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Test method to measure the efficiency of air filtration media against spherical nanomaterials —

Part 2: Size range from 3 nm to 30 nm

Méthode d'essai pour mesurer l'efficacité des médias de filtration d'air par rapport aux nanomatériaux sphériques —

Partie 2: Spectre granulométrique de 3 nm à 30 nm

Reference number
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Foreword

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This document was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 195, *Air filters for general cleaning*, in collaboration with ISO Technical Committee TC 142, *Cleaning equipment for air and other gases*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

A list of all parts in the ISO 21083 series can be found on the ISO website.

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Introduction

Nano-objects are discrete piece of material with one, two or three external dimensions in the nanoscale (see ISO/TS 80004-2) and are building blocks of nanomaterials. Nanoparticles, referring to particles with at least one dimension below 100 nm, generally have a higher mobility than larger particles. Because of their higher mobility and larger specific surface area, available for surface chemical reactions, they can pose a more serious health risk than larger particles. Thus, particulate air pollution with large concentrations of nanoparticles can result in an increased adverse effect on human health and an increased mortality (see Reference [15]).

With the increased focus on nanomaterials and nanoparticles, the filtration of airborne nanoparticles is also subject to growing attention. Aerosol filtration can be used in diverse applications, such as air pollution control, emission reduction, respiratory protection for human and processing of hazardous materials. The filter efficiency can be determined by measuring the testing particle concentrations upstream and downstream of the filter. The particle concentration may be based on mass, surface area or number. Among these, the number concentration is the most sensitive parameter for nanoparticles measurement. State-of-the-art instruments enable accurate measurement of the particle number concentration in air and therefore precise fractional filtration efficiency. Understanding filtration efficiency for nanoparticles is crucial in schemes to remove nanoparticles, and thus, in a wider context, improve the general quality of the environment, including the working environment.

Filtration testing for nanoparticles, especially those down to single-digit nanometres, is a challenging task which necessitates generation of a large amount of extremely small particles, and accurate sizing and quantification of such particles. The thermal rebound remains a question for particles down to 1 nm to 2 nm (see Reference [11]). The accuracy of particle size classification is complicated by very strong diffusion of particles below 10 nm (see References [7] and [8]). The state-of-the-art commercial condensation particle counters for general purposes can detect particles down to 1 nm to 2 nm.

A large number of standards for testing air filters exist such as the ISO 29463 and ISO 16890 series. The test particle range in the ISO 29463 series is between 0,04 µm and 0,8 µm, and the focus is on measurement of the minimum efficiency at the most penetrating particle size (MPPS). The test particle range in the ISO 16890 series is between 0,3 µm and 10 µm. The ISO 21083 series aims to standardize the methods of determining the efficiencies of filter media, of all classes, used in most common air filtration products and it focuses on filtration efficiency of airborne nanoparticles, especially for particle size down to single-digit nanometres.

Advances in aerosol instruments and studies on nanoparticle filtration in the recent years provide a solid base for development of a test method to determine effectiveness of filtration media against airborne nanoparticles down to 3 nm range.

Test method to measure the efficiency of air filtration media against spherical nanomaterials —

Part 2: Size range from 3 nm to 30 nm

1 Scope

This document specifies the testing instruments and procedure for determining the filtration efficiencies of flat sheet filter media against airborne nanoparticles in the range of 3 nm to 30 nm. The testing methods in this document are limited to spherical or nearly-spherical particles to avoid uncertainties due to the particle shape.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 5167 (all parts), *Measurement of fluid flow by means of pressure differential devices inserted in circular cross-section conduits running full*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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

ISO 27891, *Aerosol particle number concentration — Calibration of condensation particle counters*

ISO 29464, *Cleaning of air and other gases — Terminology*

3 Terms, definitions, symbols and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5167-1, ISO 5725-1, ISO 5725-2, ISO 15900, ISO 27891, and ISO 29464 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.2 Symbols and abbreviated terms

3.2.1 Symbols

Symbol	Definition
A	Source strength of the radioactive source
A_0	Original source strength of the radioactive source
A_f	Effective filtration surface area
C_up	Particle concentration upstream of the filter medium
$C_{\mathrm{up},i}$	Concentration of particles with the i_th monodisperse size upstream of the filter medium
C_down	Particle concentration downstream of the filter medium
$C_{\mathrm{down},i}$	Concentration of particles with the i_th monodisperse size downstream of the filter medium
$C_{\mathrm{n}i}$	Concentration of particles after the second DEMC for the particles with *i* charge(s)
d_d	Diameter of the initial droplet including the solvent
d_p	Diameter of the testing particle after complete evaporation of the solvent
E	Filtration efficiency of the test filter medium
E_i	Filtration efficiency of the test filter medium against the particles with the i_th monodisperse size
e	Charge of an electron
φ_v	Volume fraction of DEHS in the solution
$t_{0,5}$	Half-life of the radioactive source
N_up	Total count of particles upstream of the filter medium in a certain user-defined time interval
$N_{\mathrm{up},i}$	Counts of particles with the i_th monodisperse size upstream of the filter medium in a certain user-defined time interval
N_{down}	Total count of particles downstream of the filter medium in a certain user-defined time interval
$N_\mathrm{down,i}$	Counts of particles with the i_th monodisperse size downstream of the filter medium in a certain used-defined time interval
$N_{\mathrm{n}i}$	Total count of particles after the second DEMC for the particles with *i* charge(s)
n_p	Number of elementary charges
P	Fractional penetration of the test filter medium
P_i	Fractional penetration of particles with the i_th monodisperse size for the test filter medium
P_m	Penetration with the filter medium, before applying the correlation ratio
$P_{\mathrm{m},i}$	Measured penetration against particles with the i_th monodisperse size when the filter medium is installed in the filter medium holder, before applying the correlation ratio
q	Flow rate through the filter medium
q_e	Air flow rate through the electrometer
R	Correlation ratio
R_i	Correlation ratio for the i_th monodisperse particle size, obtained as the penetration without the filter media
R_es	Resistance of resistor
t	Time
v_f	Filter medium velocity
V	Voltage
x	Volume of the sampled air
α	Angle for the transition section in the filter medium holder
Δp	Pressure drop across the filter medium
E_0	Initial particulate efficiency of media sample
ΔE_c	Difference in particulate efficiency between E_0 and conditioned efficiency of the media sample
λ	Radioactive decay constant equal to 0,693/ $t_{0,5}$

3.2.2 Abbreviated terms

AC	Alternating current
CAS	Chemical abstracts service
CL	Concentration limit
CMD	Count median diameter
CPC	Condensation particle counter
DEHS	Di(2-ethylhexyl) sebacate
DEMC	Differential electrical mobility classifier
DMAS	Differential mobility analysing system
HEPA	High efficiency particulate air
Kr	Krypton
IPA	Isopropyl alcohol
MPPS	Most penetrating particle size
Po	Polonium
PSL	Polystyrene latex
RH	Relative humidity
SRM	Standard reference material

4 Principle

The filtration efficiency of the filter medium is determined by measuring the particle number concentrations upstream and downstream of the filter medium. The fractional penetration, P, represents the fraction of aerosol particles which can go through the filter medium, as shown in Formula (1):

$$P = C_{\text{down}} / C_{\text{up}} \quad (1)$$

where C_{down} and C_{up} are the particle concentrations downstream and upstream of the filter medium, respectively. Another way is to measure the particle counts upstream and downstream of the filter medium for a certain same user-defined time interval and sampling volume rate. Then, the penetration is the ratio between the downstream count, N_{down}, and upstream count, N_{up}, as shown in Formula (2):

$$P = N_{\text{down}} / N_{\text{up}} \quad (2)$$

The filter medium efficiency, E, is the fraction of aerosols particles removed by the filter medium, as shown in Formula (3):

$$E = 1 - P \quad (3)$$

The filter medium efficiency is dependent on the challenge particle size. If the test is performed with a number of monodisperse particles with different sizes, the expression for the penetration of particles with the i_{th} monodisperse size, P_i, can be written as shown in Formula (4):

$$P_i = C_{\text{down},i} / C_{\text{up},i} \quad (4)$$

where $C_{\text{up},i}$ and $C_{\text{down},i}$ are the concentration of particles with the i_{th} monodisperse size upstream and downstream of the filter medium, respectively. If the test is performed with a number of monodisperse particles with different sizes, the expression for the penetration of particles with the i_{th} monodisperse size, P_i can be written as shown in Formula (5):

$$P_i = N_{down,i} / N_{up,i} \tag{5}$$

where $N_{up,i}$ and $N_{down,i}$ are the counts of particles with the i_{th} monodisperse size upstream and downstream of the filter medium in the same user-defined time interval and sampling volume rate, respectively. Correspondingly, the filtration efficiency, E_i, of the test filter medium against the particles with the i_{th} monodisperse size is as shown in Formula (6):

$$E_i = 1 - P_i \tag{6}$$

The test particles in the range from 3 nm to 30 nm are generated by an evaporation-condensation method. One realization of this method is the generation of silver (Ag) particles from an electrical tube furnace.

The test particle from the generator is neutralized. The particles are mixed homogeneously with filtered test air if necessary to achieve desired concentration and flow rate, before they are used to challenge the test filter medium.

A specimen of the sheet filter medium is fixed in a test filter assembly and is subject to the test air flow corresponding to the prescribed filter medium velocity. Partial flow, which is the flow that the CPC operates with, of the test aerosol is sampled upstream and downstream of the filter medium, and the fractional penetration is determined from the upstream and downstream number concentrations or total numbers in user-defined time intervals. Furthermore, the measurement of the pressure drop across the filter medium is made at the prescribed filter medium velocity.

Additional equipment is required to measure the absolute pressure, temperature and RH of the test air. It is also needed to measure and control the air volume flow rate.

5 Test materials

5.1 General

Any aerosol used to test the filtration performance according to this test method shall only be introduced to the test section as long as needed to test the filtration performance properties of the test filter medium without changing the filtration performance properties of the subject test filter medium due to loading, charge neutralization or other physical or chemical reaction.

5.2 Solid phase aerosol — Silver test aerosol as an example

Pure silver powder source – Ag (99,999 %)

Pure silver powder properties:

Density	$10{,}49 \cdot 10^3$ kg/m^3
Melting point	1 234 K
Boiling point	2 434 K
Solubility	insoluble in water

5.3 Solid phase aerosol generation method

Silver nanoparticles or nanoparticles of other materials can be used as long as the qualification procedure is performed and the requirements are fulfilled.

Silver nanoparticles can be generated by the evaporation-condensation method (see Reference [17]). An electric furnace is used to generate silver nanoparticles from a pure silver powder source (99,999 %), and clean compressed air or other gases, such as nitrogen, is used as a carrier gas with flow rate of 16,7 m^3/s to $50 \cdot 10^{-6}$ m^3/s (1 l/min to 3,0 l/min). The silver powder source located in the centre of a heating tube is vaporized and condensed into silver nanoparticles with a relatively wide size distribution when the air flow exits the tube furnace. For very small particles a rapid temperature decrease may be applied at the exit of the tube furnace so as to produce particles in the desired size range. As an example, some technical specifications regarding tube furnaces are presented in Annex A, Tables A.1 to A.4.

Any other generator capable of producing particles in sufficient concentrations in the particle size range of 3 nm to 30 nm so that the particle concentration upstream of the test filter medium is at least 1 000 per cm^3 under any of the test mode, such as monodisperse or polydisperse test described in Clause 6, can be used.

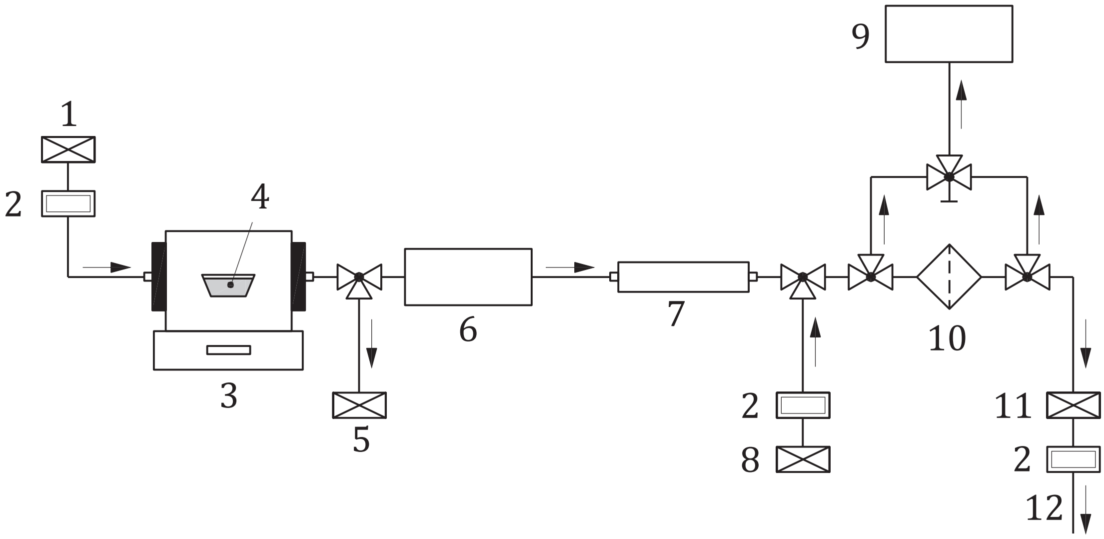
6 Test setup

6.1 General

The test setup is shown in Figure 1 for monodisperse challenge particles and in Figure 2 for polydisperse challenge particles. When the challenge particles are monodisperse, the setup consists of three sections: the one that produces the aerosol particles (which contains the aerosol generator), the particle classification section (which contains the DEMC) and the particle measuring section (which contains the CPC). When the challenge particles are polydisperse, the particle classification shall be performed after sampling the aerosol from the upstream or downstream section.

The measurement with monodisperse particles is the reference test while the measurement with polydisperse particles shall be qualified carefully and verified by comparison with monodisperse test for validating the measurement procedure.

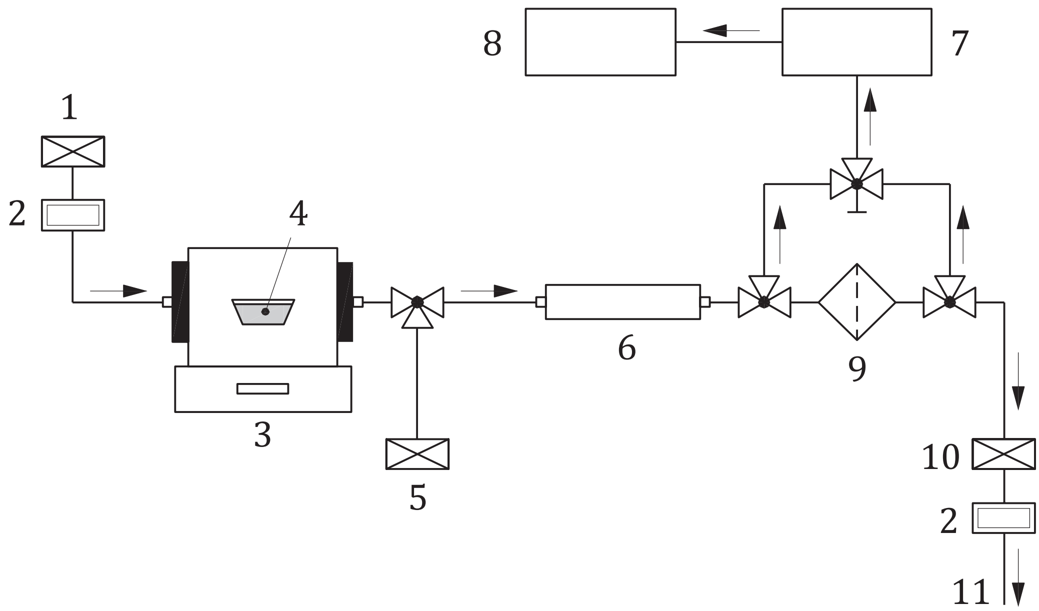
Tests using monodisperse and polydisperse aerosols should yield equivalent results if they are carried out correctly. Japuntich et al.[9] performed both polydisperse and monodisperse measurements down to 20 nm to 30 nm range and showed reasonable agreement. Buha et al.[20] compared polydisperse test results with models down to similar size range and showed good agreement. With the particles in even smaller size range, the size distribution measurement downstream of the filter is increasingly difficult.



Key

1 air or N2 through HEPA filter
2 flow controller
3 furnace
4 silver
5 excess flow with HEPA filter
6 DEMC
7 neutralizer
8 make up air with HEPA filter
9 CPC
10 filter medium holder
11 HEPA filter on the exhaust line
12 vacuum

Figure 1 — Test setup for monodisperse challenge particles



Key

1 air or N_2
2 flow controller
3 furnace
4 silver
5 flow compensation through HEPA filter
6 neutralizer
7 DEMC
8 CPC
9 filter medium holder
10 HEPA filter on the exhaust line
11 vacuum

Figure 2 — Test setup for polydisperse challenge particles

6.2 Specifications of setup

6.2.1 Aerosol generation system

The aerosol generation system is described in 5.3.

6.2.2 Tubing

Tubes shall be made of electrically conductive material (stainless steel, carbon-embedded silicon tubing, etc.) in order to minimize particle losses due to electrostatic deposition. Furthermore, the tubing length shall be minimized so as to minimize particle losses due to diffusion. The upstream and downstream sample lines shall be nominally identical in geometry and material.

6.2.3 DEMC

6.2.3.1 Principles and specifications

The DMAS consists primarily of a bipolar charger to neutralize the charges on particles, a controller to control flows and high-voltage, a DEMC (see Figure 3) which separates particles based on their electrical mobilities, a particle detector, interconnecting plumbing, a computer and suitable software.

The DEMC shall be able to classify particles in the size range of 3 nm to 30 nm and fulfil the qualification procedure described in 7.2. In case of the unipolar charger-based instrument, the manufacturer shall be contacted for suitable size range, in order to avoid errors due to multiple charge effect. The losses of the smallest particles due to diffusion within the challenge range shall be considered as well.

NOTE For more information see ISO 15900.

DEMC principles are as follows.

Particles are introduced at the circumference of a hollow tube. A radial electric field is maintained across the outer walls of this tube and a central electrode. As the charged particles flow through the tube, they are attracted towards the central electrode due to the electric field. These are removed through openings in the central electrode.

Small particles require weak electric fields to move them towards the central electrode. Larger particles require stronger fields. By adjusting the electric field, particles of a known size are attracted towards the opening in the central rod and are removed for measurements. Thus, particles with a narrow range of sizes can be extracted for each voltage setting. The narrowness is mainly determined by the geometry and uniformity of air flow in the device. By stepping through a range of voltages or electric field strengths, the number of particles in different sizes in the sample can be measured and the particle size distribution of the sample determined.

Alternatively, since the DEMC separates particles according to their electrical mobilities, if one knows the number of charges on a particle, it can be used to separate monodisperse particles from a polydisperse aerosol.

In this measurement method test particles are first generated and then sent through a neutralizer. Afterwards, the test particles have the Boltzmann equilibrium charge distribution. In this case the singly charged particles represent the largest fraction of the charged particles (see the details in 7.3.2). In addition the size distribution can be controlled so that the target monodisperse particle size is on the right side of the mode of particle size distribution (see the details in 8.2.13). Under these carefully controlled conditions it is possible to use a DEMC to classify monodisperse particles in the range of 3 nm to 30 nm. (See ISO 15900 for more details.)

A DEMC suitable for the prescribed methods in this document shall be able to separate and provide monodisperse particles in the size range from 3 nm to 30 nm with a geometric standard deviation less than 1,10. In general, the ratio of the sheath flow rate to the aerosol flow rate into the DEMC determines the sizing resolution of the DEMC. A higher ratio provides more accurate sizing and avoids excessive diffusional broadening of the particle size distribution so that better monodispersity of the aerosol exiting the DEMC is achieved (see Reference [7]). Prescribing specifications for suitable devices are beyond the scope of this document.

NOTE For more information on DEMC principles, see ISO 15900.