
**Workplace atmospheres —
Characterization of ultrafine aerosols/
nanoaerosols — Determination of the size
distribution and number concentration
using differential electrical mobility
analysing systems**

*Air des lieux de travail — Caractérisation des aérosols ultrafins/
nanoaérosols — Détermination de la distribution granulométrique et de
la concentration en nombre à l'aide de systèmes d'analyse différentielle
de mobilité électrique*



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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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ISO 28439 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 137, *Assessment of workplace exposure to chemical and biological agents*, in collaboration with Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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Introduction

Within occupational hygiene, aerosol concentrations have been traditionally measured in terms of mass concentrations. For some ultrafine aerosols and nanoaerosols, other exposure metrics such as the number and surface area concentration are likely to become important for predicting health effects, depending on chemical and physical properties. This International Standard provides a method for determining the number concentration and size distribution of ultrafine aerosols and nanoaerosols at workplaces by using differential mobility analysing systems (DMASs). This can be used by occupational hygienists and researchers to measure the concentration at some workplaces. The system is generally not suitable for personal exposure measurements.

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Workplace atmospheres — Characterization of ultrafine aerosols/nanoaerosols — Determination of the size distribution and number concentration using differential electrical mobility analysing systems

1 Scope

This International Standard provides guidelines for the determination of the number concentration and size distribution of ultrafine aerosols and nanoaerosols by use of mobility particle sizers (also called differential mobility analysers). Only the particle fraction of the aerosol is considered. For ultrafine aerosols and nanoaerosols, exposure metrics such as the number and surface area concentration are important.

This International Standard also gives guidelines for the determination of workplace exposure to ultrafine aerosols and nanoaerosols.

Specifically, the differential mobility analysing system (DMAS), now available from several vendors, is discussed. Principles of operation, problems of sampling in the workplace environment, calibration, equipment maintenance, measurement uncertainty, and reporting of measurement results are covered.

Potential problems and limitations are described, which need to be addressed when limit values are fixed and compliance measurements carried out.

2 Normative references

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

ISO/TR 27628, *Workplace atmospheres — Ultrafine, nanoparticle and nano-structured aerosols — Inhalation exposure characterization and assessment*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TR 27628 and the following apply.

3.1

critical electrical mobility

Z_{crit}

electrical mobility of particles that in the differential electrical mobility classifier are transferred from the sample air flow to the exiting monodisperse aerosol flow

NOTE Due to the finiteness of the DEMC, the exiting monodisperse flow is not strictly monodisperse, but corresponds to a range of electrical mobilities for each voltage.

3.2
particle charge equilibrium
 charging condition for aerosol particles that is stable after exposure to positive and negative ions for a sufficiently long period of time

NOTE 1 Bipolar ions are produced by either a radioactive source or a corona discharge.

NOTE 2 The electrical charge on individual particles of an aerosol at charge equilibrium is not neutral.

NOTE 3 Adapted from ISO 15900:2009^[1], 2.11.

3.3
(equivalent) particle electrical mobility diameter
 diameter of a sphere with the same electrical mobility as the particle in question

4 Symbols and abbreviated terms

4.1 Symbols

B	particle mechanical mobility	s/kg
C	Cunningham correction factor	1
C_N	aerosol number concentration	1/m ³
d	particle diameter	nm
d_p	equivalent particle electrical mobility diameter	m
$\bar{d}_p$	average equivalent particle electrical mobility diameter	m
D	particle diffusion coefficient	m ² /s
e	basic unit of charge (elementary charge)	1,602 177 × 10 ⁻¹⁹ C
q_1	DEMC sample air flow rate	m ³ /s
q_2	DEMC filtered sheath air flow rate	m ³ /s
q_3	DEMC excess air flow rate	m ³ /s
q_4	DEMC exiting air flow rate to particle detector	m ³ /s
k	Boltzmann constant	1,38 × 10 ⁻²³ N·m/K
L	length of sampling line	m
n	number of charges	1
p	penetration through sampling line	1
t	(coagulation) time	s
t_{scan}	scan time	s
T	absolute temperature at which the DEMC is operated	K
V_v	volume of buffer vessel for the sample air flow rate	m ³
Z	electrical mobility of a charged airborne particle	m ² /V·s
Z_{crit}	critical electrical mobility of a charged airborne particle	m ² /V·s
η	gas viscosity	Pa·s
μ	parameter for diffusion losses	1

4.2 Abbreviated terms

CNC condensation nuclei counter

CPC condensation particle counter

DEMC differential electrical mobility classifier

DMAS differential mobility analysing system

NOTE A DMAS is also known as a differential mobility particle sizer (DMPS) or scanning mobility particle sizer (SMPS).

HEPA high efficiency particle arrestor

5 Principle

The aerosol is sampled in the workplace at a position representative of the atmosphere to which a worker might be exposed. Larger particles than approximately 1 µm are precipitated and the particles smaller than approximately 1 µm drawn into the instrument. After charge conditioning, the aerosol particles are separated in the electrical field of the DEMC (see References [6] and [7]) according to their electrical mobility, which is given by Equation (1).

$$\left. \begin{aligned} Z &= neB \\ B &= \frac{C}{3\pi\eta d_p} \end{aligned} \right\} \quad (1)$$

where

Z is the electrical mobility, in metres squared per volt second, of a charged aerosol particle;

n is the number of electrical charges;

e is the basic unit of charge (elementary charge), $1,602\,177 \times 10^{-19}$ C;

B is the particle mechanical mobility, in seconds per kilogram;

C is the Cunningham correction factor;

η is the gas viscosity, in pascal seconds;

d_p is the equivalent particle electrical mobility diameter, in metres.

The critical particle electrical mobility, Z_{crit} , is directly related to the geometric dimensions of the DEMC. The equivalent particle electrical mobility diameter, d_p , can be determined from equations provided by the instrument manufacturer.

Particles of a certain size or size interval are counted in a condensation nuclei counter (CNC) [also known as a condensation particle counter (CPC)] or electrometer, and the particle number concentration for each size or size interval is determined. By scanning or stepwise changing the voltage of the DEMC, a number size distribution is obtained. The size range from 3 nm to 1 000 nm in electrical mobility diameter can be partly covered by different instruments (see Reference [8]). The DEMC has the advantage that the electrical mobility diameter is approximately equivalent to the projected-area diameter of particles (defined as the diameter of a sphere with the same projected area as the particles being sized) with compact geometries. The entire number concentration is obtained by adding or integrating all size channels.

Though the composition of the sampled particles cannot be obtained, the distribution of surface area and volume concentration in some instances, e.g. if the particles are known to be spherical, can be estimated from calculations provided by the manufacturer or in the literature.

6 Equipment

6.1 General

A DMAS consists of different instrument sections (see Figure 1):

- pre-separator;
- particle charger or particle charge conditioner;
- differential electrical mobility classifier (DEMC), with flow control and high voltage control;
- particle detector;
- system controller, with data acquisition and data analysis (typically built-in firmware or dedicated software on a personal computer).

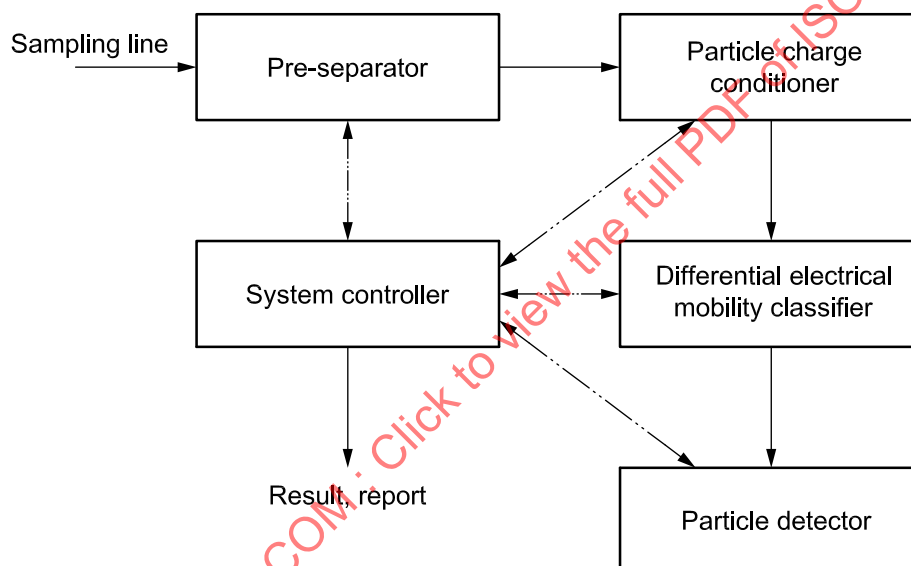


Figure 1 — Major parts of a DMAS

6.2 Sampling line

The aerosol is often sampled with a flexible tube in order to access the breathing zone of a worker. The material of the tube shall be an electrical conductor. Particle diffusion losses should be minimized. This can be accomplished by using tubes of short length. For example, the application of flexible rubber tubes of conducting material of length up to a few metres with an inner diameter of 4 mm or 6 mm ensures a short residence time in the tube (see 11.3.2). The flow in the sampling line shall be laminar.

When sampling highly fluctuating aerosols like welding fumes it is recommended additionally that a buffer vessel be used in order to average the concentration during the scan. The buffer vessel shall be electrically conducting and be earthed. The mean residence time of the buffer vessel shall be related to the scan time. In order to get a relatively stable concentration over the scan time, the volume of the vessel can be chosen in relation to the sample air flow rate according to Condition (2).

$$\frac{V_v}{q_1 t_{\text{scan}}} \geq 5 \quad (2)$$

where

V_v is the volume, in metres cubed, of the buffer vessel for the sample air flow rate;

q_1 is the DEMC sample air flow rate, in metres cubed per second;

t_{scan} is the scan time, in seconds.

EXAMPLE For a sample air flow rate of 0,3 l/min ($0,5 \times 10^{-5} \text{ m}^3/\text{s}$) and a scan time of 2 min (120 s), a vessel of 3 l ($3 \times 10^{-3} \text{ m}^3$) is appropriate.

Smaller buffer volumes are preferred if the aerosol agglomerates by coagulation (high concentration of primary particles) (see 11.3.3).

NOTE The state of knowledge at the time of publication allows no recommendation for an upper volume to be made.

6.3 Pre-separator

A pre-separator is required such that large particles above the desired measurement range are precipitated. This can be done, for example, by use of a suitable impactor or cyclone. The pre-separator shall be cleaned and, if necessary, greased regularly.

6.4 Particle charge conditioner

The aerosol is charged with free electrical charges by collisions with gas ions and electrons. The free electrical charges are usually produced by a radioactive source in the air stream, separated by a thin enclosure. Sources like ^{85}Kr , ^{210}Po or ^{241}Am are used. The entire aerosol reaches a charge equilibrium of known distribution (see Reference [9]).

NOTE The charging of non-spherical particles differs from that of spheres. Therefore the distribution of electrical charges as a function of particle size employed in the inversion of the critical electrical mobility into a particle size interval is strictly valid only for spherical particles.

6.5 DEMC

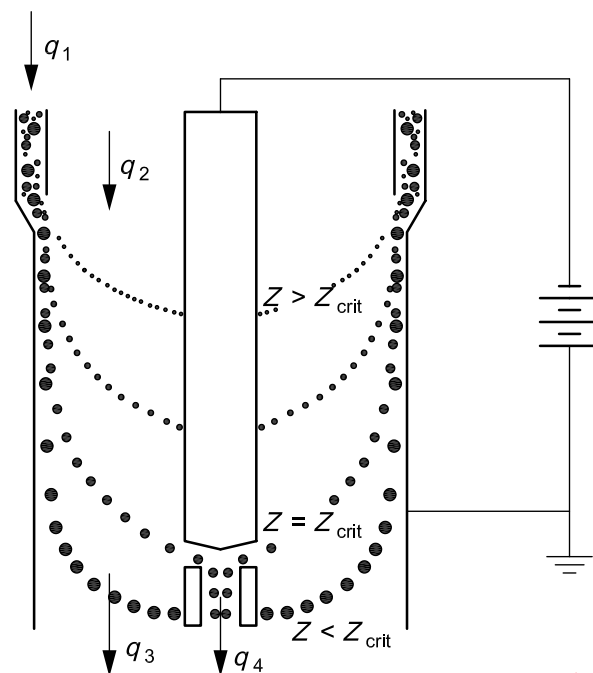
The conditioned aerosol reaches the electrical mobility classification section. A common DEMC comprises an inner and an outer electrode maintained at an electrical potential difference typically between 20 V and 10 000 V (see Figure 2).

The particles are transported in laminar flow along an annular region or tube along with a clean air sheath. The motion of the charged particles depends on their different mobilities, causing them to reach the electrode at different positions. Particles of a narrow electrical mobility range centred on the critical electrical mobility are sampled via a slit towards the end of the annular region and transported into the detection section.

6.6 Aerosol particle detector

The separated aerosol with the specified, critical, electrical mobility is led to a counter which determines the number of particles per unit volume. The most widely used counter is the CPC (CNC). In this device, the aerosol is brought into contact with supersaturated vapour (alcohol or water) which condenses on to the particles. The particles grow rapidly to large droplets, typically several micrometres in diameter, and can then be detected using optical methods. Another detector is the electrometer, which determines the net electrical current provided by the sampled particles.

After passing through the detector, further analysis of the particles is not possible. Additional samplers operated in parallel to the DMAS can be used to collect samples for further analysis, e.g. an electrostatic or thermal precipitator.



Key

- q_1 DEMC sample air flow rate
- q_2 DEMC filtered sheath air flow rate
- q_3 DEMC excess air flow rate
- q_4 DEMC exiting air flow rate to particle detector
- Z electrical mobility of a charged airborne particle
- Z_{crit} critical electrical mobility of a charged airborne particle

Figure 2 — Scheme of a DMAS

7 Measurement strategy

The DMAS gives detailed information on the aerosol size distribution within a defined range. The DMAS can be used to detect particle-emitting sources in the workplace, or measurements in the breathing zone of a worker may be carried out to get information on personal exposure. Since DMAS instruments available at the time of publication can only be used as static samplers, if possible a representative sampling position should be found with the use of tubes, as specified in 6.2. Workplace air flow pattern characteristics, e.g. direction of air flow relative to source and sampling position, and the presence of multiple sources are essential for either selection of a sampling position or interpretation for worker exposure.

For workers moving around, a more structured sampling strategy is necessary (see Annex A).

NOTE For some workplace conditions, it is not possible to find a representative sampling location. In these cases, the values of the measured data are less reliable.

General information about measurement strategy in workplaces can be found in EN 689^[2].

8 Measuring procedure

8.1 Preparation

The response of a DMAS shall be checked regularly, e.g. before and after a series of measurements, against particle size standards (see 10.1).

Before performing a series of measurements, a zero check of the DMAS is useful by applying high efficiency particle arrestor (HEPA) filters to both the sampling line and the sheath air.

8.2 Sampling

If worker exposure is to be estimated, the aerosol shall be sampled in the breathing zone of the worker. All existing DMASs at the time of publication are large static instruments. The sample should be taken at a representative position in or very near the worker's breathing zone. A sampling line as specified in 6.2 can be applied. If the worker is not stationary, samples from different locations can be taken by moving either the entry of the sampling line or the complete DMAS.

Diffusion losses in the line shall be taken into account (see Clause 11).

If the instrument performs a scanning or stepping voltage mode, the time for one measurement is a few minutes, typically 3 min to 6 min. During this time, the aerosol should not differ in size and number of particles. If highly fluctuating concentrations, such as in welding workplaces, are to be measured, it is recommended that a buffer vessel of a few litres' volume be used in the sampling line to avoid artificial peaks during a scan. Another option is to average a series of samples if short-term emissions appear regularly.

Some DMAS devices allow a selection of instrument parameters like sampling air flow rate, sheath air flow rate, scan times or even a change of the DEMC type. These settings affect the measurement range. It is recommended that the range be selected in such a way as to cover the whole size distribution of the ultrafine particles at the workplace. Practical experience shows that the maxima of the particle number size distributions lie between approximately 10 nm and 500 nm, depending on the occurrence of primary, aggregated and agglomerated ultrafine particles.

The total number concentration integrated over the measurement range found at workplaces varies between 10^9 particles/m³ and 10^{14} particles/m³ in clean air sections or urban background and welding plumes, respectively (see Reference [10]).

9 Presentation and evaluation of data

The resulting distribution of the number concentration, C_N , shall be given as differential concentration in terms of the equivalent particle electrical mobility particle diameter, d_p , e.g. dC_N/dd_p or $d\log C_N/d\log d_p$, in order to make different instrument settings (number of channels per decade, width of channels) comparable.

Depending on the object of the measurements, the number size distribution data can be presented in a number of different ways, for example:

- the calculation of statistical data like maximum or median values is of value and is possible if the entire distribution is sufficiently covered;
- calculation of the total number concentration in the measurement range, the total number concentration for the range below 100 nm, or any other sub-fraction could be reported as a single value for comparison of different exposure scenarios;
- the time-weighted average size distribution and the total number concentration can be determined for the whole reference period (e.g. 8 h or 15 min);
- in order to present the variation in concentration over the whole size distribution for the whole reference period or measurement period, plot the size distributions as a two-dimensional contour diagram (with time on the x -axis, particle size on the y -axis and with concentration as contours) or as a three-dimensional surface or wire-frame diagrams (with time on the x -axis, particle size on the y -axis, and concentration on the z -axis);
- peaks related to process or workplace activities.

NOTE The particle surface area and particle volume distribution can be estimated from the number size distribution if mainly compact or spherical particles or agglomerates and aggregates are sampled. For other non-spherical particle shapes, e.g. tubes and ropes, both these estimates as well as the number size distribution are less accurate. These distributions can be presented in similar ways to the number size distribution data listed above.

An alternative presentation can be achieved by splitting the size distribution into its constituent modes (peaks), determining the characteristics of each mode (median diameter, geometric standard deviation and particle concentration), and plotting the temporal evolution of the characteristics of the modes in a few diagrams.

All relevant instrument parameters like measurement range, air flow rates, scan time, as well as DMAS and CPC model identification, shall be stated in the measurement report. In addition, sampling equipment such as a buffer vessel or sampling line shall be described. Diffusion loss corrections shall be detailed. Sampling time shall be given.

The principal measure when using DMAS devices is the number concentration. A comparison to mass-based occupational limit values of airborne hazardous substances is not possible. For example, scanning electron microscopy or transmission electron microscopy can be used for the determination of chemical composition and can also provide an alternate determination of particle size (see ISO/TR 27628).

10 Check of DMAS performance

10.1 Check on particle classification

Particle size calibration is possible with use of polystyrene reference particles, which are available in the size range 20 nm to 900 nm for use with DMAS and certified against standards. It is useful to have three different sizes for control purposes of a wide measurement range. The particles are suspended in deionized water, sprayed with an atomizer and partially dried before sampling.

The smallest polystyrene particle that can be used for calibration of a DMAS is approximately 100 nm. The reasons for this are twofold: the particle size peak of smaller particles is usually superposed on the background of particles generated from water impurities or residuals; and the surface of the sprayed polystyrene particles can contain a thin film consisting of water impurities causing the particles to behave larger than specified.

NOTE This means that the performance of a DMAS cannot easily be tested by the user in the size range of ultrafine particles and nanoparticles smaller than 100 nm. The only available method at the time of publication for calibration with particles smaller than 100 nm is to use particles generated by electro-spray or electro-sparks. This method, however, is at the time of publication too complicated to be used for end-user applications. See also ISO 15900^[1] for a test of the calibration of a DMAS.

10.2 Check on particle number-counting efficiency

A calibration against the number concentration is only possible at the time of publication with a reference instrument and a stable particle source. At the time of publication, there is no primary standard for particle number concentration. A possible standard method could be based on a stable coagulation effect (see Reference [11]).

At the time of publication, the manufacturers of DMAS use reference instruments to set the count rate of the instruments they supply. An alternative recommended way is to participate in round robin tests (see Reference [12]) which are commercially available (see, for example, "calibration workshops" in Reference [13]). For a comparison of a CPC with an electrometer, see ISO 15900^[1].

11 Problems and errors

11.1 CPC (CNC) counting efficiency

The counting efficiency of a CPC (CNC) varies over the available measurement range. For example, the CPC is characterized by a lower particle size at which the counting efficiency is 50 %. Information from the manufacturer shall be taken into account and the applicable measurement range shall be defined. The aerosol particle detector efficiency as a function of particle size shall be known and included in the data inversion, which is usually included in the manufacturer's software. All corrections to the counting efficiency applied to the raw data shall be noted in the report. The maximum detectable number concentration of a CPC (CNC) is limited by coincidence counts, leading to an underestimation of the concentration. Dilution systems at the front of the sampling line can be used to avoid this effect. If the counting efficiency is not known, it shall be stated in the test report.

11.2 Particles with multiple charges

Multiple charges of the particles can lead to an underestimation of the diameter of the particles. A DMAS usually offers a correction for this effect. The correction is based on known proportions of multiple-charged particles at charge equilibrium. These effects shall be calculated and shall be corrected for in the software of the instrument. The method of multiple charges shall be documented in the test report.

11.3 Sampling losses

11.3.1 General

There are three types of sampling loss that modify (bias) the measured size distribution:

- a) diffusion losses inside the sampling tube (if used), see 11.3.2;
- b) coagulation inside the buffer vessel (if used), see 11.3.3;
- c) diffusion losses inside the DMAS, see 11.3.4.

11.3.2 Diffusion losses in a sampling tube

For a sampling line (with circular cross-section), the diffusion losses can be estimated using Equation (3) (see Reference [14], section 7.4):

$$p = \begin{cases} 1 - 5,50\mu^{2/3} + 3,77\mu & \mu < 0,009 \\ 0,819 \exp(-11,5\mu) + 0,0975 \exp(-70,1\mu) & \mu \geq 0,009 \end{cases} \quad (3)$$

$$\mu = \frac{DL}{q_1}$$

$$D = kTB$$

where

- p is the penetration through the sampling line;
- μ is the parameter for diffusion losses;
- B is the particle mechanical mobility, in seconds per kilogram;
- D is the particle diffusion coefficient, in metres squared per second;
- k is the Boltzmann constant, $1,38 \times 10^{-23}$ N·m/K;
- L is the length of the sampling line, in metres;
- q_1 is the DEMC sample air flow rate, in metres cubed per second;
- T is the absolute temperature at which the DEMC is operated, in kelvins.

An example of diffusion losses is shown in Figure 3. The losses are calculated for laminar flow in a tube of length 1 m for air flows of 0,3 l/min and 1 l/min (equal to $0,5 \times 10^{-5}$ m³/s and $1,7 \times 10^{-5}$ m³/s), respectively. The diameter of the tube is irrelevant for the calculation of diffusion losses. For particle diameters below 40 nm (equal to 40×10^{-9} m), the losses in this example exceed 5 %.

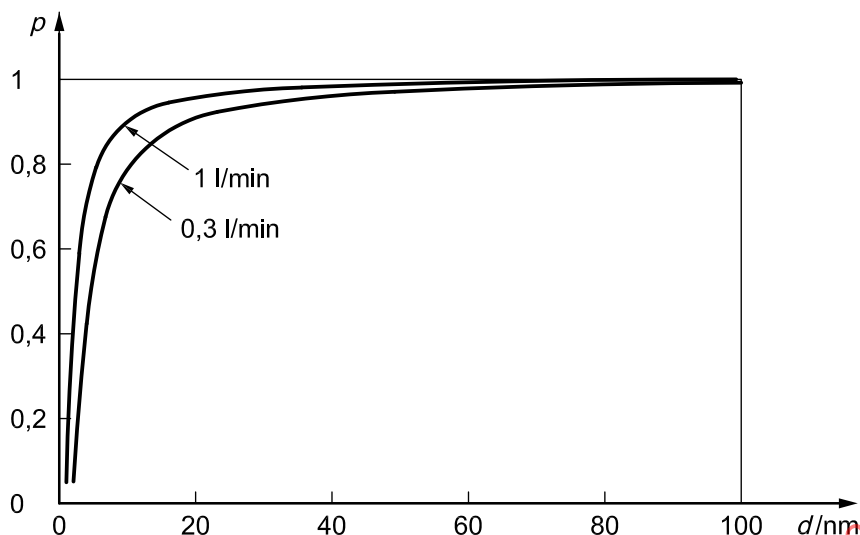
**Key** p penetration d particle diameter

Figure 3 — Diffusion losses calculated for a tube of 1 m length operated at two flow rates

11.3.3 Bias generated by a buffer vessel

Applying a buffer vessel (see 6.2) in the sampling line can lead to a change both in the aerosol number concentration and in the size distribution. The residence time inside a buffer vessel causes high-concentration aerosols, e.g. of welding fume or wood smoke, to coagulate. This lowers the particle number concentration and increases the count median particle diameter. For an aerosol with a unimodal size distribution, a buffer vessel shall not be used if the number concentration, C_N , exceeds $0,3 \times 10^{12}/\text{m}^3$.

NOTE Wall losses occur in the buffer vessels, but no recommendation can be given on how to estimate them.

11.3.4 Internal diffusion losses

Particle losses inside the DMAS should be specified by the manufacturer and taken into account by the instrument's software, but this needs to be verified by the user. Diffusion losses are highest for low sample air flow rates, generally lower than 0,5 l/min. The user can determine the internal losses of a low-flow DMAS by parallel sampling with a high-flow DMAS using a test aerosol generated in a laboratory, homogeneous in space and constant over time, and, by comparing the measured concentrations, arrive at a particle size-dependent correction formula. Another method is to compare a tandem DMAS (without charge conditioning before the second DMAS) with a single DMAS, which are all set to the same particle mobility.

11.4 Uncertainties

11.4.1 General

Uncertainties for the determination of the electrical mobility Z originate either from the operating conditions like air flow rates, the applied electrical voltage of the DEMC or the efficiency of aerosol charging, or from the design of the DMAS like tolerances of the geometry or internal particle losses. The combined uncertainty is described in ISO 15900^[1].

11.4.2 Air flow rates

The stability of the sample air flow rate affects both the determination of the mobility diameter and the number concentration in the detector.

The stability of the sheath air flow rate influences the determination of the mobility diameter. Often ratios of 1:10 for the sample to sheath air flow rate ratio are used. Differing ratios than those set by the manufacturer change the transfer function of the DMAS and thus the resolution in particle size.

The air flow rates of the instruments shall be checked regularly and the air flow rate measurement devices shall be calibrated. Turbulent air flows inside the mobility analyser section shall be avoided. This is usually taken into account when following the manufacturer's recommendations.

11.4.3 Electrical voltage

The voltage inside the DEMC directly affects the selection of particles according to their electrical mobility. Fluctuations lead to different mobility diameters and broaden the transfer function. A calibration of the high-voltage source and meter is important.

11.4.4 Design of DMAS

Inaccurate geometrical dimensions of the DMAS lead to deviations of the selection of particle mobility. Additionally internal particle losses in the sample flow system shall be taken into account.

11.5 Overloading

At very high particle number concentrations, a small number of particles can remain inside the DMAS, e.g. in the downstream part of the classifier, for a time longer than one scan period. When starting a subsequent scan these particles are initially counted at an incorrect size position and lead to artificial concentrations. Allowing a longer time period between scans can eliminate this behaviour (see Reference [15]).

11.6 Sampling of fibres

If fibres or chain-like aggregates are sampled, the corresponding mobility diameter is not the same as the fibre diameter or its length. To get more information on the shape at a certain mobility diameter, it is useful to sample those particles at a fixed mobility diameter, e.g. by use of an electrostatic precipitator behind the classifier instead of the particle detector, and to analyse them with a scanning or transmission electron microscope.

11.7 Humidity

Humidity effects can influence the particle size especially when sampling hygroscopic aerosols or under strongly changing humidity. Drying of the aerosol prior to sampling using a diffusion dryer or dry dilution air or other humidity control devices can be useful to get stable conditions. In these cases, the test report shall contain a full description of the humidity control of the measurement system.

11.8 Maintenance

The DMAS shall be cleaned regularly in order to avoid changes of the electrical field inside the DMAS. The maintenance of the instrument shall be carried out according to the manufacturer's instructions, depending on the frequency of use. For example, the liquid in the CPC shall be replaced regularly.

Local, national and international regulations (e.g. Council Directive 96/29/Euratom, see Reference [5]) for radiological protection shall be followed when using and storing the radioactive source in the particle charge conditioner and when transporting the DMAS to different places. Usually it is necessary for the management of a company visited for purposes of sampling to be informed about the use of the radioactive source.