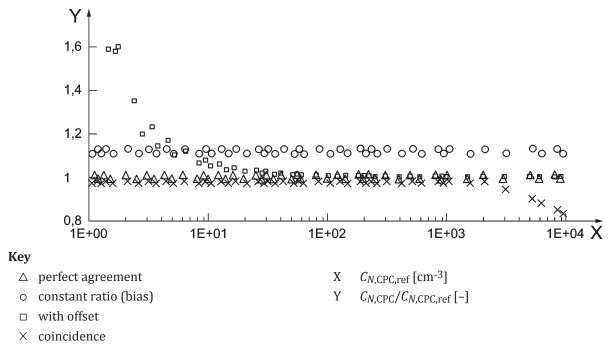


Figure A.4 — Typical patterns of residual plots with good precision

A.3.1.2 Sample size calculation

The main purpose of the sample size calculation is to have a high chance of detecting a statistically significant effect, or in other words, to check if two arbitrary data sample arithmetic means μ_1 and μ_2 differ significantly. The fundamental assumption is that the data sample arithmetic means have normal distributions.

For any set of samples with continuous measurements, the required sample size can be determined by specifying the following quantities:[13][17]

- the true standard deviation σ of the true value μ , which is approximated by the standard deviations of the sample arithmetic means s;
- a relevant positive difference δ between two arithmetic means;
- the significance level α ;
- the power 1β .

The significance level α quantifies the probability for neglecting a true difference, while β quantifies the probability for approving a non-significant difference; hence, the power $1 - \beta$ is the probability for approving a significant difference.

A minimum sample size *n* can be estimated as the lowest integer which fulfils the inequality

$$n > \left(\frac{z_{1-\alpha} + z_{1-\beta}}{\varepsilon}\right)^2 \tag{A.1}$$

where

 $z_{1-\alpha}$ and $z_{1-\beta}$ are respective percentiles of the normal distribution and

 ε is the effect size.

$$\varepsilon = \frac{\delta}{s} \tag{A.2}$$

Commonly used in statistical hypothesis testing are a significance level α of 0,05 and a power of 0,8 (with 1,65 and 0,842 for the 95 percentile and the 20 percentile respectively). In this example, an effect size ε = 0,5 (which is a medium effect in statistics) requires a sample size of 25. The sample size increases when ε decreases. Thus, detecting small but significant differences in sample arithmetic means needs large sample sizes.

A.3.1.3 Linear regression

If the residual plot shows that there is a linear relation between the test CPC and the reference instrument, then linear regression (ordinary least-squares regression analysis, unless specified otherwise) can be applied (x-axis is the reference instrument concentration and y-axis is the test CPC concentration). Note that, in order to have unbiased results from linear regression the following conditions must hold [14]: (i) the x values (reference instrument) are measured with negligible error; (ii) the relationship between each x (reference instrument) and y (test CPC) is linear; (iii) successive errors (differences between y and x) are not correlated (independent); (iv) the variance of the error terms is constant for each value of x; (v) the error terms are normally distributed. The assumption testing is conducted by plotting the residuals (difference between the y value and the predicted y value) versus x. (similarly with Figure A.3). When one of the assumptions is violated, then transformation of the data can help (see Reference [21]).

Usually in regression, the bias in the CPC results is indicated by the difference of the slope from one. The precision of the individual measurements is given by the residual standard deviation of the fit. The offset of the system under calibration is indicated by the value of the intercept. The inverse of the slope is used as the correction factor for the CPC (so that the plateau reaches the value 1). Another key

quantity, the uncertainty in the slope, is obtained from the formulae for regression analysis found in the literature or statistical packages. Where possible, the regression line typically is forced through zero because a properly operating CPCs should have no offset. Figure A.5 shows the regression graph for the data of Table A.1. The regression was forced through zero because there was no offset.

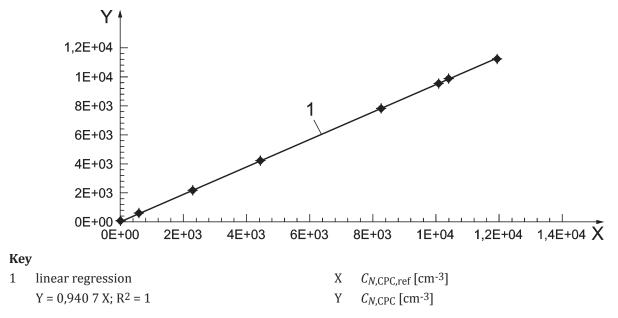


Figure A.5 — Simple linear regression plot between the test CPC and the reference instrument (data from <u>Table A.1</u>). The slope is 0,940 7

The cases of Figure A.4 are shown in Figure A.6 in a reference instrument — test CPC graph. Due to the big concentration range measured by the CPCs, it is difficult to identify any non-ideal behaviour.

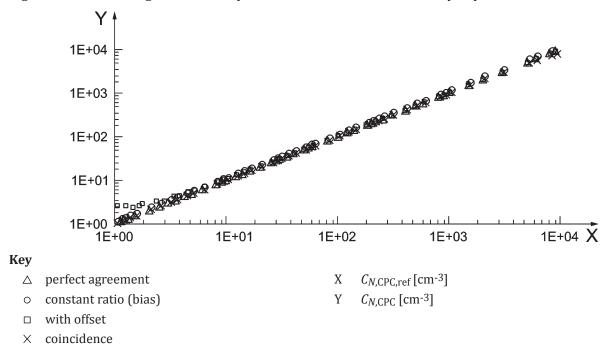


Figure A.6 — Typical patterns of non-ideal behaviour of CPCs (see Figure A.4 and discussion there)

A.3.1.4 Evaluation of the CPC linearity

Two correction factors can be calculated depending on the method used: One is the arithmetic mean of the ratios of the two instruments (Figure A.3, 0,938) and the other is the slope from the linear regression between the two instruments (Figure A.5, 0,940 7). When the response of the test CPC is linear and the other assumptions underlying the linear regression are met, the regression results are statistically optimal, however, for data sets with fairly evenly spaced measurements the two results will often be almost identical. The difference in the two analyses arises because the estimated slope from the regression analysis is a weighted arithmetic mean with heavier weight on the more extreme concentrations, which correctly leverages the relative information about the value of the slope in each data point. In contrast, the weights for all data points are the same if individual ratios are averaged. The degree of difference between the two analyses is determined by the placement of the points along the x-axis.

Typically the results of the two methods are very close. If big differences are found, then it has to be examined if one of the assumptions is not valid. If this is not the case, then the user should decide which factor is appropriate for the specific application (e.g. linear regression is appropriate when measurements are at the high end of the calibration).

A.3.2 Lower detection limit

The detection efficiency of a CPC is a sigmoid like curve that goes from 0 to a maximum value (the plateau efficiency). It can reach 1 after correcting with the inverse of the slope, see above.

The size for which the detection efficiency is half of the plateau efficiency (or 50 % if the plateau efficiency has been normalized to 1) is often called lower detection limit or cut-off size of the CPC. The above mentioned diameters can be determined experimentally (i.e. finding these sizes) or they can be estimated by using formulae that fit the measured detection efficiencies at the steep part of the curve. Various simple analytic formulas have been used. For a review, see References [28], [45], [47], and [59].

An example is found in References [33], [46], and [55]:

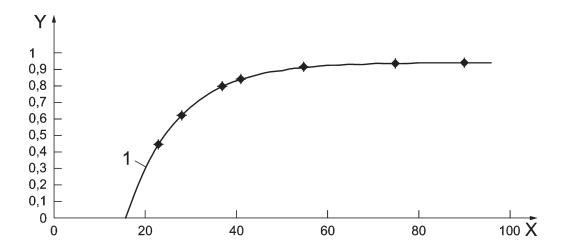
$$\eta(d) = b \left[1 - e^{\left(\frac{a_1 - d}{a_2 - a_1} \ln 2\right)} \right] \tag{A.3}$$

where

b is the slope and

 a_1 and a_2 are free parameters. The value of a_2 gives the lower detection limit.

Figure A.7 shows the measured detection efficiencies for the example of Table A.1 and the fit according to Formula (A.3). Based on the fit: $a_1 = 15,1$ nm and $a_2 = 23,6$ nm.



Key

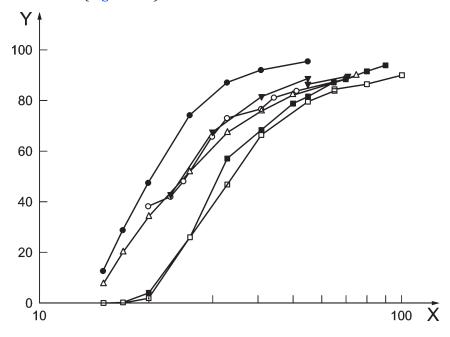
- 1 fit
- X d [nm]
- Y detection efficiency [-]

Figure A.7 — Fitted detection efficiency curve of the test CPC as a function of the size

Annex B (informative)

Effect of particle surface properties on the CPC detection efficiency

There are several data sets in the literature that show dependence of the detection efficiency of CPCs on, for example, the chemical composition of sampled particles.[8][9][23][26][28][29][33][34][47][50][54][57] [58] Published data in earlier work were on 1-butanol-based CPCs in which the material dependence was studied mainly with sodium chloride and silver. Some difference in the detection efficiency was observed between the two materials in the size range near the lower detection limit, and silver seemed to show slightly higher detection efficiencies than sodium chloride. More recent work with 1-butanol-based CPCs investigated a wider variety of materials which showed that oils were easiest to detect while inorganic salts were hardest (Figure B.1).



Key

- poly-alpha-olefin (PAO)
- **▼** diffusion flame soot
- sucrose
- o polystyrene latex (PSL)
- \triangle oxidized silver
- □ sodium chloride

X particle size [nm]

Y detection efficiency [%]

Figure B.1 — An example data set that presents the chemical composition dependence of the detection efficiency of a 1-butanol-based CPC¹⁾

For recently commercialized water-based CPCs, dependence of the detection efficiency on the particle chemical composition has been also studied and reported in the literature. In contrast to the case of 1-butanol-based CPCs, inorganic salts are easier to detect than oils for water-based CPCs.

¹⁾ Reprinted from Reference [57] with permission from Elsevier.

BS ISO 27891:2015 ISO 27891:2015(E)

Background theory for the dependence of the CPC detection efficiency on particle chemical composition was discussed in References [8], [9], [25], and [42] in terms of solubility and surface wettability, i.e. two physicochemical parameters that indicate the strength of the interaction between the CPC working fluid and the particle.

These observations in the literature provide evidence that detection efficiencies of the same CPC obtained by calibrations with particles of different chemical compositions may not agree with each other, especially at particle sizes near the lower detection limit. Therefore, the particle chemical composition shall always be specified when detection efficiencies are certified, and it cannot be assumed that comparable results would be obtained for particles of different chemical composition. In addition, for the reference CPC, the certificate can only be assumed to be valid for the same particle material that is specified on the certificate (see <u>5.4.6</u>).

Annex C (informative)

Example calibration certificates

C.1 General

This Annex contains examples of the certificates referred to within the International Standard. Specifically these are the following:

- Example completed certificate for a CPC calibrated against an FCAE (C.2);
- Example blank certificate for a CPC calibrated against a reference CPC (C.3);
- Example blank calibration certificate for an FCAE (C.4);
- Example blank calibration certificate for a reference CPC (C.5).

C.2 Example completed certificate for a CPC calibrated against an FCAE

Calibration results in the example certificate of this subclause are used in **Annex I**.

	Name and Address of the Institution Issuing the Certificate				
	CPC Model:	ABC 2000			
	CPC identification / serial number:	35701			
	Customer:	Quality Emissions Inc			
		For attention of A N Other			
	Order number:	2012/A/4235			
	Description: Calibration of a Model ABC (CPC)	2000 Condensation Particle Counter			
	Date of calibration:	1 June 2012			
		Page 1 of 2			
(Certificate) R	eference:				
Date of Issue:	Signed:	(Authorised Signatory)			
Checked by:	Name:	for Managing Director			

Date of instrument receipt in calibration room: 31 May 2012 (Calibration institute's) instrument identification number: CPC/2012/5/3 Instrument model and serial number: ABC 2000 S/N 35701

Result of initial inspection: No damage observed Result of instrument functionality test: No problems observed Nominal flow rate 1 l/min Settings of internal parameters (if any):

Concentration ranges of instrument counting modes Single particle counting mode (with coincidence

correction) up to 10 000 cm⁻³ (taken from operating manual):

Type and identification of Faraday Cup Aerosol Electrometer: XYZ 1000

FCAE calibration certificate reference: NMI_FCAE_CAL_60

Corrections made to FCAE reading, e.g. if the measured flow within tolerance: certificate flow 0,99 l/min;

differs from the certificate flow: measured flow 0,95 l/min Data acquisition method: ABC software version 3

Date of calibration: 1 June 2012

21 °C, 996 mbar Laboratory temperature and pressure: Sample pressure relative to laboratory pressure: -1.4 mbar

Measured test CPC flow rate (volumetric flow under laboratory 0.99 l/min

conditions):

Uncertainty in measured flow rate a:

Flow meter used (model, identification, calibration due date): IJK 179A Mass Flow Meter S/N 12341, calibration

due November 2012.

Particle material and gas used for calibration: soot in air

Method of particle generation: diffusion flame generator with conditioning Instrument zero reading: 0 particles per cm³ (mean value of 2 minutes)

Table of results:

Particle size (nm)	70	
Uncertainty in size (nm) ^a	5	
Nominal concentration (cm ⁻³)	7 000	
Measured fraction of doubly charged particles during calibration	0.049 9	
Measured fraction of triply charged particles during calibration	0,001 6	
Detection efficiency η	0,963 5	
Uncertainty in detection efficiency ^a	0,069 2	

Reference of calibration procedure followed:

Certificate expiry date (the certificate will also be invalid if operational problems are observed or the CPC is exposed to

excessive quantities of particles):

Internal procedure QPAS-548

31 May 2013

Reference: Page 2 of 2

Checked by:

The reported expanded uncertainty is based on standard uncertainty multiplied by a coverage factor k = 2, providing a level of confidence of approximately 95%.

C.3 Example blank certificate for a CPC calibrated against a reference CPC

	Name and Address of the Insti	tution Issuing the Certificate
	CPC Model: CPC identification / serial number	r:
	Customer:	
	Order number:	
	Description: Calibration of a Mod	el XX Condensation Particle Counter (CPC)
	Date of calibration:	
		Page 1 of 2
(Certificate) Re	ference:	J
Date of Issue:	Signed:	(Authorised Signatory)
Checked by:	Name:	for Managing Director

Date of instrument receipt in calibration room:			
(Calibration institute's) instrument identification nu	ımber:		
Instrument model and serial number:			
Result of initial inspection:			
Result of instrument functionality test:			
Settings of internal parameters (if any):			
Concentration ranges of instrument counting mode	S		
(taken from operating manual):			
Type and identification of Reference CPC:			
Reference CPC calibration certificate reference:			
Corrections made to Reference CPC reading, e.g. if	f the measured flow		
differs from the certificate flow:			
Data acquisition method:			
Date of calibration:			
Laboratory temperature and pressure:			
Sample pressure relative to laboratory pressure:			
Managed Asset CDC Garage (C. 1)	1-b		
Measured test CPC flow rate (volumetric flow undeconditions) [cm ³ /s]:	er laboratory		
Uncertainty in measured flow rate a:	d d-4-2		
Flow meter used (model, identification, calibration	aue datej:		
Particle material and gas used for calibration:			
Method of particle generation:			
		3.6 1	<i>c</i>
Instrument zero reading:		x particles per cm³ (mean valu	ie of y minutes j
Table of results:			
Particle size (nm)			
Uncertainty in size (nm) ^a			
Nominal concentration (cm ⁻³)			
Measured fraction of doubly charged particles			
during calibration			
Measured fraction of triply charged particles			
during calibration			
Detection efficiency η			
Uncertainty in detection efficiency ^a			
Reference of calibration procedure followed:			
Certificate expiry date (the certificate will also	be invalid if		
operational problems are observed or the CPC is			
excessive quantities of particles):			
Reference:			Page 2 of 2
Checked by:			
,			
^a The reported expanded uncertainty is based on level of confidence of approximately 95 %.	standard uncertainty	multiplied by a coverage factor	k=2, providing a
rever of confidence of approximately 75 70.			
		<u> </u>	

C.4 Example blank calibration certificate for an FCAE

Name and Address of the Institution Issuing the Certificate				
		ISO 17025 accreditation (or equivale		
		is not a National Measurem		
			,	
Aerosol Electr	ometer Model:			
Aerosol Electr	ometer identificat	ion / serial number:		
Customer:				
Order number	:			
Description: C	alibration of a Mod	lel XX Aerosol Electrometer		
Date of calibra	ntion:			
			Page 1 of 2	
			-	
(Certificate) Reference:				
Date of Issue:	Signed:	(Authorised Signatory)		
Checked by:	Name:	for Managing Director		

Date of instrument receipt in calibration room: (Calibration institute's) instrument identification num	nber:				
Instrument model and serial number:					
Result of initial inspection: Result of instrument functionality test: Data acquisition method: Date of calibration:					
Laboratory temperature and pressure: Sample pressure relative to laboratory pressure:					
Nominal set flow rate [cm³/s]: Measured flow rate (volumetric flow under laboratory conditions [of Uncertainty in measured flow rate a: Particle material and gas used for calibration: Method of particle generation:	cm ³ /s]:				
Instrument zero reading ^b : Table of results:	[C.cm ⁻³]				
Particle size (nm)					
Uncertainty in size (nm) ^a					
Zero-corrected reference charge concentration (C.cm ⁻³) ^b					
Standard uncertainty in zero-corrected reference charge concentration (C.cm ⁻³) b					
Instrument zero-corrected charge concentration (C.cm ⁻³) ^b					
Repeatability of instrument zero-corrected charge concentration (C.cm ⁻³) b,c					
Detection efficiency η					
Uncertainty in detection efficiency ^a					
Observations/comments:					
Equipment used (model, identification, calibration du Reference of calibration procedure followed: Certificate expiry date:	e date): E.g. re	ference electrometer, fl	ow meter		
Reference:			Page 2 of 2		
Checked by:					
The reported expanded uncertainty is based on standard uncertainty multiplied by a coverage factor $k = 2$, providing a level of confidence of approximately 95%.					
b This may be derived from a combination of current a	nd flow rate.				
^c The repeatability is the standard deviation of X repeated measurements.					

C.5 Example blank calibration certificate for a reference CPC

Name and Address of the Institution Issuing the Certificate				
		ISO 17025 accreditation (or equivalent) reference (if the Institution issuing the certificate is not a National Measurement Institute)		
CPC Model: CPC identifica	ntion / serial nun	nber:		
Customer:				
Order numbe	r:			
Description: (Calibration of a M	Model XX Condensation Particle Counter (CPC)		
Date of calibr	ation:			
		Page 1 of 2		
(Certificate) Reference:				
Date of Issue:	Signed:	(Authorised Signatory)		
Checked by:	Name:	for Managing Director		

Date of instrument receipt in calibration room: (Calibration institute's) instrument identification nur Instrument model and serial number:	mber:			
Result of initial inspection: Result of instrument functionality test: Settings of internal parameters (if any): Concentration ranges of instrument counting modes (taken from operating manual):				
Data acquisition method: Date of calibration:				
Laboratory temperature and pressure: Sample pressure relative to laboratory pressure: Nominal set flow rate [cm ³ /s]: Measured flow rate (volumetric flow under laboratory conditions) [cm ³ /Uncertainty in measured flow rate ^a : Particle material and gas used for calibration:	[s]:			
Method of particle generation:				
Instrument zero reading:		x particles per cm ³ (me	an value of y minutes).	
Table of results:	_			
Particle size (nm)				
Uncertainty in size (nm) ^a				
Nominal concentration (cm ⁻³)				
Measured fraction of doubly charged particles during calibration				
Measured fraction of triply charged particles during calibration				
Detection efficiency η				
Uncertainty in detection efficiency ^a				
Estimates of CPC d_{90} and d_{50} values (and how they were obtained): Equipment used (model, identification, calibration do Reference of calibration procedure followed: Certificate expiry date (the certificate will also be operational problems are observed or the CPC is excessive quantities of particles):	e invalid if	neter, flow meter		
Reference:			Page 2 of 2	
Checked by:				
•				
The reported expanded uncertainty is based on standard uncertainty multiplied by a coverage factor $k=2$, providing a level of confidence of approximately 95%.				

Annex D

(normative)

Calculation of the CPC detection efficiency

D.1 Sizes and concentrations of particles in the calibration aerosol from the DEMC

For calibration measurements according to <u>Clauses 6</u> and <u>7</u>, particles from a primary aerosol source pass through a DEMC, which delivers particles with known electrical mobility. The characteristic electrical mobility of particles classified by a DEMC is a function of the geometry, volumetric flow rates and supplied voltage of the DEMC. For a cylindrical DEMC with the sheath air re-circulated at flow rate q_s , the characteristic electrical mobility, when the voltage U is supplied, is

$$Z(U) = \frac{q_{\rm S} \ln(r_2/r_1)}{2\pi L U} \propto \frac{1}{U}$$
 (D.1)

where

L is the effective electrode length between the aerosol inlet and outlet;

 r_1 and r_2 are the radii of the inner and outer electrodes, respectively.

For a detailed discussion, see ISO 15900:2009, Annex E.

The electrical mobility of a particle depends on its size and electric charge. The relationship between electrical mobility and particle size for spherical particles can be described by

$$Z(d,p) = p \frac{e}{3\pi\mu_{\text{gas}}d} \left\{ 1 + \frac{2l_{\text{gas}}}{d} \left[1,165 + 0,483 \exp\left(-\frac{0,997d}{2l_{\text{gas}}}\right) \right] \right\}$$
 (D.2)

where

d is the particle diameter;

p is the number of net charges on a particle;

e is the elementary charge;

 μ_{gas} is the dynamic viscosity of the carrier gas;

 l_{gas} is the mean free path of the carrier gas molecules.

For dry air at 296,15 K and 101,3 kPa, the following values apply (ISO 15900):

$$\mu_{\rm gas}$$
 = 1,832 45 × 10⁻⁵ kg m⁻¹ s⁻¹

$$l_{\rm gas}$$
 = 6,730 × 10⁻⁸ m

Figure D.1 shows the relationship between electrical mobility, DEMC-voltage and particle sizes. When the DEMC voltage is set at U, the aerosol coming from the DEMC has the electrical mobility of Z(U) and may contain singly charged particles of size $d_1(U)$, doubly charged particles of $d_2(U)$, triply charged particles of $d_3(U)$, and so on.

The total number concentration $C_N(U)$ of particles contained in the DEMC outlet flow can therefore consist of particles of $C_N(d_p(U))$ with p net charges and corresponding sizes $d_p(U)$. For simplicity, the argument U is omitted in the following formulae when not explicitly needed.

$$C_N = \sum_{p \ge 1} C_N(d_p) \tag{D.3}$$

The fraction of the particles with p net charges, ϕ_p , which is defined by Formulae (6) and (15), can be expressed with the notation here as

$$\phi_p = \frac{C_N(d_p)}{C_N} \tag{D.4}$$

and depends on the size distribution and charge distribution of the primary aerosol. The knowledge of ϕ_p and C_N is necessary for calculation of the detection efficiency of the test CPC from measurement results (see D.3).

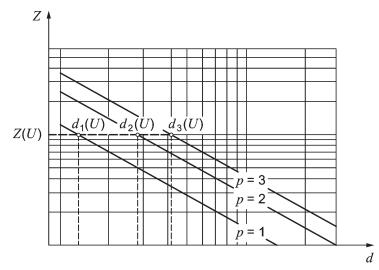


Figure D.1 — Relationship between electrical mobility, DEMC-voltage and particle sizes

D.2 Formulae for calculation of the detection efficiency of the test CPC

D.2.1 General formulae

An FCAE will measure a current (or derived particle number concentration $C_{N,\text{FCAE}}$ assuming singly charged particles) according to the number of elementary charges per particle, its detection efficiency $\eta_{\text{FCAE}}(d_n)$ and splitter bias β :

$$C_{N,\text{FCAE}} = \sum_{p \ge 1} C_N(d_p) \cdot p \cdot \eta_{\text{FCAE}}(d_p) \cdot \beta$$

$$= C_N \cdot \beta \sum_{p \ge 1} \phi_p \cdot p \cdot \eta_{\text{FCAE}}(d_p)$$
(D.5)

A test CPC will count these particles according to its size dependent detection efficiency $\eta_{\text{CPC}}(d_p)$:

$$C_{N,CPC} = \sum_{p \ge 1} C_N(d_p) \cdot \eta_{CPC}(d_p)$$

$$= C_N \sum_{p \ge 1} \phi_p \cdot \eta_{CPC}(d_p)$$
(D.6)

For the reference CPC the splitter bias β has to be considered:

$$C_{N,\text{CPC,ref}} = \sum_{p \ge 1} C_N(d_p) \cdot \eta_{\text{CPC,ref}}(d_p) \cdot \beta$$

$$= C_N \cdot \beta \sum_{p \ge 1} \phi_p \cdot \eta_{\text{CPC,ref}}(d_p)$$
(D.7)

It is assumed below that the detection efficiencies of the FCAE and reference CPC are constant in the size range above d_1 . Based on this assumption, Formulae (D.5) and (D.7) give the following:

$$C_{N,\text{FCAE}} = \eta_{\text{FCAE}} \cdot \beta \cdot C_N \sum_{p \ge 1} \phi_p \cdot p \qquad \text{(for the FCAE case)}$$
(D.8)

$$C_{N,CPC,ref} = \eta_{CPC,ref} \cdot \beta \cdot C_N$$
 (for the reference CPC case)

where

 η_{FCAE} is the efficiency of the FCAE; and

 $\eta_{\text{CPC,ref}}$ is the size-independent plateau efficiency of the reference CPC.

From Formulae (D.6), (D.8), and (D.9), the detection efficiency at size d_1 of the test CPC is described as

$$\eta_{\text{CPC}}(d_1) = \frac{C_{N,\text{CPC}} - C_N \sum_{p \ge 2} \phi_p \cdot \eta_{\text{CPC}}(d_p)}{C_{N,\text{FCAE}} - \eta_{\text{FCAE}} \cdot \beta \cdot C_N \sum_{p \ge 2} \phi_p \cdot p} \cdot \eta_{\text{FCAE}} \cdot \beta \qquad \text{(for the FCAE case)}$$
(D.10)

$$\eta_{\text{CPC}}(d_1) = \frac{C_{N,\text{CPC}} - C_N \sum_{p \ge 2} \phi_p \cdot \eta_{\text{CPC}}(d_p)}{C_{N,\text{CPC},\text{ref}} - \eta_{\text{CPC},\text{ref}} \cdot \beta \cdot C_N \sum_{p \ge 2} \phi_p} \cdot \eta_{\text{CPC},\text{ref}} \cdot \beta \qquad \text{(for the reference CPC case)}$$
(D.11)

To calculate $\eta_{\text{CPC}}(d_1)$ with Formula (D.10) or (D.11), the values of $\eta_{\text{CPC}}(d_p)$ shall be known for $p \geq 2$, which are usually not available. Therefore, further assumptions are needed to obtain $\eta_{\text{CPC}}(d_1)$, which are described in D.2.2 and D.2.3. The fraction of particles with net charge ϕ_p equals zero, when multiply charged particles are absent (this case is dealt with in D.2.4). Subclause D.3 describes how to calculate ϕ_p and C_N when the calibration aerosol contains multiply charged particles.

D.2.2 Calibration in the size range where the detection efficiency of the test CPC varies with size

In the following procedure, the value of $\eta_{CPC}(d_1)$ is estimated as the arithmetic mean of $\eta_{CPC}(d_1)$ values under two extreme assumptions.

Calculate $\eta_{CPC}(d_1)$ assuming that $\eta_{CPC}(d_p)$ for $p \ge 2$ are constant and equal to $\eta_{CPC}(d_1)$. Since we underestimate $\eta_{CPC}(d_p)$ for $p \ge 2$ in this case, $\eta_{CPC}(d_1)$ will be overestimated, according to Formulae (D.10) and (D.11).

Under this assumption, Formula (D.6) turns out to be

$$C_{N,\text{CPC}} = \eta_{\text{CPC}}(d_1) \cdot C_N \tag{D.12}$$

With this and Formulae (D.8) and (D.9), the following formulae can be derived:

$$\eta_{\text{CPC,a}}(d_1) = \frac{C_{N,\text{CPC}}}{C_{N,\text{FCAE}}} \cdot \eta_{\text{FCAE}} \cdot \beta \sum_{p \ge 1} \phi_p \cdot p \qquad \text{(for the FCAE case)}$$

$$\eta_{\text{CPC,a}}(d_1) = \frac{C_{N,\text{CPC}}}{C_{N,\text{CPC,ref}}} \cdot \eta_{\text{CPC,ref}} \cdot \beta \qquad \text{(for the reference CPC case)}$$
(D.13)

$$\eta_{\text{CPC,a}}(d_1) = \frac{C_{N,\text{CPC}}}{C_{N,\text{CPC,ref}}} \cdot \eta_{\text{CPC,ref}} \cdot \beta \qquad \text{(for the reference CPC case)}$$

The subscript "a" in $\eta_{CPC,a}(d_1)$ indicates that the value is obtained under the assumption above and gives the upper limit for $\eta_{CPC}(d_1)$. For using Formula (D.13), the values of ϕ_p shall be known (see D.3).

Calculate $\eta_{CPC}(d_1)$ assuming that $\eta_{CPC}(d_p)$ are constant for $p \ge 2$ and equal to η'_{CPC} where η'_{CPC} is the detection efficiency of the test CPC at a large size (e.g. 100 nm) where it is constant against size. Since we overestimate $\eta_{CPC}(d_p)$ for $p \ge 2$ in this case, $\eta_{CPC}(d_1)$ will be underestimated, according to Formulae (D.10) and (D.11).

Under this assumption, the following formulae can be derived from Formulae (D.10) and (D.11):

$$\eta_{\text{CPC,b}}(d_1) = \frac{C_{N,\text{CPC}} - \eta'_{\text{CPC}}C_N \sum_{p \geq 2} \phi_p}{C_{N,\text{FCAE}} - \eta_{\text{FCAE}} \cdot \beta \cdot C_N \sum_{p \geq 2} \phi_p \cdot p} \cdot \eta_{\text{FCAE}} \cdot \beta \quad \text{(for the FCAE case)}$$

$$\tau_{\text{CPC,b}}(d_1) = \frac{C_{N,\text{CPC}} - \eta'_{\text{CPC}}C_N \sum_{p \geq 2} \phi_p}{C_{N,\text{CPC,ref}} - \eta_{\text{CPC,ref}} \cdot \beta \cdot C_N \sum_{p \geq 2} \phi_p} \cdot \eta_{\text{CPC,ref}} \cdot \beta \quad \text{(for the reference CPC case)}$$

$$\tau_{\text{CPC,b}}(d_1) = \frac{C_{N,\text{CPC,ref}} - \eta_{\text{CPC,ref}} \cdot \beta \cdot C_N \sum_{p \geq 2} \phi_p}{C_{N,\text{CPC,ref}} \cdot \beta \cdot C_N \sum_{p \geq 2} \phi_p} \cdot \eta_{\text{CPC,ref}} \cdot \beta \quad \text{(for the reference CPC case)}$$

$$\tau_{\text{CPC,b}}(d_1) = \frac{C_{N,\text{CPC,ref}} - \eta_{\text{CPC,ref}} \cdot \beta \cdot C_N \sum_{p \geq 2} \phi_p}{C_{N,\text{CPC,ref}} \cdot \beta \cdot C_N \sum_{p \geq 2} \phi_p} \cdot \eta_{\text{CPC,ref}} \cdot \beta \quad \text{(for the reference CPC case)}$$

$$\eta_{\text{CPC,b}}(d_1) = \frac{C_{N,\text{CPC}} - \eta'_{\text{CPC}}C_N \sum_{p \ge 2} \phi_p}{C_{N,\text{CPC,ref}} - \eta_{\text{CPC,ref}} \cdot \beta \cdot C_N \sum_{p \ge 2} \phi_p} \cdot \eta_{\text{CPC,ref}} \cdot \beta \quad \text{(for the reference CPC case)}$$
(D.16)

The subscript "b" in $\eta_{CPC,h}(d_1)$ indicates that the value is obtained under the assumption above and gives the lower limit for $\eta_{CPC}(d_1)$. For using Formulae (D.15) and (D.16), the values of C_N as well as ϕ_p shall be known (see D.3).

c) Calculate $\eta_{CPC,b}(d_1)$ as the arithmetic mean of $\eta_{CPC,a}(d_1)$ and $\eta_{CPC,b}(d_1)$, i.e.

$$\eta_{\text{CPC}}(d_1) = \frac{\eta_{\text{CPC,a}}(d_1) + \eta_{\text{CPC,b}}(d_1)}{2}$$
(D.17)

The difference between $\eta_{CPC,a}(d_1)$ and $\eta_{CPC,b}(d_1)$ tells the magnitude of the uncertainty in evaluating $\eta_{\rm CPC}(d_1)$ by the above method and shall be taken into account in the uncertainty evaluation for $\eta_{\rm CPC}(d_1)$ in <u>Clauses 6</u> and <u>7</u>.

D.2.3 Calibration in the size range where the detection efficiency of the test CPC is known to be constant with size

When calibration is performed in the size range where the detection efficiency of the test CPC is known to be constant against size, formulae in <u>D.2.2</u> a) can be used, i.e.

$$\eta_{\text{CPC}}(d_1) = \frac{C_{N,\text{CPC}}}{C_{N,\text{FCAE}}} \cdot \eta_{\text{FCAE}} \cdot \beta \sum_{p \ge 1} \phi_p \cdot p \qquad \text{(for the FCAE case)}$$
(D.18)

$$\eta_{\text{CPC}}(d_1) = \frac{C_{N,\text{CPC}}}{C_{N,\text{CPC,ref}}} \cdot \eta_{\text{CPC,ref}} \cdot \beta$$
 (for the reference CPC case)

For using Formula (D.18), the values of ϕ_p shall be known (see D.3).

D.2.4 Calibration when multiply charged particles are absent in the calibration aerosol

When the calibration aerosol from the DEMC contains only singly charged particles with the size d_1 , no multiple charge corrections are necessary. In this case, ϕ_1 equals 1 and Formulae (D.8), (D.9), and (D.6) give the following:

$$C_{N,\text{FCAE}} = C_N \cdot \beta \cdot \eta_{\text{FCAE}}(d_1)$$
 (for the FCAE case)

$$C_{N,CPC,ref} = C_N \cdot \beta \cdot \eta_{CPC,ref}(d_1)$$
 (for the reference CPC case) (D.21)

$$C_{N,CPC} = C_N \cdot \eta_{CPC}(d_1)$$
 (for the test CPC case)

The detection efficiency of the test CPC at size d_1 , $\eta_{CPC}(d_1)$, can be obtained as

$$\eta_{\text{CPC}}(d_1) = \frac{C_{N,\text{CPC}}}{C_{N,\text{FCAE}}} \cdot \eta_{\text{FCAE}}(d_1) \cdot \beta \qquad \text{(for the FCAE case)}$$
(D.23)

$$\eta_{\text{CPC}}(d_1) = \frac{C_{N,\text{CPC}}}{C_{N,\text{CPC},\text{ref}}} \cdot \eta_{\text{CPC},\text{ref}}(d_1) \cdot \beta \qquad \text{(for the reference CPC case)}$$
(D.24)

D.3 Procedures for determination of ϕ_p and C_N

D.3.1 General

Two procedures for determination of ϕ_p and C_N are described below. The first procedure in <u>D.3.2</u> assumes that the primary aerosol reaches a known equilibrium charge distribution when passing through the bipolar charger. This assumption will be approximately met when the requirements of <u>5.4.2</u> are satisfied. The formulae given here only allow for the presence of particles with up to three elementary charges.

The second procedure in <u>D.3.3</u> relies on a DMAS determining an accurate size distribution of the particles. This DMAS needs to be an additional instrument. The result will depend upon the size of the particles, assumptions about their charge distribution within the DMAS software, and other DMAS software corrections. The knowledge of the charge distribution of the primary aerosol is not needed.

Published equilibrium charge distributions (see ISO 15900:2009, 4.5 and Annex A) are strictly valid only for spherical particles. Therefore it should be noted that both methods will be less accurate the more the particle shape differs from spherical particles. In extreme cases like fibres or loose agglomerates, the use of a second DEMC to eliminate multiply charged particles in the calibration aerosol becomes the method of choice.

Although the two procedures in principle should give the same results, users should determine which procedure is appropriate for their purposes.

D.3.2 Determination of the fraction of multiply charged particles using multiple DEMC voltages

D.3.2.1 Principle

D.3.2.1.1 General

According to Formulae (D.1) and (D.2), the diameter of doubly charged particles classified by a DEMC at voltage U, $d_2(U)$, is equal to the diameter of singly charged particles classified by the same DEMC at voltage 2U under the same flow condition, $d_1(2U)$, i.e. $d_2(U) = d_1(2U)$. Similarly, $d_3(U) = d_1(3U)$. Figure D.2 shows the relationship between these diameters and electrical mobility.

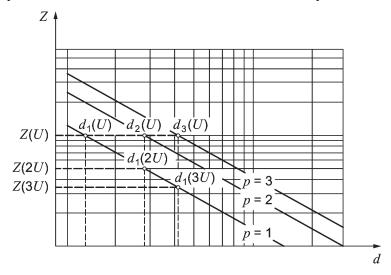


Figure D.2 — Relationship between particle sizes classified by a DEMC at voltages U, 2U and 3U

In the following, it is assumed that there are only particles of up to three charges in the aerosol from the DEMC at voltage U, and that there are only singly charged particles in the aerosol from the DEMC at voltage 2U and 3U, i.e.

$$C_N(U) = \sum_{p=1}^{3} C_N(d_p(U))$$
 (D.25)

$$\phi_p = \frac{C_N(d_p(U))}{C_N(U)} \quad (p = 1, 2, 3)$$
 (D.26)

$$C_N(2U) = C_N(d_1(2U))$$
 (D.27)

$$C_N(3U) = C_N(d_1(3U))$$
 (D.28)

When the charging probability of a particle with diameter d to carry p charges is expressed as $f_p(d)$ for the charge conditioner before the DEMC, the following relationships apply:

$$C_N(d_2(U)) = C_N(d_1(2U)) \cdot \frac{f_2(d_1(2U))}{f_1(d_1(2U))}$$
(D.29)

$$C_N(d_3(U)) = C_N(d_1(3U)) \cdot \frac{f_3(d_1(3U))}{f_1(d_1(3U))}$$
(D.30)

If alpha or beta radiation is used for ionization in the charge conditioner before the DEMC, the equilibrium charge distribution (see ISO 15900:2009, 4.5 and Annex A) applies to $f_p(d)$. For other charge conditioners, the appropriate charge distributions should be applied accordingly.

NOTE The above calculations do not take into account contributions of larger, multiply charged particles contained in the concentrations $C_N(2U)$ and $C_N(3U)$. For most primary aerosols, the error due to this simplification is not significant. Second order corrections using $C_N(4U)$ to correct $C_N(2U)$ and eventually $C_N(6U)$ to correct $C_N(3U)$ may become necessary if the primary aerosol has a wide size distribution (geometric standard deviation σ_g larger than 2) and if the calibration aerosol diameter d_1 for singly charged particles is larger than 100 nm. For $d_1 = 100$ nm and $\sigma_g = 2,1$, $C_N(d_1(U))$ changes by approximately 3 % if the second order correction is applied.

D.3.2.1.2 For calibration with an FCAE

 $For the \ case \ with \ the \ FCAE, the \ following \ relationship \ between \ actual \ and \ measured \ concentrations \ applies:$

$$C_N(U) = \eta_{\text{FCAE}} \sum_{p=1}^{3} C_N(d_p(U)) \cdot p$$
 (D.31)

$$C_N(2U) = \eta_{\text{FCAE}} \cdot C_N(d_1(2U)) \tag{D.32}$$

$$C_N(3U) = \eta_{\text{FCAE}} \cdot C_N(d_1(3U)) \tag{D.33}$$

where $C_N(U)$, $C_N(2U)$ and $C_N(3U)$ are the measured concentrations using the FCAE when the DEMC is set at voltages U, 2U and 3U, respectively. From Formulae (D.32) and (D.33), the actual concentrations $C_N(d_1(2U))$ and $C_N(d_1(3U))$ can be derived from the measured concentrations $C_N(2U)$ and $C_N(3U)$, i.e.

$$C_N(d_1(2U)) = \frac{C_N(2U)}{\eta_{\text{FCAE}}} \tag{D.34}$$

$$C_N(d_1(3U)) = \frac{C_N(3U)}{\eta_{\text{FCAE}}}$$
 (D.35)

Formulae (D.29) and (D.30) can be transformed with Formulae (D.34) and (D.35), respectively, as

$$C_N(d_2(U)) = \frac{C_N(2U)}{\eta_{\text{FCAE}}} \cdot \frac{f_2(d_1(2U))}{f_1(d_1(2U))}$$
(D.36)

$$C_N(d_3(U)) = \frac{C_N(3U)}{\eta_{\text{FCAE}}} \cdot \frac{f_3(d_1(3U))}{f_1(d_1(3U))} \tag{D.37}$$

The actual concentration $C_N(d_1(U))$ is then derived from Formulae (D.31), (D.36) and (D.37) as

$$C_{N}(d_{1}(U)) = \frac{C_{N}(U)}{\eta_{\text{FCAE}}} - 2C_{N}(d_{2}(U)) - 3C_{N}(d_{3}(U))$$

$$= \left[C_{N}(U) - 2C_{N}(2U) \cdot \frac{f_{2}(d_{1}(2U))}{f_{1}(d_{1}(2U))} - 3C_{N}(3U) \cdot \frac{f_{3}(d_{1}(3U))}{f_{1}(d_{1}(3U))}\right] / \eta_{\text{FCAE}}$$
(D.38)

With $C_N(d_2(U))$, $C_N(d_3(U))$ and $C_N(d_1(U))$ from Formulae (D.36), (D.37) and (D.38), respectively, $C_N(U)$ and ϕ_p can be obtained with Formulae (D.25) and (D.26).

D.3.2.1.3 For calibration with a reference CPC

For the case with the reference CPC, the following relationship between actual and measured concentrations applies:

$$C_N(U) = \eta_{\text{CPC,ref}} \sum_{p=1}^{3} C_N(d_p(U))$$
 (D.39)

$$C_N(2U) = \eta_{\text{CPC.ref}} \cdot C_N(d_1(2U)) \tag{D.40}$$

$$C_N(3U) = \eta_{CPC, ref} \cdot C_N(d_1(3U)) \tag{D.41}$$

where $C_N(U)$, $C_N(2U)$ and $C_N(3U)$ are the measured concentrations using the reference CPC when the DEMC is set at voltages U, 2U and 3U, respectively. From Formulae (D.40) and (D.41), the actual concentrations $C_N(d_1(2U))$ and $C_N(d_1(3U))$ can be derived from the measured concentrations $C_N(2U)$ and $C_N(3U)$, i.e.

$$C_N(d_1(2U)) = \frac{C_N(2U)}{\eta_{\text{CPC,ref}}}$$
(D.42)

$$C_N(d_1(3U)) = \frac{C_N(3U)}{\eta_{\text{CPC.ref}}}$$
(D.43)

Formulae (D.29) and (D.30) can be transformed with Formulae (D.42) and (D.43), respectively, as

$$C_N(d_2(U)) = \frac{C_N(2U)}{\eta_{\text{CPC,ref}}} \cdot \frac{f_2(d_1(2U))}{f_1(d_1(2U))}$$
(D.44)

$$C_N(d_3(U)) = \frac{C_N(3U)}{\eta_{\text{CPC,ref}}} \cdot \frac{f_3(d_1(3U))}{f_1(d_1(3U))} \tag{D.45}$$

The actual concentration $C_N(d_1(U))$ is then derived from Formulae (D.39), (D.44) and (D.45) as

$$C_{N}(d_{1}(U)) = \frac{C_{N}(U)}{\eta_{\text{CPC,ref}}} - C_{N}(d_{2}(U)) - C_{N}(d_{3}(U))$$

$$= \left[C_{N}(U) - C_{N}(2U) \cdot \frac{f_{2}(d_{1}(2U))}{f_{1}(d_{1}(2U))} - C_{N}(3U) \cdot \frac{f_{3}(d_{1}(3U))}{f_{1}(d_{1}(3U))} \right] / \eta_{\text{CPC,ref}}$$
(D.46)

With $C_N(d_2(U))$, $C_N(d_3(U))$, and $C_N(d_1(U))$ from Formulae (D.44), (D.45) and (D.46), respectively, $C_N(U)$ and ϕ_p can be obtained with Formulae (D.25) and (D.26).

D.3.2.2 Measurement procedure

- a) Measure $C_N(U)$, $C_N(2U)$ and $C_N(3U)$ using the FCAE or the reference CPC. U is the voltage set at the DEMC for singly charged calibration particles with diameter d_1 .
- b) Calculate $C_N(d_p(U))$ (p = 1, 2, 3) using Formulae (D.36) to (D.38) in case with the FCAE, or Formulae (D.44) to (D.46) in case with the reference CPC.
- c) Calculate $C_N(U)$ and ϕ_p using Formulae (D.25) and (D.26).
- d) At least 5 determinations shall be made at each particle size in order to evaluate the repeatability of the multiple charge correction, as required for the uncertainty calculation in <u>6.4</u>. The uncertainty components u(1), u(2), and u(3), referred to in <u>6.4</u>, shall be the standard deviations of the determinations of ϕ_1 , ϕ_2 , and ϕ_3 respectively.

NOTE It is recommended to monitor if the ratio $C_N(d_2(U))/C_N(U)$ remains constant. Variations in this ratio indicate either non-equilibrium charge distribution or non-repeatable p. For further testing see Annex K.

D.3.3 Measurement of the fraction of multiply charged particles with DMAS

The measurement of the various charge and size fractions in the calibration aerosol can be made with a DMAS as described in ISO 15900. To allow adjustment of the correct inlet flow for the DMAS, it is connected to the outlet of the manifold with mixing device (see Figure D.3).

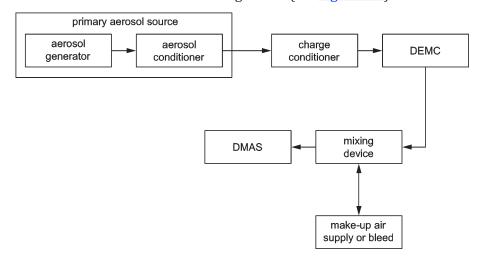
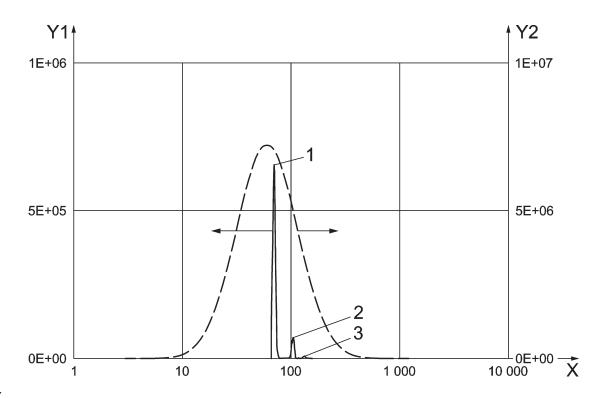


Figure D.3 — Setup for the measurement of the fraction of multiply charged particles with a DMAS

The data inversion of the DMAS measurement shall include multiple charge correction and diffusion loss correction. Measurement of the fraction of multiply charged particles by this method assumes that these corrections are correct and accurate. It is also assumed that aerosol reaches the known equilibrium charge distribution when passing through the charge conditioner of the DMAS.

If the electrical mobility distribution coming from the DEMC is narrow (i.e. the geometric standard deviation of singly charged particles less than 1,1) and if the size resolution of the DMAS is sufficient, the result of the measurement will consist of clearly separated peaks for each charge and size fraction. Figure D.4 shows an example.



Key

X d [nm]

Y1 calibration aerosol – $dC_N/d\log(d)$ [cm⁻³]

Y2 primary aerosol – $dC_N/d\log(d)$ [cm⁻³]

- 1 one elementary charge
- 2 two elementary charges
- 3 three elementary charges

NOTE 1 Primary aerosol: atomized NaCl, number median diameter 60 nm, geometric standard deviation 1,9.

NOTE 2 Calibration aerosol: after DEMC with sheath flow to aerosol flow ratio 10:1, set for singly charged particle size of 70 nm.

 $Figure \ D.4 - Example for size resolved peaks of calibration particles that were singly, doubly, and triply charged at the DEMC outlet and size distribution of the primary aerosol measured by DMAS$

The leftmost peak corresponds to the size of the calibration aerosol selected with the DEMC of the calibration setup. The next peak represents particles that were doubly charged at the DEMC outlet and so on. Each peak shall be integrated to obtain the corresponding particle number concentration as shown in Formula (D.47).

$$C_N(d_p) = C_{N,p,\text{DMAS}}(d_p) \tag{D.47}$$

The fraction ϕ_p for p net charges is calculated using Formula (D.48).

$$\phi_p = \frac{C_{N,p,\text{DMAS}}(d_p)}{\sum_{i\geq 1} C_{N,i,\text{DMAS}}(d_i)}$$
(D.48)

NOTE The number median diameter of each peak (either directly measured or curve fitted) is the representative size d_p for each charge fraction. Inserting d_p and p into Formula (D.2) may be used to verify that the electrical mobility of the DEMC remains unchanged for all found charge fractions. This is a good indication for a valid measurement.

Due to different flow rates for the calibration measurements and the measurement with the DMAS, the number concentrations may change in the calibration experiment. (That is, for example, C_N in the calibration measurement may be different from $\sum_{i\geq 1} C_{N,i,\mathrm{DMAS}}(d_i)$). The fractions ϕ_p , however, remain

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unchanged. The concentrations C_N can be calculated with Formula (D.49) using ϕ_p for the case when an FCAE is used:

$$C_N = \frac{C_{N,\text{FCAE}}}{\eta_{\text{FCAE}} \sum_{p \ge 1} \phi_p \cdot p} \tag{D.49}$$

Formula (D.49) assumes that the detection efficiency of the FCAE is constant for sizes d_p , $p \ge 1$ and equal to η_{FCAE} .

Similarly, for the case when a reference CPC is used for calibration, the total number concentration of particles is calculated with Formula (D.50):

$$C_N = \frac{C_{N,\text{CPC,ref}}}{\eta_{\text{CPC,ref}}} \tag{D.50}$$

Formula (D.50) assumes that the detection efficiency of the reference CPC is constant for sizes d_p , $p \ge 1$ and equal to $\eta_{\text{CPC,ref.}}$

Annex E (informative)

Traceability diagram

In order that results of CPC calibration that is performed according to this International Standard are regarded traceable to national standards, instruments used in the calibration, including the FCAE and reference CPC, shall be calibrated with metrological traceability to national standards. The figures in this Annex illustrate the metrological traceability chains for quantities that are influential to results of calibration of a CPC.

Measurement of volumetric flow rate includes measurement of temperature and pressure of the flow. Therefore, calibration traceability of volumetric flow rate includes calibration traceability of temperature and pressure.

See Figure E.1 below.

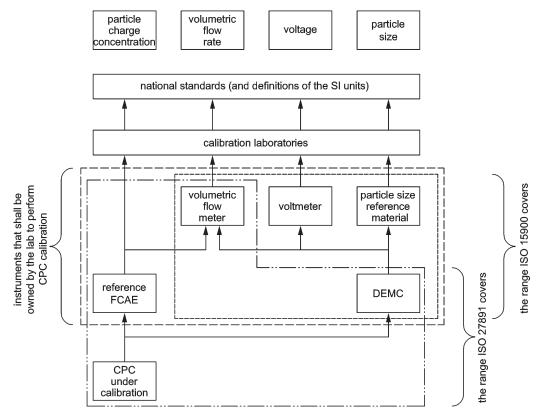


Figure E.1 — Traceability diagram for calibration with an FCAE

The four directly traceable quantities do not completely underpin the quantity measured by the CPC, particle number concentration. The extra parameter, the mean charge per particle used for the calibration, is determined by the experimental setup, and is controlled by the procedures and checks described in Annex D.

See <u>Figure E.2</u> below.

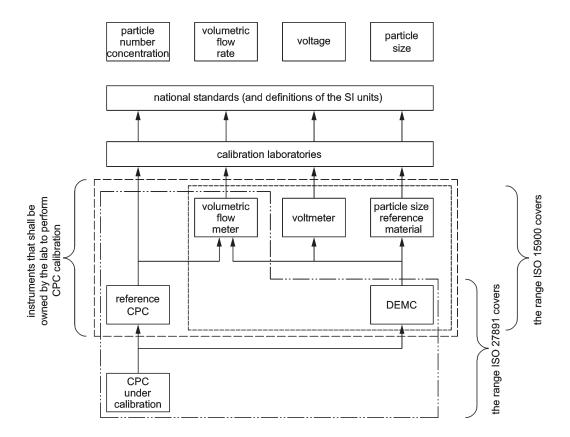


Figure E.2 — Traceability diagram for calibration with a reference CPC

Annex F (informative)

Diluters

F.1 General

Diluters are tools that allow reduction of particle concentration in a controlled manner. This Annex describes typical diluters. All of them have a mechanism to add clean, particle free dilution air, and efficient mixing after it is essential. For CPC calibration with the setup described in this International Standard, it is desirable that diluters have the same inlet and outlet flow rates.

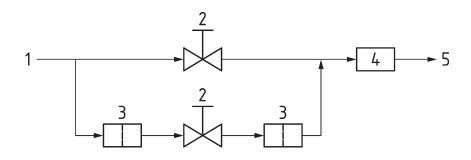
If the primary aerosol source makes high dilution factors necessary, cascades or combinations of dilution systems can become necessary. In general, dilution systems used for calibration should fulfill the following requirements:

- particle concentration in the dilution air less than 0,1 % of the number concentration of the aerosol in the diluted air flow;
- number median diameter of the calibration aerosol before and after dilution unchanged within ± 3 %;
- geometric standard deviation of the calibration aerosol before and after dilution unchanged within ± 3 %;
- if dilution air is delivered by an external source, the relative humidity of the dilution air should be less than 40% to avoid condensation growth of particles;
- flow variations within a diluter should be kept small enough to keep the diluted particle number concentration stable.

F.2 Dilution bridge

The dilution bridge has two flow paths with a flow restriction adjustment on each, while a particle filter is inserted in one of the paths. The incoming flow splits into the two flow paths at flow rates determined by relative restriction settings. If most of the flow goes into the path without the filter, the particle concentration does not change much. As the flow rate through the filter is increased relative to that through the path without the filter, the dilution effect increases and the concentration of the outgoing flow is reduced. To avoid unnecessary pressure drop in a dilution bridge, one of the two flow restriction adjustments should always be kept completely open.

See Figure F.1 below.



Key

- 1 in
- 2 valve
- 3 filter
- 4 mixer
- 5 out

Figure F.1 — Schematic of the dilution bridge

F.3 Dilution loop

In a dilution loop, filtered dilution air from downstream of the mixing device is continuously pumped back as dilution air. A flow restrictor (e.g. a needle valve or a mass flow controller) is used to set the dilution air flow and hence the dilution ratio. Compared to a dilution bridge, the dilution loop does not need a flow restriction adjustment in the aerosol flow. This keeps the pressure drop negligible in all operating conditions and avoids sheer stress on the aerosol particles.

See Figure F.2 below.

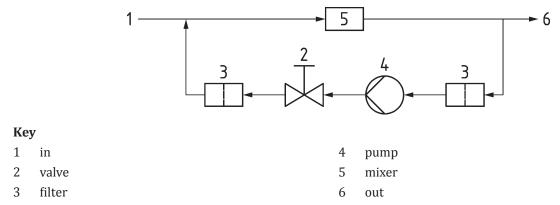
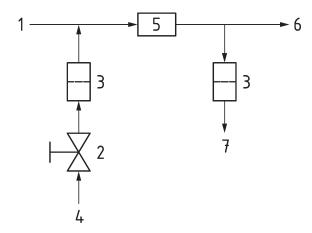


Figure F.2 — Schematic of the dilution loop

F.4 Dilution by addition of clean air and bleeding of excess air

In this type of diluter, the dilution air is delivered by an external source of compressed air. The combined inlet flow and dilution air flow should be higher than the diluted outlet flow. If the diluted outlet flow is controlled, e.g. by the flow control of the connected instrument(s), the excess flow may be simply vented to ambient air through a filter. The backpressure of the filter should be kept low to avoid forcing a flow through the connected instrument(s). Without such flow control, the excess flow path itself should have a mechanism to pull the flow by a pump and with a flow rate controller.

See Figure F.3 below.



Key

- 1 in 5 mixer 2 valve 6 out 3
 - filter ambient air
- compressed air

Figure F.3 — Schematic of the diluter with addition of clean air and bleeding of excess air

The dilution air and the inlet flow can be mixed in a venturi ejector, creating an under pressure at the inlet port. This so-called ejector diluter draws the inlet flow in a controlled manner, which may be of advantage if the primary aerosol source operates at reduced pressure.

F.5 Other dilution methods

Depending on the nature of the primary aerosol source, other dilution methods (e.g. rotating disc diluters) may be required or may have advantages over the methods discussed above. As long as the requirements outlined in <u>Clauses 5</u>, <u>6</u>, and <u>7</u> are fulfilled, such dilution methods may be applied.

Annex G

(normative)

Evaluation of the concentration bias correction factor between the inlets of the reference instrument and test CPC

G.1 General

The procedures in this Annex are intended to be performed prior to calibrating a CPC for detection efficiency and the resulting measures of bias are only strictly valid for the flow rates and the particle size present during the measurement. The correction factor β is configuration dependent and shall be measured according to the convention described in this Annex. If CPC detection efficiency calibrations are performed over a range of particle sizes, the splitter biases should be measured for each particle size. This check is not necessary when only the concentration is changed.

The evaluation of the bias correction factor β is feasible if

- a) two particle detectors are attached to a flow splitter that sample with *equal* (i.e. within 10 %) and constant inlet flow rates. (see <u>G.2</u> for further details), or if
- b) two particle detectors are attached to a flow splitter that sample with *unequal* and constant inlet flow rates (see <u>G.3</u> for further details).

To perform a measurement of the bias correction factor, the tubing connecting the detectors to the splitter and mixing device shall be electrically conductive.

G.2 Measurement of the bias correction factor β for equal detector flow rates

The technique does not require knowledge of the detection efficiency of either the reference instrument or the test CPC providing the detection efficiencies stay constant during the evaluation. Therefore, the procedure shall be performed keeping both the aerosol concentration and particle size as constant as possible. $|1 - \beta|$ will increase when conditions are not constant.

The bias correction factor β is defined as a correction factor for the concentration measurement made by the detectors attached to tubes A and B in two configurations, shown in Figures G.1 and G.2 respectively.

G.2.1 Measurement in the first configuration

With the configuration in Figure G.1, set the flow rates at q and feed particles of size d from the calibration particle generator system. Let the setup stabilize for 30 s. When using a CPC as the reference instrument, record n pairs of concentration readings (at least one pair of readings per second) of the reference instrument, $C_{N,\text{ref},1a}$, and that of the test CPC, $C_{N,\text{CPC},1a}$ during the next 30 s. Calculate the arithmetic mean concentrations with Formulae (G.1) and (G2):

$$\overline{C}_{N,CPC,1a} = \frac{1}{n} \sum_{i=1}^{n} C_{N,CPC,1a,i}$$
 (G.1)

$$\overline{C}_{N,\text{ref},1a} = \frac{1}{n} \sum_{i=1}^{n} C_{N,\text{ref},1a,i}$$
 (G.2)

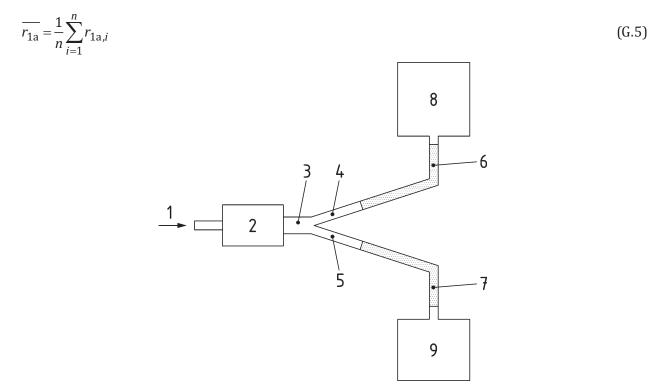
When using an FCAE as the reference instrument, the background and signal charge concentrations shall be measured. See Annex L for a recommended data recording method. Use Formulae (G.3) and (G.2) to calculate the arithmetic mean concentrations for the FCAE.

$$C_{N,\text{ref},1a,i} = \frac{C_{Q,1a,i} - C_{Q,0,1a,i}}{e}$$
(G.3)

Calculate r_{1a} with Formula (G.4)

$$r_{1a} = \frac{\overline{C}_{N,CPC,1a}}{\overline{C}_{N,ref,1a}}$$
 (G.4)

Repeat the above process at least two more times for a total of at least three measurements of r_{1a} . Calculate the arithmetic mean value of r_{1a} with Formula (G.5)



Key

1 calibration aerosol 6 tube A
2 mixer 7 tube B
3 flow splitter 8 reference instrument
4 port A 9 test CPC
5 port B

Figure G.1 — First connection configuration of the flow splitter and the tubes after it

G.2.2 Measurement in the second configuration

Change the experimental setup to the configuration in Figure G.2, in which the mixing device, the flow splitter, and the tubes to the instruments were flipped over. Let the setup stabilize for 30 s. When using a CPC as the reference instrument, record n pairs of concentration readings (at least one pair of

readings per second) of the reference instrument, $C_{N,ref,2}$, and that of the test CPC, $C_{N,CPC,2}$ during the next 30 s. Measure and calculate the average fraction r_2 in the second configuration similar to the first configuration.

Calculate r_2 with Formula (G.6)

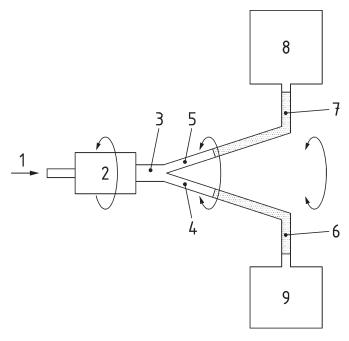
$$r_2 = \frac{\overline{C}_{N,\text{CPC},2}}{\overline{C}_{N,\text{ref},2}} \tag{G.6}$$

Repeat the above process at least two more times for a total of at least three measurements of r_2 . Calculate the arithmetic mean value of r_2 with Formula (G.7)

$$\overline{r_2} = \frac{1}{n} \sum_{i=1}^{n} r_{2,i} \tag{G.7}$$

NOTE This measurement can be substituted, without flipping over the mixing device, the flow splitter, and the tubes, by moving the two instruments, with the test CPC replacing the reference instrument, while the reference instrument is placed at the position of the test CPC in the first connection configuration.

After this measurement the mixing device and splitter assembly shall be returned to the configuration in Figure G.1 and one more repetition will be conducted.



Key

1	calibration aerosol	6	tube A
2	mixer	7	tube B
3	flow splitter	8	reference instrument
4	port A	9	test CPC
5	port B		

 $\begin{tabular}{ll} Figure~G.2-Schematic~when~the~mixing~device, the~flow~splitter, and~the~tubes~after~them~were~flipped~over \end{tabular}$

G.2.3 Measurement repeated in the first configuration

Calculate r_{1b} as in <u>G.2.1</u>. Calculate r_1 as the arithmetic mean of r_{1a} and r_{1b} .

G.2.4 Calculation of β and the associated uncertainty

If the measurement was performed at a constant particle size and concentration, the bias β and its uncertainty are given by Formulae (G.8) to (G.11):

$$\beta = \sqrt{\frac{\overline{r_2}}{\overline{r_1}}} \tag{G.8}$$

$$\left[\frac{u(\beta)}{\beta}\right]^2 = \left[\frac{1}{2} \cdot \frac{u(\overline{r}_1)}{\overline{r}_1}\right]^2 + \left[\frac{1}{2} \cdot \frac{u(\overline{r}_2)}{\overline{r}_2}\right]^2 \tag{G.9}$$

$$u(\overline{r}_{1}) = \sqrt{\frac{\sum_{i}^{n} (r_{1,i} - \overline{r}_{1})^{2}}{n(n-1)}}$$
(G.10)

$$u(\overline{r}_{2}) = \sqrt{\frac{\sum_{i}^{n} (r_{2,i} - \overline{r}_{2})^{2}}{n(n-1)}}$$
(G.11)

G.3 Measurement of the bias correction factor β for unequal detector flow rates

If the test CPC and the reference instrument cannot be operated at the same flow rate, for example because the flow rate of the reference instrument is fixed or the flow rate of the test CPC is outside of the range for which the reference instrument is specified, the bias correction factor β cannot be determined by switching the positions of the detectors and a value of β = 1 shall be used instead. The measurement uncertainty will increase compared to the method with equal flow rates described in <u>G.2</u>. The maximum ratio between the two flow rates shall be 7:1.

To estimate the measurement uncertainty of β for unequal flow rates, two particle counters with equal flow rates must be used. The sum of the equal flow rates must be less than the sum of the unequal flow rates. First, using the two particle counters with equal flow rates, perform the splitter bias measurement according to G.2. Calculate β using Formulae (G.1) to (G.8) and denote this value of β as $\beta_{\rm equal}$. The value of $\beta_{\rm equal}$ shall be 0,95 < $\beta_{\rm equal}$ < 1,05. If $\beta_{\rm equal}$ is outside of this range then the measurement cannot proceed. Calculate the uncertainty of $\beta_{\rm equal}$ using Formulae (G.9) to (G.11) and denote this value of uncertainty as $[u(\beta_{\rm equal})/\beta_{\rm equal}]^2$. Calculate the additional uncertainty contribution associated with unequal flow rates using Formula (G.12):

$$u(\beta_{\text{unequal}}) = |1 - \beta_{\text{equal}}|$$
 (G.12)

Calculate the total uncertainty of β for unequal flow rates using Formula (G.13):

$$\left[\frac{u(\beta)}{\beta}\right]^{2} = \left[\frac{u(\beta_{\text{equal}})}{\beta_{\text{equal}}}\right]^{2} + \left[\frac{u(\beta_{\text{unequal}})}{\beta_{\text{unequal}}}\right]^{2}$$
(G.13)

In Formula (G.13) and throughout the calibration, use $\beta_{unequal} = 1$.

When the two instruments with different flow rates are connected to the splitter, the length of the connection tubing shall be used to compensate the difference in the transfer losses according to Formula (G.15). For laminar tube flow, diffusion losses are independent of the tube diameter. [3] However, to decrease

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the uncertainty of the compensation, both sampling tubes should be equal in terms of their diameter and material, and selected such that the tube flow Reynolds number in both tubes is smaller than 1 500:

$$Re = \frac{4q}{\pi d\nu} < 1500 \tag{G.14}$$

where

Re is the tube flow Reynolds number;

q is the volumetric inlet flow rate of the test CPC or the reference instrument;

d is the inner diameter of the tube;

v is the kinematic viscosity of the carrier gas (air) in the tube.

With the length of the tubing to the reference instrument and the flow rate to the reference instrument given, the required length of the tubing to the test CPC can be calculated as shown in Formula (G.15):

$$l_{\text{CPC}} = l_{\text{ref}} \frac{q_{\text{CPC}}}{q_{\text{ref}}} \tag{G.15}$$

where

 l_{CPC} is the length of the tubing to the test CPC;

 l_{ref} is the length of the tubing to the reference instrument;

 $q_{\rm CPC}$ is the volumetric inlet flow rate of the test CPC;

 $q_{\rm ref}$ is the volumetric inlet flow rate of reference instrument.

Annex H

(informative)

Extension of calibration range to lower concentrations

H.1 General

This Annex describes a calibration process to measure the proportionality of the CPC response to an aerosol concentration. This is a relative calibration of the CPC response over a wide range of concentrations and is used to calibrate the CPC below the lower limit of the FCAE, as referenced in 6.3.6 and 7.3.6. Separate measurements made with an FCAE or reference CPC at high concentration are required to measure the constant of proportionality and provide an absolute calibration with traceability to SI units. A proportional response is a special case of a linear response with the ordinate intercept equal to zero, so a single correction factor applies over the range of concentrations tested.

The calibration process requires a stable aerosol diluter and two CPCs: the test CPC and a dummy CPC. Neither CPC is required to have an absolute calibration. All concentration measurements shall be corrected for coincidence, with the correction made using either a dead time subtraction using the CPC software or an iterative correction based on the CPC pulse width.

H.2 Diluter

H.2.1 Diluter requirements

The two requirements for the diluter are that it must produce a stable dilution factor with respect to time and over the range of aerosol concentrations of interest. In order to ensure stability of the dilution factor with respect to time, the volumetric flow rates through the diluter must remain constant and the particle loss through the diluter must not change over time. A monodisperse aerosol can be used to keep the particle loss constant with time. The stability of the diluter over the range of aerosol concentrations of interest must be measured according to the process in this Annex.

Figure H.1 shows the recommended diluter configuration for the proportionality test, but any diluter fulfilling the two requirements above can be used. This diluter is similar to the dilution bridge in Annex F. The diluter splits the incoming flow into two paths: one with a flow restriction and the other with a valve and filter. The flow restriction can be an orifice, a thin tube, or a valve, but it must not generate additional particles. The two paths recombine at the downstream end of the diluter. To monitor the stability of the diluter volumetric flow rates, pressure taps are located across the flow restriction and are used to monitor the stability of the unfiltered volumetric flow. If the CPC uses a critical orifice for flow control, it is ensured that the total volumetric flow through the diluter is held constant over time. If the CPC does not use a critical orifice, either the CPC volumetric flow rate must be monitored with a flow meter or the pressure drop across the filtered flow path must be monitored for stability.

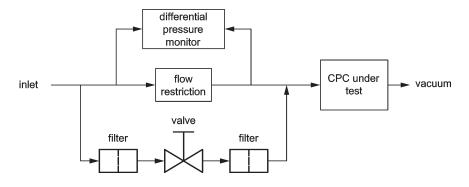


Figure H.1 — Schematic of the recommended diluter and CPC configuration

H.2.2 Diluter validation: the effect of particle concentration on flow rate

For the diluter to provide a stable dilution factor during the proportionality test, the diluter flow rates must be insensitive to a changing aerosol concentration. There are three flow rates through the diluter: the total diluter flow, the unfiltered flow through the flow restriction, and the filtered flow. The effect of aerosol on two of these flow rates must be measured. The flow meter used for this measurement should have a low pressure drop; it should also not be affected by changing particle concentration.

The process below is used to validate the diluter flow rate stability with respect to the presence of aerosol for the recommended setup shown in <u>Figures H.1</u> and <u>H.2</u>. In this process the total diluter flow and the unfiltered flow through the flow restriction are checked.

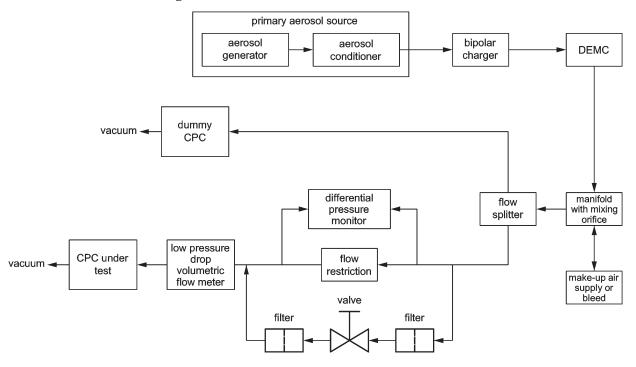


Figure H.2 — Schematic of the setup to validate the recommended diluter and CPC configuration

- a) Set up the experiment according to <u>Figure H.2</u>, but remove the low pressure drop volumetric flow meter from the inlet of the test CPC. Allow the instruments to warm up for 1 h, and let the flows and aerosol generator stabilize.
- b) With the DEMC voltage and sheath flow rate set to select the particle size of interest, adjust the aerosol conditioner so that the dummy CPC reads about 1 000 cm⁻³.

- c) Adjust the valve and/or flow restriction in the aerosol diluter so that the test CPC reads about 100 cm⁻³.
- d) Record the differential pressure across the flow restriction. It is helpful to use a flow restriction that provides at least 500 Pa differential pressure.
- e) Set the DEMC voltage to 0 V and wait for the CPCs to zero. Install the low pressure drop volumetric flow meter at the inlet of the test CPC. Let the flows and aerosol generator stabilize.
- f) Set the DEMC voltage to select the particle size of interest, and adjust the aerosol conditioner so that the dummy CPC reads the highest particle concentration desired for the proportionality test.
- g) Adjust the valve and/or flow restriction in the aerosol diluter so that the differential pressure is equal to that recorded in step d).
- h) Record the differential pressure across the flow restriction as $dP_{\rm ON}$. Then record the inlet flow rate of the CPC using the low pressure drop volumetric flow meter as $Q_{\rm ON}$.
- i) Set the DEMC voltage to 0 V and let the CPCs zero. Record the differential pressure across the flow restriction as $dP_{\rm OFF}$. Then record the inlet flow rate of the test CPC using the low pressure drop volumetric flow meter as $Q_{\rm OFF}$.
- j) Set the DEMC voltage to the value in step f).
- k) Repeat steps h) through j) at least two more times.
- l) Calculate the arithmetic mean values of the two differential pressures and the two CPC inlet flow rates as $dP_{\rm ON}$, $dP_{\rm OFF}$, $Q_{\rm ON}$, and $Q_{\rm OFF}$.
- m) Calculate the ratios $r_{\rm dP} = dP_{\rm ON}/dP_{\rm OFF}$ and $r_{\rm Q} = Q_{\rm ON}/Q_{\rm OFF}$.

For the diluter to be acceptable, the ratio r_Q shall equal unity within the expanded uncertainty of the volumetric flow meter at 95 % confidence. Also the ratio $r_{\rm dP}$ shall be within 0,98 < $r_{\rm dP}$ < 1,02.

H.3 Proportionality calibration

The proportionality calibration requires two steps: a direct comparison between the test CPC and the dummy CPC, and the measurement of dilution factor over a range of aerosol concentrations.

H.3.1 Experimental setup

The user shall define the concentration test points that the calibration will be performed at, using a nominal dilution factor of 10:1. A minimum of 7 test points shall be used in the desired concentration range, and these points shall be spaced at approximately equal intervals on a logarithmic scale. For example, when calibrating over the range of 10 cm^{-3} to 10^4 cm^{-3} , use the data points of (10, 30, 100, 300, 1000, 300, 3000, and <math>10000) cm⁻³.

The test points shall be arranged in a random sequence using a pseudo-random number generator.

The dilution ratio of the diluter shall be between 9:1 and 11:1 and nominally 10:1.

The experimental setup for the proportionality test is shown in Figure H.3.

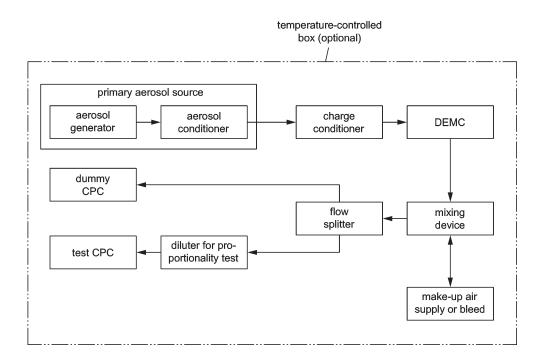


Figure H.3 — Schematic of the calibration setup for the proportionality test

H.3.2 CPC zero

A HEPA filter shall be connected to the inlet of the CPC while sampling room air and allowed to zero. The number of counts shall be measured over 5 min and used together with the sample time and flow rate to calculate a concentration. The calculated concentration shall be at least 100 times lower than the lowest concentration chosen in <u>H.3.1</u>.

H.3.3 CPC correlation

A direct comparison between the dummy CPC and the test CPC is performed to account for any differences between the two detectors. Set up the experiment according to Figure H.3, but with the diluter for proportionality test removed so that a parallel measurement of the same aerosol can be performed. Turn on the instruments and let them warm up for 1 h. Set the DEMC flows and voltage to values corresponding to the particle diameter of interest. For each concentration test point specified in H.3.1, measure the arithmetic mean aerosol concentration with both CPCs using an appropriate sample time. Sample times should be longer at lower concentrations to reduce measurement uncertainty. Calculate the correction factor k as shown in Formula (H.1):

$$k(C_{\text{dummy}}) = C_{\text{test}}/C_{\text{dummy}}$$
 (H.1)

The factor *k* correlates the dummy CPC to the test CPC.

H.3.4 Proportionality measurements

Follow the procedure for the proportionality measurement listed below.

- a) Set up the system according to Figure H.3.
- b) Set the DEMC voltage and sheath flow rate to provide the desired particle size.
- c) Adjust the aerosol concentration measured by the dummy CPC to a test point where the coincidence correction is about 1 %.

- d) Adjust the diluter so that the test CPC reads a concentration that is approximately 10 times lower than the dummy CPC.
- e) Record simultaneous measurements of the CPC concentrations for a total of 10 measurements using an appropriate sample time.

NOTE Sample times should be longer at lower concentrations to increase the number of particles counted in the time interval to reduce measurement uncertainty.

Record any differential pressure or flow rate measurements of the diluter flows.

- f) Adjust the aerosol concentration to the first (or next) value of the random sequence of test points, as measured by the dummy CPC.
- g) Record simultaneous measurements of the CPC concentrations, taking at least 10 measurements using an appropriate sample time. Record any differential pressure or flow rate measurements of the diluter flows.
- h) Repeat steps f) and g) until half of the test points of the random sequence are measured.
- i) Adjust the aerosol concentration to the concentration used to set up the diluter in step c). Record simultaneous measurements of the CPC concentrations, taking at least 10 measurements using an appropriate sample time. Record any differential pressure or flow rate measurements of the diluter flows.
- j) Repeat steps f) and g) for the remaining test points of the random sequence.
- k) Repeat step i).

For each test point, calculate the arithmetic mean concentration measured by each CPC as shown in Formula (H.2):

$$C = \frac{1}{n} \sum_{i=1}^{n} C_i$$
 (H.2)

Calculate the dilution factor at each test point as shown in Formula (H.3):

$$R_{\rm D} = \frac{C_{\rm test}}{k(C_{\rm dummy}) \times C_{\rm dummy}} \tag{H.3}$$

H.4 Data analysis and test acceptance

This section describes two statistical tests that are performed on the calibration data. The first tests the CPC response for proportionality, and the second tests the diluter for drift over time. Both tests shall pass for the calibration to be considered valid.

H.4.1 Dependence of dilution ratio with concentration

To determine if the dilution ratio changes with concentration, the dilution ratio can be plotted as a function of concentration measured by the dummy CPC. If the dilution ratio is independent of concentration, then the slope of a line fit to the data set will be zero.

To perform a quantifiable test, the slope of the data set shall be calculated using linear least square regression and a t-test shall be performed to determine if the slope is significantly different from zero. Each test point in H.3.4 has values of C_{test} , C_{dummy} , k, R_{D} , and measurement number m. Calculate the

slope of the line \hat{b} , the intercept \hat{a} , and the standard error of the slope $SE_{\hat{b}}$, where n is the number of measurements, as:

$$\hat{b} = \frac{n\sum_{i=1}^{n} R_{D,i} - \left(\sum_{i=1}^{n} C_{dummy,i}\right) \left(\sum_{i=1}^{n} R_{D,i}\right)}{n\sum_{i=1}^{n} C_{dummy,i}^{2} - \left(\sum_{i=1}^{n} C_{dummy,i}\right)^{2}}$$
(H.4)

$$\hat{a} = \frac{1}{n} \left(\sum_{i=1}^{n} R_{D,i} - \hat{b} \sum_{i=1}^{n} C_{\text{dummy},i} \right)$$
(H.5)

$$SE_{\hat{b}} = \left\{ \frac{\sum_{i=1}^{n} \left[R_{D,i} - \left(\hat{a} + \hat{b} C_{\text{dummy},i} \right) \right]^{2}}{\left(n - 2 \right) \sum_{i=1}^{n} \left(C_{\text{dummy},i} - \overline{C_{\text{dummy}}} \right)^{2}} \right\}^{\frac{1}{2}}$$
(H.6)

Calculate the test statistic Z, with the hypothesized slope $b_0 = 0$, as

$$Z = \frac{\hat{b} - b_0}{SE_{\hat{b}}} \tag{H.7}$$

From the t-distribution table record the statistic for 95 % confidence using n-2 degrees of freedom as $T_{95\%}$. If $Z < T_{95\%}$, then the data suggests that the slope is not significantly different from zero and the analysis can continue. Otherwise the slope is significantly different than zero and the CPC response is not proportional. Check the coincidence correction, check the diluter flows and differential pressures for drift, and/or perform the calibration using a lower maximum concentration.

H.4.2 Dependence of dilution ratio with time

To determine if the dilution ratio is drifting over time, the dilution ratio can be plotted as a function of measurement number, which is roughly proportional to time. If the dilution ratio is not drifting with time then the slope of a line fit to the data set will be zero.

To perform a quantifiable test, the slope of the data set shall be calculated using linear least square regression and a t-test shall be performed to determine if the slope is significantly different from zero. Each test point in H.3.4 has values of C_{test} , C_{dummy} , k, R_{D} , and measurement number m. Calculate the slope of the line \hat{b} , the intercept \hat{a} , and the standard error of the slope $\text{SE}_{\hat{b}}$, where n is the number of measurements, as:

$$\hat{b} = \frac{n \sum_{i=1}^{n} m_i \cdot R_{D,i} - \left(\sum_{i=1}^{n} m_i\right) \left(\sum_{i=1}^{n} R_{D,i}\right)}{n \sum_{i=1}^{n} m_i^2 - \left(\sum_{i=1}^{n} m_i\right)^2}$$
(H.8)

$$\hat{a} = \frac{1}{n} \left(\sum_{i=1}^{n} R_{D,i} - \hat{b} \sum_{i=1}^{n} m_i \right)$$
(H.9)

$$SE_{\hat{b}} = \left\{ \frac{\sum_{i=1}^{n} \left[R_{D,i} - \left(\hat{a} + \hat{b} m_{i} \right) \right]^{2}}{\left(n - 2 \right) \sum_{i=1}^{n} \left(m_{i} - \overline{m} \right)^{2}} \right\}$$
(H.10)

Calculate the test statistic Z, with the hypothesized slope $b_0 = 0$, as

$$Z = \frac{\hat{b} - b_0}{\text{SE}_{\hat{b}}} \tag{H.11}$$

From the t-distribution table record the statistic for 95 % confidence using n-2 degrees of freedom as $T_{95\%}$. If $Z < T_{95\%}$, then the data suggests that the slope is not significantly different from zero and the analysis can continue. Otherwise the slope is significantly different than zero and the dilution ratio is drifting with time; check for leaks, flow stability, or variations in system temperature and pressure and perform the calibration again.

H.5 Uncertainty analysis

If the calibration data passes the two statistical tests in <u>H.4</u>, the uncertainty of the CPC proportional response can be calculated as shown below.

a) Calculate the arithmetic mean dilution factor as

$$R_{\rm D} = \frac{1}{n} \sum_{i=1}^{n} R_{{\rm D},i} \tag{H.12}$$

b) Calculate the relative standard uncertainty of the CPC proportional response as

$$u_{\rm r}(R_{\rm D}) = \frac{100}{R_{\rm D}} \cdot \sum_{i=1}^{n} \sqrt{\frac{\left(R_{\rm D} - R_{{\rm D},i}\right)^2}{n(n-1)}}$$
(H.13)

This uncertainty can be combined in quadrature with the uncertainties from an absolute calibration using a reference instrument.

Annex I (informative)

Example of a detection efficiency measurement

I.1 General

This Annex provides an example of a test CPC detection efficiency measurement using an FCAE as reference instrument. The analysis is for one particle size (70 nm) and one particle concentration (approximately 7 000 cm^{-3}). The procedure described in <u>Clause 6</u> will be followed. The final certificate of calibration of the specific example can be found in <u>C.2</u>.

The detection efficiency of the FCAE, η_{FCAE} , and its standard uncertainty, $u(\eta_{FCAE})$, at 70 nm and at 7 000 cm⁻³ are given in the FCAE calibration certificate as 0,997 and 0,006, respectively. The flow rate of the FCAE at time of calibration was 0,99 l/min. The test CPC used in this study has a critical orifice for flow control and a nominal inlet volumetric flow rate of 1,00 l/min used in the CPC's internal concentration calculation. The measurement of the fraction of multiply charged particles in the calibration aerosol will be performed by doubling and tripling the voltage of the DEMC.

I.2 Overview of the calibration setup and procedure (see 6.1)

A schematic of the setup can be seen in Figure I.1. The whole setup was in a temperature-controlled room at 23 °C. The ambient pressure was 98 kPa. The aerosol generator was a diffusion flame based soot generator. The aerosol conditioner was a hot diluter at 150 °C followed by an evaporation tube at 350 °C and a diluter at ambient temperature to reduce the temperature of the sample at ambient temperature. The relative humidity of the diluted sample was approximately 5 % (<40 %).

The charge conditioner was a Kr-85 bipolar charger (neutralizer). A DEMC was used to select monodisperse particles. The make-up air flow was adjusted by a throttle valve. The humidity of the make-up air was approximately 20 % (<40 %). A mixing orifice was used as a mixing device and the FCAE and the test CPC were sampling from a 2-way splitter with silicone conductive hoses of equal tube lengths with almost equal flow rates (\sim 1 l/min).

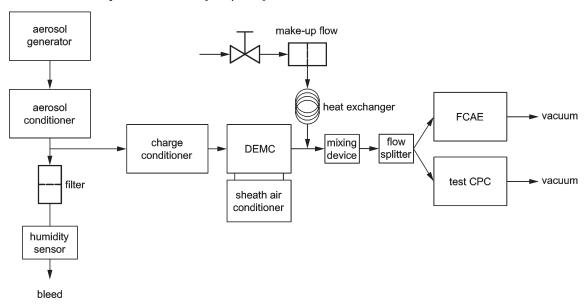


Figure I.1 — Experimental setup of the hypothetical example

I.3 Preparation (see <u>6.2</u>)

I.3.1 General preparation

Initially all instruments were checked separately (i.e. not connected as shown in Figure I.1).

I.3.2 Primary aerosol

The aerosol generator and conditioner were turned on and left to stabilize for 1 h. The size distribution of the aerosol that would be fed to the DEMC was measured with a DMAS. The median of the size distribution was 49 nm and the geometric standard deviation 1,46.

I.3.3 Other equipment

The pressure sensor, the temperature sensors, the flow meter and the humidity sensor were turned on. They were checked according to manufacturers' specifications.

I.3.4 DEMC

The DEMC was checked according to ISO 15900.

I.3.5 FCAE

The FCAE was turned on and left to stabilize for 1 h. A HEPA filter was placed at the inlet of the FCAE and a zero was conducted.

a) Zero check

The recorded current levels after the zero of the FCAE can be seen in Figure I.2. In the same figure the internally zeroed arithmetic mean and the standard deviation are shown. The arithmetic mean has an absolute values of 0.133 fC/s (less than the maximum 1 fC/s required) and the standard deviation is 0.36 fC/s (less than the 0.5 fC/s required). So this measurement is considered valid and the calibration procedure can proceed.

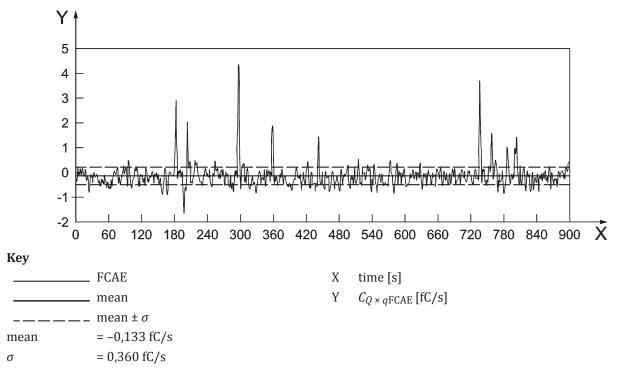


Figure I.2 — Zero levels of the FCAE

b) FCAE overall leak test

The particle counts from the HEPA filtered air sampled by the CPC $N_{\rm HEPA}$, the ambient counts measured by the CPC $N_{\rm ambient}$, and the particle counts resulting from room air passing through the FCAE $N_{\rm FCAE}$, are, 3, 1 056 780, and 5 respectively. The ratio $R_{\rm FCAE}$ is 1,9E-6, much lower than the threshold 1E-4, so the calibration procedure can proceed.

c) Flow rate measurement

The FCAE inlet volumetric flow rate measured with a calibrated flow meter, $q_{\text{FCAE,cal,amb},i}$, along with the indicated reading from the FCAE flow meter, $q_{\text{FCAE,amb},i}$, are measured five times over 15 min. The data are shown in Table I.1.

Arithmetic Test i = 1i = 2i = 3i = 4i = 5mean 0.99 1.00 $q_{\text{FCAE.amb.}i}$ [l/min] 1.00 1.01 1.00 1.00 1,00 1,01 1,02 1,01 1,01 1,01 q_{FCAE,cal,amb,i} [l/min]

Table I.1 — FCAE volumetric flow rate measurements

The arithmetic mean value of $q_{FCAE,cal,amb}$ is 1,01 l/min. The CV of $q_{FCAE,cal,amb}$ is 0,007 1 corresponding to 0,70 % (less than the maximum 2 % required), and there is no decreasing or increasing trend in the values. The difference of the flow rates is 1 %, which is within the specifications of the FCAE manufacturer (3,5 %). So this measurement is considered valid and the calibration procedure can proceed.

I.3.6 Test CPC

The test CPC was turned on, filled with the working fluid and left 1 h to stabilize with a HEPA filter at the inlet.

a) Zero count check

The zero check of the CPC was easily passed. The arithmetic mean concentration of 5 min measurement (1-s reading and 1-s averaging) with a HEPA filter at the inlet and their standard deviation was close to 0 cm^{-3} .

b) High response check

Measurement of ambient air showed approximately 3 $500~\text{cm}^{-3}$, thus it can be assumed that the instrument works properly.

c) Flow rate measurement

The test CPC has a critical orifice and no internal flow measurement. The nominal value of the flow rate is 1 l/min. The CPC uses the nominal flow rate for the internal particle number concentration calculations. The CPC inlet volumetric flow rate is measured with a calibrated flow meter, $q_{\text{FCAE,cal,amb},i}$, five times over 15 min. The data are shown in Table I.2.

Arithmetic i = 3i = 4Test i = 1i = 2i = 5mean q_{CPC,amb,i} [l/min] 1,00 1,00 1,00 1,00 1,00 1,00 1,01 1,02 1,01 q_{CPC,cal,amb,i} [l/min) 1,00 1,01 1,01

Table I.2 — Test CPC volumetric flow rate measurements

The arithmetic mean measured inlet flow rate is 1,01 l/min, and the CV of $q_{\rm CPC,cal,amb}$ is 0,007 0 corresponding to 0,70 %. Also, there is no decreasing or increasing trend in the values, Their arithmetic mean was 1 % more than the nominal value of the CPC. The manufacturer specifies flow accuracy of 5 %, so this measurement is considered valid and the calibration procedure can proceed. It is written

down that the measured CPC inlet flow rate is 1 % higher than the nominal value used by the CPC for the internal particle number concentration calculation.

I.3.7 Check of the complete setup

The instruments were connected as shown in Figure I.1.

a) DEMC flows

The DEMC flows were adjusted: 10 l/min sheath flow and 1 l/min sample flow, thus ratio 10:1 (>7:1 required) ensuring a narrow monodisperse distribution downstream of the DEMC. The voltage is set to 0 (off).

b) FCAE flow rate measurement

The calibrated flow meter was inserted in the setup between the splitter and the inlet of the FCAE. The volumetric flow measured was $q_{\text{FCAE,cal}} = 0.95$ l/min. This value was written down for the calibration certificate. This value was compared with the indicated (q_{FCAE}) 0.98 l/min. The difference (-3,1 %) is within the manufacturer's specifications (3,5 %). The difference compared to the value in the FCAE certificate is -4 %. This is less than the deviation allowed by Formula (3) (7,6 %).

c) Test CPC flow rate measurement

The calibrated flow meter was inserted in the setup between the splitter and the inlet of the test CPC. The volumetric flow measured was 0,99 l/min. This value was written down for the calibration certificate. This value differs by 1 % from the nominal value (1 l/min) and is < 5 %, which is allowed by the manufacturer.

d) Zero levels

The DEMC voltage was set to 0 (or off) and the zero levels of the FCAE and test CPC were recorded for 2 min (see Figure I.3, 40 - 160 s). The 30-s arithmetic means were calculated. The values of the 30-s arithmetic means of the FCAE current were between -0.37 and +0.24 (thus within the \pm 1 fC/s) and the standard deviation of the arithmetic means 0.13 fC/s (thus below the maximum permitted of 0.5 fC/s). Similarly the test CPC arithmetic means were 0; much below the maximum permitted level of 1 cm⁻³.

e) Minimum FCAE level

The sum of the arithmetic mean of the last 30 s (0,23 fC/s) and 3 times the standard deviation of the 2 min measurement (0,13 fC/s), i.e. 0,62 fC/s, was compared with the lowest value of $C_{Q \times qFCAE}$ in the calibration certificate of the FCAE, which was 2,67 fC/s in this example. Since the latter was greater, the minimum permitted current that the FCAE can measure during the calibration procedure was 2,67 fC/s.

I.4 Calibration procedure of detection efficiency (see 6.3)

I.4.1 General

The following procedure describes the measurement of the detection efficiency of the test CPC at one particle size and number concentration.

I.4.2 DEMC diameter adjustment

The DEMC was adjusted to 70 nm. This size is big enough so that the test CPC has reached its maximum detection efficiency. Note that it is also bigger than the median of the generated size distribution entering the DEMC in order to minimize the effect of multiply charged particles.

I.4.3 Primary aerosol adjustment

The aerosol conditioner was adjusted to have a number concentration of approximately 7 000 cm⁻³ (as measured by the test CPC). The following were fulfilled:

a) Minimum level

The FCAE measured approximately 19 fC/s, which is higher than the minimum permitted level of 2,67 fC/s

b) Maximum level

The FCAE measured approximately 19 fC/s, which is lower than the maximum permitted level specified in the FCAE certificate of 1 500 fC/s.

The concentration was low enough to reach charge equilibrium in the charge conditioner. This was checked according to Annex K (see below).

c) Fraction of multiply charged particles

The multiply charged particles fraction was determined according to the method described in <u>Annex D</u>, i.e. by doubling the voltage (see <u>Figure I.3</u>).

See Table I.3 below.

Table I.3 — Measurements for determination of the fractions of multiply charged particles

	U	2 <i>U</i>	3 <i>U</i>
Voltage [V]	-767	-1 533	-2 300
Size [nm]	70	103	130
FCAE [fC/s]	19,0	5,8	1,3
Test CPC [cm-3]	6 871	2 229	447

It is assumed that there are only particles of up to three charges in $C_N(U)$ and that there are only singly charged particles in $C_N(3U)$ and $C_N(3U)$. The concentrations measured at U, 2U, and 3U are calculated to be

$$C_N(U) = (C_Q \times q_{FCAE})/(e \cdot q_{FCAE}) = (19.0E - 15)/[(1.602E - 19)(0.95 \cdot 1000 / 60)] = 7490.6 \text{ cm}^{-3}$$
 (I.1)

$$C_N(2U) = (C_Q \times q_{FCAE}) / (e \cdot q_{FCAE}) = (5.8E - 15) / [(1.602E - 19)(0.95 \cdot 1.000 / 60)] = 2.286.6 \text{ cm}^{-3} \text{ (I.2)}$$

$$C_N(3U) = (C_Q \times q_{\text{FCAE}}) / (e \cdot q_{\text{FCAE}}) = (1.3E - 15) / [(1.602E - 19)(0.95 \cdot 1.000 / 60)] = 512.5 \text{ cm}^{-3}$$
 (I.3)

The charging fractions for each particle diameter are used to calculate the concentrations of multiply charged particles passing through the DEMC, according to Formula (D.36) to Formula (D.38)

$$C_N(d_2(U)) = \frac{C_N(2U)}{\eta_{\text{FCAE}}} \cdot \frac{f_2(d_1(2U))}{f_1(d_1(2U))} = \frac{2286,6 \text{ cm}^{-3}}{0,997} \cdot \frac{0,0333}{0,2146} = 355,9 \text{ cm}^{-3}$$
(I.4)

$$C_N(d_3(U)) = \frac{C_N(3U)}{\eta_{\text{FCAE}}} \cdot \frac{f_3(d_1(3U))}{f_1(d_1(3U))} = \frac{512.5 \,\text{cm}^{-3}}{0.997} \cdot \frac{0.004 \,\text{8}}{0.216 \,\text{8}} = 11.38 \,\text{cm}^{-3}$$
(I.5)

$$C_N(d_1(U)) = \frac{C_N(U)}{\eta_{\text{FCAE}}} - 2 \cdot C_N(d_2(U)) - 3 \cdot C_N(d_3(U))$$

$$= \frac{7 \cdot 490,6 \,\text{cm}^{-3}}{0,997} - 2 \cdot \left(355,9 \,\text{cm}^{-3}\right) - 3 \cdot \left(11,38 \,\text{cm}^{-3}\right) = 6767,2 \,\text{cm}^{-3}$$
(I.6)

Formulae (D.25) and (D.26) are used to calculate the fractions of multiple charged particles:

$$C_N = \sum_{p=1}^{3} C_N(d_p(U)) = (6767, 2+355, 9+11, 38) \text{ cm}^{-3} = 7134, 5 \text{ cm}^{-3}$$
(I.7)

$$\phi_1 = C_N(d_1(U))/C_N = 6767,3/7134,5 = 0,9485$$
 (I.8)

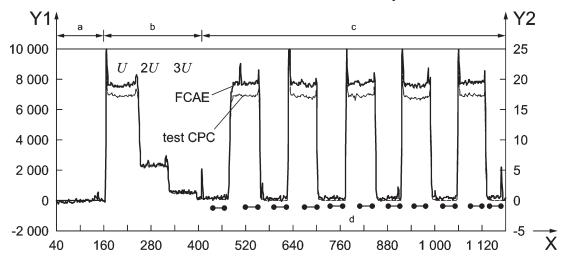
$$\phi_2 = C_N(d_2(U))/C_N = 355,9/7 \, 134,5 = 0,049 \, 9$$
 (I.9)

$$\phi_3 = C_N(d_3(U))/C_N = 11,38/7 \ 134,5 = 0,001 \ 6$$
 (I.10)

The total fraction of multiple charged particles is calculated by Formula (7):

$$\Phi = \sum_{p \ge 2} \phi_p = \phi_2 + \phi_3 = 0.049 \ 9 + 0.001 \ 6 = 0.051 \ 5 \tag{I.11}$$

Based on the concentrations measured at the different voltages the multiply charged particles fraction was estimated to be 5,15 %, which is lower than the 10 % maximum permitted.



Key

C

a zero measurement

b multiple charge measurement Y1 $C_{N, CPC}$ [cm⁻³]

detection efficiency measurement Y2 $C_{Q \times qFCAE}$ [fC/s]

d time intervals for data acquisition during efficiency measurement

Figure I.3 — Example of calibration procedure of one detection efficiency

time [s]

Although not shown in the figure, the concentration was reduced to half and the determination of the fraction of multiply charged particles was repeated. The fraction was measured at 5,5 %, which is similar to the previous value. So it can be assumed that aerosol reaches charge equilibrium with the original concentration.

I.4.4 Splitter performance

According to <u>6.3.4</u>, refer to <u>Annex G</u> to perform the splitter bias measurement. Following Formula (G.1) in <u>G.2.1</u>, record 5 pairs of concentration readings from the FCAE and test CPC when the instruments are set up according to configuration 1 in <u>Figure G.1</u>. The data recording method given in <u>Annex L</u> is followed to record the FCAE current readings which will be converted to concentrations. The current and concentration readings are shown in <u>Table I.4</u>.

Table I.4 — Splitter bias FCAE current and CPC concentration readings from configuration 1a

	zero	<i>i</i> = 1	zero	<i>i</i> = 2	zero	<i>i</i> = 3	zero	i = 4	zero	<i>i</i> = 5	zero
FCAE [fC/s]	0,4	19,5	0,5	19,1	0,5	19,5	0,6	19,8	0,4	19,5	0,6
CPC [cm ⁻³]	0	6 970	0	6 980	0	6 980	0	6 960	0	6 970	0

Flip the splitter and mixing device or exchange the instrument positions according to <u>G.2.2</u> and record 5 pairs of concentration readings from the FCAE and test CPC when the instruments are set up according to configuration 2 in <u>Figure G.2</u>.

Table I.5 — Splitter bias FCAE current and CPC concentration readings from configuration 2

	zero	<i>i</i> = 1	zero	i = 2	zero	i = 3	zero	i = 4	zero	<i>i</i> = 5	zero
FCAE [fC/s]	0,5	19,6	0,6	19,6	0,4	19,6	0,5	19,6	0,4	19,6	0,6
CPC [cm ⁻³]	0	6 970	0	6 980	0	6 970	0	6 960	0	6 970	0

Flip the splitter and mixing device or exchange the instrument positions according to <u>G.2.3</u> and record 5 pairs of concentration readings from the FCAE and test CPC when the instruments are set up according to configuration 1 in <u>Figure G.1</u>.

Table I.6 — Splitter bias FCAE current and CPC concentration readings from configuration 1b

	zero	<i>i</i> = 1	zero	i = 2	zero	i = 3	zero	i = 4	zero	<i>i</i> = 5	zero
FCAE [fC/s]	0,2	19,2	0,4	19,6	0,7	19,4	0,6	19,7	0,5	19,3	0,6
CPC [cm-3]	0	7 001	0	6 965	0	6 970	0	6 940	0	6 975	0

Assume that the FCAE flow rate is exactly 1 l/min and apply no flow correction. Also assume that all of the particles carry a single charge. Calculate the FCAE concentration by Formula (8) in 6.3.5, and calculate the arithmetic mean of the zero levels before and after test i. Then calculate the ratios $r_{1,i}$ and $r_{2,i}$ using Formulae (G.4) and (G.7). The results are in Table I.7.

Table I.7 — Concentrations and ratios from the splitter bias test

i	1	2	3	4	5
$C_{N,\mathrm{ref},1a,i}$	7 135	6 966	7 097	7 228	7 116
$C_{N,\mathrm{ref},1a,i}$	6 970	6 980	6 980	6 960	6 970
$r_{1a,i}$	0,976 9	1,002 0	0,983 5	0,962 9	0,979 5
$C_{N,\text{ref},2,i}$	7 135	7 154	7 172	7 172	7 154
$C_{N, \mathrm{CPC}, 1a, i}$	6 970	6 980	6 970	6 960	6 970
$r_{2,i}$	0,976 9	0,975 7	0,971 8	0,970 4	0,974 3
$C_{N,\text{ref,1b},i}$	7 079	7 135	7 022	7 172	7 022
$C_{N,\text{ref,1b},i}$	7 001	6 965	6 970	6 940	6 975
$r_{1\mathrm{b},i}$	0,989 0	0,976 2	0,992 5	0,967 6	0,993 2

The arithmetic mean ratios $\overline{r_1}$ and $\overline{r_2}$ are calculated from Formula (G.5) and Formula (G.7) using the data in <u>Table I.7</u>. The splitter bias correction factor is calculated by Formula (G.8), and found to be β = 0,995 7. This value is greater than 0,95 and less than 1,05 so the calibration procedure can proceed. The uncertainty is found by the standard deviation of the measurements (0,012 1 and 0,002 7) and is 0,008 8.

I.4.5 Test CPC detection efficiency

<u>Figure I.3</u> shows the 5 repetitions of high and zero concentrations for both the FCAE and the test CPC. The arithmetic means of the last 30 s and their standard deviations for each measurement are given in <u>Table I.8</u>.

i = 1i = 2i = 3i = 4i = 5zero zero zero zero zero zero **FCAE** 0.3 19.3 0.3 0.3 19.4 19.5 19.0 0.4 18.8 0.4 0.5 [fC/s] Std Dev 0,2 0,2 0,2 0,3 0,2 0,3 0.2 0,2 0,2 0,1 0,2 [fC/s] CV [%] 1,04 1,58 1,55 1,06 0.51 CPC 0,1 6 887,4 0,1 6807,4 0,1 6 914,7 0,3 6712,2 6 908,2 0,1 1,6 [cm⁻³] Std Dev 31.6 64.8 59.9 42 34.2 [cm⁻³] CV [%] 0,46 0,95 0,87 0,63 0,50

Table I.8 — 30-s arithmetic mean FCAE current and test CPC concentration readings

In all cases, the requirements were fulfilled (i.e. for the zero measurements the arithmetic mean and standard deviation were <1 and <0,5 fC/s for the FCAE, respectively, and the mean concentration was <1 cm $^{-3}$ for the test CPC). For the high concentration measurement the standard deviation was <0,5 fC/s for the FCAE and the CV < 3 % for both the FCAE and the test CPC.

This subclause uses formulae from <u>Clause 6</u> and <u>Annex D</u> to compute the CPC detection efficiency using the data recorded during the measurement process.

Calculate the FCAE concentration by Formula (8) in <u>6.3.5</u>, and calculate $C_{Q,0,i} \times q_{\text{FCAE}}$ as the arithmetic mean of the zero current before and after test i in <u>Table I.8</u>. An example calculation for test i = 1 is shown in Formula (I.12) and Formula (I.13). The results are in <u>Table I.9</u>.

$$C_{Q,0,1} \times q_{\text{FCAE}} = (0,3+0,3)/2 = 0,3$$
 (I.12)

$$C_{N,\text{FCAE},1} = \frac{\left| C_{Q,1} \times q_{\text{FCAE}} - C_{Q,0,1} \times q_{\text{FCAE}} \right|}{e \cdot q_{\text{FCAE}}} = \frac{\left| 19,0 - 0,3 \right| \cdot 10^{-15} \text{ C/s}}{\left(1,602 \cdot 10^{-19} \text{ C} \right) \cdot \left(0,95 \text{ l/min} \cdot \frac{1000 \text{ cm}^3 \text{ min}}{\text{s}} \right)} = 7 \text{ 490,6 cm}^{-3} \quad \text{(I.13)}$$

Table I.9 — FCAE net current and concentration and CPC concentration readings

i	1	2	3	4	5
FCAE [fC/s]	19,00	18,70	19,05	18,40	19,05
FCAE [cm ⁻³]	7 490,6	7 372,4	7 510,3	7 254,1	7 510,3
CPC [cm ⁻³]	6 887,4	6 807,4	6 914,7	6 712,2	6 908,2
$\eta_{\mathrm{CPC},i}$	0,961 2	0,965 3	0,962 5	0,967 3	0,961 6

The test CPC detection efficiency is calculated for each measurement using Formula (D.18), with the test CPC concentrations from <u>Table I.8</u>, the FCAE concentrations corrected for flow and splitter bias, the

FCAE detection efficiency from the calibration certificate, and the fraction of particles carrying a single, double, and triple charges. An example is shown below for the first measurement:

$$\eta_{\text{CPC,1}} = \frac{C_{N,\text{CPC,1}}}{C_{N,\text{FCAE,1}}} \eta_{\text{FCAE}} \cdot \beta \sum_{p \ge 1} \phi_p \cdot p$$

$$= \frac{6887.4 \text{ cm}^{-3}}{7490.6 \text{ cm}^{-3}} \cdot (0.997) \cdot (0.9957) \cdot \left[0.9485 + 2(0.0499) + 3(0.0016)\right] = 0.9612$$
(I.14)

The arithmetic mean detection efficiency and the standard deviation are, respectively, 0,963 5 and 0,002 6.

I.5 Uncertainty

Repeatability

The calculation of uncertainty in the detection efficiency is summarized in <u>Table I.10</u> below:

Value Component Symbol Obtained from % the FCAE calibration certificate — the relative standard (non-expanded) uncertainty value FCAE detection efficiency $u_r(FCAE)$ 0,90 applying to the current relevant to the test CPC calibration Based on 3 measurements in one week Multiple charge correction $u_{\rm r}({\rm MCC})$ 2,4 0,88 Splitter bias correction factor $u_{\rm r}(\beta)$ I.4.4 FCAE flow rate deviation 2,33 I.3.7 b) $u_{\rm r}(q_{\rm FCAE})$

Table I.10 — Relative uncertainty components

In this case the combined relative standard uncertainty is given in Formula (I.15):

 $u_{\rm r}(\eta_{\rm rep})$

$$u_{c,r}(\eta) = \sqrt{0.90^2 + 2.4^2 + 0.88^2 + 2.33^2 + 0.27^2}$$

= 3.59 % (I.15)

0,27

I.4.5

The relative expanded uncertainty for a coverage factor k = 2 is given in Formula (I.17):

$$U_{\rm r}(\eta) = 2.3,59 \% = 7,18 \%$$
 (I.16)

I.6 An example protocol for calibration with an FCAE

6.2 Preparation

6.2.2 Aerosol generator and conditioner (size distribution)

Median [nm]	49	Geo Std Dev	1,46
RH aerosol	5 %		

6.2.3 Other equipment (e.g. mass flow meters, etc.)

6.2.4 DEMC Checked according to ISO 15900

6.2.5 FCAE

a) Zero check

The FCAE was zeroed internally

Abs. zero mean <1,0 fC/s, Std Dev <2,5 fC/s (from 1 s averages over 15 min)

	Reading	Limit	Result
Largest abs. FCAE zero mean [fC/s]	-0,133	±1,0	ok
Largest FCAE Std Dev [fC/s]	0,36	2,5	ok

b) FCAE overall leak test

N _{HEPA}	3
N _{ambient}	1 056 780
N _{FCAE}	5
N _{leak}	2
R _{FCAE}	1,9E -06
Limit	1,0E -04
Result	ok

$q_{ m FCAE,cert}$	0,99
r _{q,FCAE}	3,5 %
$u(q_{\text{FCAE,cert}})$	2 %
$\eta_{ ext{FCAE}}$	0,997
<i>u</i> (η) [%]	0,90
FCAE max current [fC/s]	1 500

from manufacturer in certificate

c) Flow rate measurement

Flow measurement and stability < 2 % (from 5 measurements in 15 min).

Indicated and measured differences within manufacturer's specification.

All values below in units of l/min unless otherwise noted.

Measurement	FCAE indicated flow rate	FCAE measured flow rate
1	0,99	1,00
2	1,00	1,01
3	1,01	1,02
4	1,00	1,01
5	1,00	1,01
Mean	1,00	1,01
Std Dev	0,007 1	0,007 1
CV [%]		0,70
CV limit		< 2 %
Result		ok

Difference from indicated 1,0 %

Difference from certificate 2,0 %

Limit from manufacturer

3,5 % ok

Limit from flow meter uncertainties
7,6 % ok a

6.2.6 Test CPC

a) Zero count check

Zero mean <0,1 cm⁻³ (from 1 s averages of 5 min)

	Reading	Limit	Result
Largest CPC zero reading [cm-3]	0	0,1	ok

b) High response check

	Reading	Limit	Result
CPC [cm-3]	3 500	> 500	ok

 $^{^{\}rm a}$ Assuming 2,5 % uncertainty of the flow meter that was used during this calibration.

c) Flow rate measurement

Flow measurement and stability <2 % (from 5 measurements in 15 min).

Indicated and measured differences within manufacturer's specification.

All values below in units of l/min unless otherwise noted.

Measurement number	CPC indicated flow rate	CPC measured flow rate
1	1,00	1,00
2	1,00	1,01
3	1,00	1,02
4	1,00	1,01
5	1,00	1,01
Mean	1,00	1,01
Std Dev	0,00	0,007 1
CV [%]		0,70
CV limit		< 2 %
Result		ok
Difference from indica	ated	Limit from manufact

Difference from indicated
1 %

Limit from manufacturer <5 % ok

6.2.7 Connect the instruments and the aerosol generator/conditioner to the DEMC

a) DEMC flow rate

DEMC (voltage off), sheath to sample > 7:1	Reported value	10:1	Result:	ok
---	----------------	------	---------	----

b) FCAE flow measurement [l/min]

1	0,98	0,95		
Difference from indica	ated	Limit from manufac	cturer	
-3,1 %		3,5 %	ok	
Difference from certif	icate	Limit from flow me	ter uncertainties	
-4,0 %		7,6 %	ok ^a	2,33 %
2 4 : 0 = 0/		1 .	1 1 1 11	

^a Assuming 2,5 % uncertainty of the flow meter that was used during this calibration.

c) Test CPC flow rate measurement [l/min]

Measurement	FCAE indicated flow rate	FCAE measured flow rate			
1	1,00	0,99			
Difference from indicated					

Difference from indicated -1,0 %

Limit from manufacturer
5,0 % ok

d) Zero levels

FCAE zero level < 1 fC/s, Std Dev < 0,5 fC/s (from 30 s averages of 2 min)

	Reading	Limit	Result	
Largest FCAE zero current	-0,37	±1	ok	optional
Largest FCAE Std Dev	0,13	0,5	ok	

Test CPC zero mean < 1 cm⁻³, Std Dev < 0,5 cm⁻³ (from 30 s averages of 2 min)

	Reading	Limit	Result
Largest CPC zero current	0	1	ok
Largest CPC Std Dev	0	0,5	ok

e) Determine the minimum level of FCAE

Last 30 s zero current [fC/s]	0,23
Std Dev of 2 min measurement [fC/s]	0,13
Sum of the above [fC/s]	0,62
Lowest $C_{Q \times qFCAE}$ in certificate [fC/s]	2,67
Minimum level [fC/s]	2,67

6.3 Detection efficiencies

6.3.2 DEMC diameter adjustment (70 nm)

6.3.3 Primary aerosol adjustment

The concentration is within the capability of the charge conditioner.

		,	,
Voltage	1 <i>U</i>	2 <i>U</i>	3 <i>U</i>
	-767	-1 533	-2 300
Size [nm]	70	103	130
FCAE [fC/s]	19	5,8	1,3
C _{CPC} [cm ⁻³]	6 871	2 229	447
C _{FCAE} [cm ⁻³]	7 491	2 287	513

f _{103nm,+1}	f _{103nm,+2}	f _{130nm,+1}	f _{130nm,+3}
0,214 6	0,033 3	0,2168	0,0048

$C_N(2U)$	355,9
$C_N(3U)$	11,38
$C_N(U)$	6 767
C_N	7 135

ϕ_1	0,948 5
ϕ_2	0,049 9
ϕ_3	0,001 6
Φ	0,051 5
$\phi_{ m LIMIT}$	< 0,1
Result	ok

Concentration within the range of the FCAE

The FCAE current of 19 fC/s is above the lower limit of detection and also below the max of the FCAE.

6.3.4 Splitter bias β measurement

Configuration 1				
test	FCAE [fC/s]	CPC [cm-3]	FCAE [cm-3]	r_1
zero	0,4	0		
i = 1	19,5	6 970	7 135	0,976 9
zero	0,5	0		
i = 2	19,1	6 980	6 966	1,002 0
zero	0,5	0		
i = 3	19,5	6 980	7 097	0,983 5
zero	0,6	0		
i = 4	19,8	6 960	7 228	0,962 9
zero	0,4	0		
i = 5	19,5	6 970	7 116	0,979 5
zero	0,6	0		

Configuration 2				
test	FCAE [fC/s]	CPC [cm ⁻³]	FCAE [cm ⁻³]	r_2
zero	0,5	0		
<i>i</i> = 1	19,6	6 970	7 135	0,976 9
zero	0,6	0		
i = 2	19,6	6 980	7 154	0,975 7
zero	0,4	0		
i = 3	19,6	6 970	7 172	0,971 8
zero	0,5	0		
i = 4	19,6	6 960	7 172	0,970 4
zero	0,4	0		
i = 5	19,6	6 970	7 154	0,974 3

Configuration 2			
zero	0,6	0	

Configuration 1, repeat				
test	FCAE [fC/s]	CPC [cm-3]	FCAE [cm-3]	r_1
zero	0,2	0		
<i>i</i> = 1	19,2	7 001	7 079	0,989 0
zero	0,4	0		
i = 2	19,6	6 965	7 135	0,976 2
zero	0,7	0		
i = 3	19,4	6 970	7 022	0,992 5
zero	0,6	0		
i = 4	19,7	6 940	7 172	0,967 6
zero	0,5	0		
<i>i</i> = 5	19,3	6 975	7 022	0,993 2
zero	0,6	0		

< r ₁ >	0,982 3	Std Dev r_1	0,012 1
< r ₂ >	0,973 8	Std Dev r ₂	0,002 7
β	0,995 7	<i>u</i> (β)	0,0088

Limits 0,95 - 1,05

6.3.5 Test CPC efficiency measurements at the specific diameter and concentration:

Recorded data

test	FCAE [fC/s]	Std Dev [fC/s]	FCAE CV [%]	CPC [cm ⁻³]	Std Dev [cm ⁻³]	CPC CV [%]
zero	0,3	0,2	_	0,1	_	_
<i>i</i> = 1	19,3	0,2	1,04	6 887,4	31,6	0,46
zero	0,3	0,2	_	0,1	_	_
i = 2	19	0,3	1,58	6 807,4	64,8	0,95
zero	0,3	0,2	_	0,1	_	_
i = 3	19,4	0,3	1,55	6 914,7	59,9	0,87
zero	0,4	0,2	_	0,3	_	_
i = 4	18,8	0,2	1,06	6 712,2	42	0,63
zero	0,4	0,2	_	0,1	_	_
<i>i</i> = 5	19,5	0,1	0,51	6 908,2	34,2	0,50
zero	0,5	0,2	_	1,6	_	_

Calculated values

i	FCAE [fC/s]	FCAE [cm-3]	$\eta_{ ext{CPC}}$
1	19,0	7 490,6	0,961 2
2	18,7	7 372,4	0,965 3
3	19,1	7 510,3	0,962 5
4	18,4	7 254,1	0,967 3
5	19,1	7 510,3	0,961 6

< η _{CPC} >	0,963 5
Std Dev η	0,002 6

 $\label{eq:condition} \max \mbox{ difference from mean} \\ 0.003 \mbox{ 7 < 0.02} \quad \mbox{ ok}$

6.4 Uncertainty

Component	Symbol	Value [%]	
FCAE detection efficiency	$u_{\rm r}({\rm FCAE})$	0,90	
Multiple charge correction	$u_{\rm r}({ m MCC})$	2,40	
Splitter bias correction factor	$u_{\rm r}(\beta)$	0,88 2,33	
FCAE flow rate deviation	$u_{\rm r}(q_{ m FCAE})$		
Repeatability	$u_{\rm r}(\eta_{ m rep})$	0,27	
Relative combined	$u_{\mathrm{c,r}}(\eta)$	3,59	
Relative expanded	$U_{\mathrm{r}}(\eta)$	7,18	

From the CPC detection efficiency (η_{CPC} = 0,963 5) and the relative expanded uncertainty ($U_r(\eta)$ = 7,18 %), the expanded uncertainty $U(\eta)$ results as 0,069 2.

Annex J

(normative)

Volumetric flow rate calibration

The volumetric flow rate through an FCAE or CPC directly influences the particle count rate (number of particles per unit of time). Incorrect flow rate calibration leads to an error in the calculation of the particle number concentration. Therefore, properly calibrated flow meters and controllers are needed to perform accurate measurements of the sample flow rate and to provide traceability.

Generally three types of flow controls are used in FCAEs and CPCs: Thermal flow meters with a control valve, critical orifices, or flow restrictions (capillary or uncritical orifice) with a pressure difference measurement and a control valve. Instead of a control valve, the pump speed may also be controlled to maintain constant flow.

The first approach results in constant volumetric flow rate under a normalized condition (e.g. 273,15 K and 101,3 kPa), which is converted to the actual volumetric flow rate by taking into account the temperature and pressure in the measurement device (i.e. FCAE or CPC). This is typically done by the manufacturer.

If an instrument reports the normalized volumetric flow rate (q_n) , use Formula (J.1) to convert it to the actual volumetric flow rate (q):

$$q = q_n \frac{T}{T_n} \frac{P_n}{P} \tag{J.1}$$

where T and P the actual temperature and pressure at the flow measurement of the device and T_n and P_n the normalization temperature and pressure of the thermal flow meter.

The second approach (i.e. critical orifice) ensures constant volumetric flow rate through the orifice independent of the pressure upstream of the critical orifice, provided that the pressure downstream of the orifice is sufficiently low to achieve critical conditions. The volumetric flow rate through the orifice is only affected by the inlet temperature at the orifice *T* as shown in Formula (J.2):

$$q = q_0 \frac{T}{T_0} \tag{J.2}$$

where q_0 is the nominal volumetric flow rate at the nominal temperature T_0 . Typically, the temperature at the critical orifice is kept constant (i.e. $T_0 = T$), thus the volumetric flow rate through the orifice is constant. If temperature or pressure at the inlet of the instrument differ from temperature or pressure at the orifice, a further correction becomes necessary as shown in Formula (J.3):

$$q_{\rm in} = q \frac{T_{\rm in}}{T} \frac{P}{P_{\rm in}} = q_0 \frac{T_{\rm in}}{T_0} \frac{P}{P_{\rm in}} = q_0 \frac{T_{\rm in}}{T_0} \frac{P_{in} - \Delta P}{P_{\rm in}}$$
(J.3)

where $T_{\rm in}$ and $P_{\rm in}$ are the temperature and pressure at the inlet, respectively. ΔP is the pressure difference between the inlet of the instrument and upstream of the critical orifice. As long as ΔP is small compared to $P_{\rm in}$, the influence of changing inlet pressure remains small.

For the third approach, i.e. flow restrictions with a pressure difference measurement, the volumetric flow rate depends on both temperature and pressure.

Regardless of the method used to measure or control the flow, periodic calibration reduces errors that arise from changing environmental conditions or partial clogging of the orifice, as well as the effects

arising from the use of different aerosol working fluids, for example, the presence of CO₂ in some electrospray applications.

EXAMPLE The choked flow going to the CPC is affected by the composition of the gas. The inlet volumetric flow is inversely proportional to the square root of the relative molecular mass (formerly called molecular weight) of the gas. Examples of aerosols with gases present other than air include dioctylphthlate aerosols generated from a 2-propanol solution and electrospray aerosols where $\rm CO_2$ has been added to avoid corona discharge. A saturated 2-propanol vapour concentration at 25 °C would result in about a 3 % reduction in the flow rate and a 10 % concentration of $\rm CO_2$ would result in about a 2,5 % decrease. These examples assume that the gas composition is not diluted as it passes through the DEMC. This would be the case if the DEMC were operated in the recirculation mode. To avoid this error in the flow, the flow calibration measurement should be made with a displacement type flow meter using the aerosol with its unique gaseous composition. The flow error can be reduced to a much smaller amount by operating the DEMC without circulation. This will dilute the concentration of the $\rm CO_2$ or 2-propanol by at least the ratio of the aerosol flow to the total flow. For the case of a 10 to 1 flow ratio for sheath to aerosol flow, the flow rate would be reduced by at most 0,3 % in both cases considered above. For many applications this is a negligible amount.

The calibration procedures in <u>Clauses 6</u> and 7 may require the comparison of measured volumetric inlet flows $q_{\rm meas}$ of the reference instrument and the test CPC with the corresponding values on the instruments' certificates or the value reported by the instruments; see Formulae (2) to (4) and (11) to (13). Before these comparisons, the volumetric flow rate $q_{\rm meas}$ measured at $T_{\rm amb}$ and $P_{\rm amb}$ must be converted to $q_{\rm cal}$ at conditions $P_{\rm cert}$ and $T_{\rm cert}$ reported together with $q_{\rm cert}$. The conversion must consider the way the flow control of the instrument works when P and T change. Three different cases are most common.

a) Critical orifice with constant temperature T_0 ; the volumetric flow through the orifice is constant:

$$q_{\text{cal}} = q_{\text{meas}} \frac{T_{\text{cert}}}{T_{\text{amb}}} \frac{P_{\text{amb}}}{P_{\text{cert}}} \frac{P_{\text{cert}} - \Delta P}{P_{\text{amb}} - \Delta P}$$
(J.4)

b) Critical orifice with floating temperature $T_{\rm in}$ + ΔT , the temperature difference ΔT between inlet and critical orifice is kept constant:

$$q_{\text{cal}} = q_{\text{meas}} \frac{T_{\text{cert}}}{T_{\text{amb}}} \frac{T_{\text{amb}} + \Delta T}{T_{\text{cert}} + \Delta T} \frac{P_{\text{amb}}}{P_{\text{cert}}} \frac{P_{\text{cert}} - \Delta P}{P_{\text{amb}} - \Delta P}$$
(J.5)

c) Mass flow control with a thermal flow meter; the result is a constant volumetric flow rate under normalized conditions:

$$q_{\rm cal} = q_{\rm meas} \frac{T_{\rm cert}}{T_{\rm amb}} \frac{P_{\rm amb}}{P_{\rm cert}}$$
 (J.6)

If an instrument uses an active flow control system where the volumetric flow is kept constant, Formula (J.4) must be applied if the temperature at the point of the flow measurement is kept constant. Otherwise, Formula (J.5) must be used.

Annex K

(normative)

Testing the charge conditioner and the DEMC at maximum particle number concentration

This Annex describes the method to determine whether the charge conditioner reaches charge equilibrium and the DEMC operates unbiased by the charges on the particles at the maximum concentration level. This test is required in <u>6.3.3</u> b and <u>7.3.3</u> b. All prior preparations described in <u>Clauses 6</u> and <u>7</u> shall be completed successfully before this test is made. This test becomes important if

- the primary aerosol concentration entering the DEMC is higher than 10⁶ cm⁻³,
- the calibration aerosol contains doubly charged particles and therefore the detection efficiency calculations require corrections according to <u>Annex D</u>, or
- multiple charge correction measurements according to <u>D.3.2</u> show variations in the concentration ratios.

Follow the testing procedure below.

- a) Set the DEMC to the desired particle size (6.3.2 or 7.3.2).
- b) Set the aerosol source to the operating conditions for the desired maximum concentration level.
- c) Measure either the charge concentration [for an FCAE, use procedure in <u>6.3.5</u> b) and c)] or the particle number concentration [for a reference CPC, use procedure in <u>7.3.5</u> a) and b)].
- d) Double the voltage of the DEMC.
- e) Repeat the current or concentration measurement with the reference instrument [step c)].
- f) Calculate the ratio of the measured currents (FCAE) or particle number concentrations (reference CPC) from steps c) to e).
- g) Set the DEMC back to the desired particle size, as in step a).
- h) Reduce the primary aerosol concentration to approximately half the maximum concentration level using the diluter in the aerosol conditioner (if this concentration is too low for the reference instrument, alternatively double the aerosol concentration to twice the maximum value for the calibration experiment). Note that this concentration change should not be made by changing the operating parameters of the primary aerosol generator itself, as this might change the charge distribution in the primary aerosol.
- i) Repeat steps c) to f).
- j) If the current or concentration ratios calculated in step f) for both concentration levels do not differ by more than 10 %, the charge conditioner and the DEMC work correct with respect to the aerosol concentration.

Annex L (informative)

A recommended data recording method when using a reference FCAE

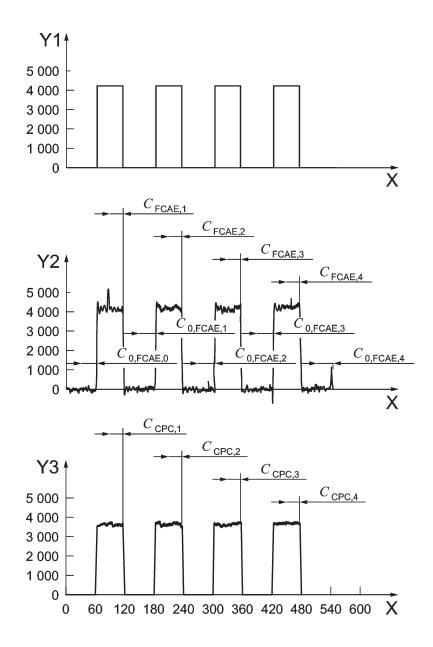
L.1 General

In calibration of a CPC by comparison with a reference instrument, particle number concentrations of an aerosol measured simultaneously by the two instruments are recorded and used to calculate the detection efficiency of the test CPC. For several reasons, measured concentrations of both the reference instrument and the test CPC usually show some time variability. For example, measured concentrations show fluctuations when the concentration is low. This occurs in CPCs because the number of particles counted per unit time at low concentrations is too few. In FCAEs, noise is more significant at low concentrations compared to CPCs because the current measurement is in the low femtoampere-range and suffers from electrical noise. Even at high concentrations, measured concentrations may vary significantly if the aerosol particle generation is unstable, or flows in the calibration system, such as those in the dilution, are not controlled well in a stable manner. These are some of the sources of random errors in calibration of the detection efficiency. The magnitude of the fluctuation of the detection efficiency can be studied by repeating the measurement. The technique described in the following section is an example of how to make such repeated measurements.

L.2 DEMC voltage cycling for repeated concentration measurements

Figure L.1 describes an example of a method to make repeated concentration measurements while the DEMC voltage was toggled between zero and the voltage for the particle size desired at a constant interval, which was 60 s in the example. In this example, an FCAE was used as the reference. Correspondingly, the test CPC and FCAE cycled through high and low concentrations every 60 s. The high concentration represented the concentration of the desired particle size, and the low concentration represented the FCAE zero offset or test CPC false counts (if there were any). The net particle concentration was then the difference between the high concentration ($C_{FCAE,i}$ and $C_{CPC,i}$ in the figure) and the arithmetic mean of two neighbouring low concentrations ($C_{0,FCAE,i}$ in the figure). Because it took a few seconds for the concentration to stabilize after the DEMC voltage toggled, only one reading was obtained for the FCAE and test CPC for each 60 s interval, as the arithmetic mean of the last 30 s of stable concentrations as indicated by the double-ended arrows in Figure L.1.

The subtraction of the FCAE offset in this manner has an advantage that the net concentration measurement would be more accurate even when the offset is drifting, compared to the subtraction of the offset value which was measured only occasionally, often only at the beginning or the end of a series of measurements.



Key

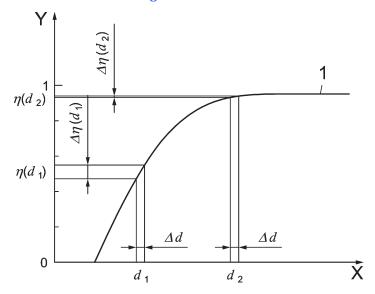
- X time [s]
- Y1 voltage set on DEMC [V]
- Y2 concentration by FCAE [cm⁻³]
- Y3 concentration by Test CPC [cm⁻³]

Figure L.1 — Example of DEMC voltage cycling and corresponding FCAE and test CPC concentration readings

Annex M (informative)

Uncertainty of detection efficiency due to particle size uncertainty

Errors may occur in the CPC detection efficiency obtained as a result of calibration if there is an error in the size of particles used in the calibration. Figure M.1 illustrate this issue.



Key

- 1 efficiency curve
- X particle diameter
- Y detection efficiency

Figure M.1 — Errors in the CPC detection efficiency due to errors in particle size and their dependence on the slope of the efficiency curve

As <u>Figure M.1</u> depicts, the magnitude of the error may vary depending on the slope of the detection efficiency curve, and the error is more significant near the lower size limit where the curve is steep. The efficiency error, $\Delta \eta$, at size d_1 can be expressed as a function of the size error, Δd , as

$$\Delta \eta(d_1) \cong \frac{\mathrm{d}\,\eta}{\mathrm{d}d}\bigg|_{d=d_1} \cdot \Delta d \tag{M.1}$$

The size of particles used in CPC calibration has some uncertainty. This means that the detection efficiency obtained by calibration has some uncertainty due to the size uncertainty, which shall be evaluated and included in estimation of the overall uncertainty. The efficiency uncertainty due to the size uncertainty, $u(\eta_{\text{size}})$, can be estimated by the following Formula (M.2):

$$u(\eta_{\text{size}}) = \frac{d\eta}{dd} \bigg|_{d=d_1} \cdot u(d)$$
(M.2)

Formula (M.2) means that, for estimating the efficiency uncertainty due to size uncertainty, $u(\eta_{\text{size}})$, the knowledge of both the efficiency curve of the test CPC, $\eta(d)$, and the uncertainty of particle size, u(d), is required.

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The uncertainty of particle size arises from several reasons, such as the uncertainty of the size of size-standard particles, uncertainties due to the finite width of the size distribution of particles classified by the DEMC, and so on.

The slope of the efficiency curve of the CPC, $(d\eta/dd_{d=d1})$ is often not well known, because the efficiency curve itself is the target of determination in a calibration. If this is the case, the slope needs to be estimated based on previous calibration data of the same CPC or data of similar CPCs (e.g. another CPC of the same model).

To minimize the uncertainty, sizing of the DEMC shall be calibrated with size-standard particles with small uncertainties. The method for calibration of DEMC sizing is described in ISO 15900.

Annex N

(informative)

Application of calibration results

The user of the CPC is primarily interested in the corrected particle concentration and its uncertainty. The result of the instrument calibration can be expressed as the corrected particle concentration, C, as a function of the measured CPC concentration, C_{CPC}

$$C = C_{\text{CPC}} / \eta \tag{N.1}$$

The most elementary practical example is the case where there is a single particle diameter in the plateau region, a single concentration is considered, and the CPC is operating in the single particle counting mode. Using the law of propagation of uncertainties, one obtains the following expression for the relative combined standard uncertainty, $u_{c,r}(C)$:

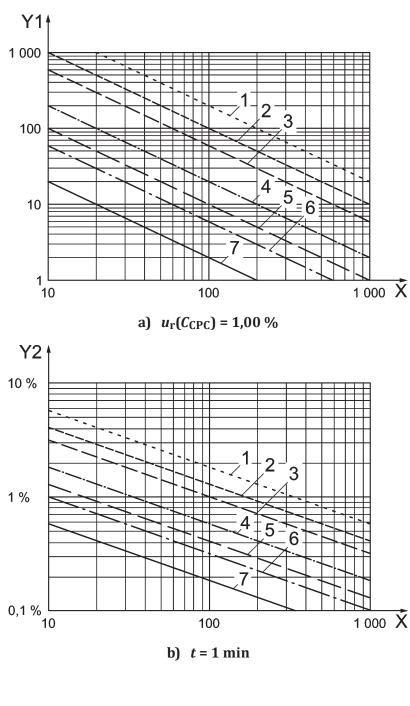
$$u_{c,r}(C) = \frac{u_c(C)}{C} = \sqrt{u_r^2(C_{CPC}) + u_r^2(\eta)}$$
(N.2)

The value of $u_r(\eta)$ for the particle diameter and concentration of interest is computed from Formula (10) based on calibration with an FCAE or Formula (17) based on calibration with a reference CPC. A lower bound value on the CPC uncertainty can be obtained from the uncertainty in the counting error, which is generally assumed to follow a Poisson probability distribution. The uncertainty from counting is expressed as:

$$u_{\rm r}(C_{\rm CPC}) = \sqrt{\frac{1}{C_{\rm CPC}q_{\rm CPC}t}}$$
 (N.3)

where q_{CPC} is the CPC detector flow rate and t is the sampling time. Formula (N.3) is valid if the expression $C_{\text{CPC}}q_{\text{CPC}}t$ is greater than 100. Figure N.1 visualizes Formula (N.3) for a given standard uncertainty of 1 % and for a given measurement time of 1 min.

A 1 % uncertainty would correspond to a concentration of 60 cm⁻³ based on a flow of 1 l/min and an averaging time of 10 s.



$q_{\rm cp}$	c [l/min]		
1	0,03	6	1,0
2	0,06	7	3,0
3	0,1	X	$C_{\rm CPC}$ [cm ⁻³]
4	0,3	Y1	t [s]to achieve $u_{\rm r}(C_{\rm CPC})$ of 1,0 %
5	0,6	Y2	$u_{\rm r}(C_{\rm CPC})$ [%] when $t = 1$ min

Figure~N.1 - Diagrams~that~illustrate~the~relationship~of~the~counting~uncertainty~with~the~CPC~detector~flow~rate~and~averaging~time

Other possible contributions to the CPC uncertainty include a drift in the aerosol source concentration over the measurement interval and an error in the flow measurement. These effects depend on the particular application of the CPC and the investigator will have to take these into account when computing the combined uncertainty.

Bibliography

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