
**Nanotechnologies — Air filter media
containing polymeric nanofibres —
Specification of characteristics and
measurement methods**

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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.

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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Air filter media play an important role in the performance and efficiency of different types of air filters. In this respect, most air filter media take advantage of nonwovens to separate solid or liquid particles. Air filter media have a wide range of applications such as gas turbine inlet air, industrial dust collectors, respiratory masks, personal protective equipment, heating, ventilation and air conditioning systems, cleanrooms, etc.

In recent years, air filter media containing nanofibres have been commercialized and widely used by different industries due to their high filtration efficiency along with a low pressure drop created by the slip flow effect^[6]. Air filter media containing nanofibres are normally produced by depositing one or more types of polymer-based nanofibres directly on the surface of a suitable porous substrate during spinning.

Since the diameter of nanofibres is significantly smaller than that of conventional microfibrils employed in filters, it offers a higher chance of inertial impaction and interception, i.e. a more optimum filtration efficiency. The slip flow also results in a reduced pressure drop and more contaminants passing near the surface of the nanofibres. Hence, the inertial impaction and interception efficiencies rise. As a result, the filtration capability of the nanofibres layer increases for the same pressure drop as compared with a conventional fibre layer. Additionally, the very high surface area of nanofibres facilitates the adsorption of contaminants from the air. All these desirable features have led to a wide range of air filter media containing nanofibres being used for air filtration applications^{[6][7][8]}.

Different techniques such as electrospinning, force spinning and other methods have been used to produce air filter media containing nanofibres. The deposited nanofibres form a web-like nonwoven layer on the surface of the substrate. Nanofibres can exhibit different crystalline structures, morphology and diameter. The surface area and crossed-fibres porosity of the formed nonwoven layer is mainly affected by the nanofibre diameter as well as the morphology. Polymeric nanofibres such as polyamide, polyvinylidene fluoride (PVDF), polyacrylonitrile (PAN) and polyurethane (PU) are normally used for air filter media. Nanofibres can be deposited on different kinds of woven and nonwoven substrates. [Annex A](#) shows a schematic of the cross-section of an air filter medium (see [Figure A.1](#)) and SEM images relating to the morphology of the nanofibres (see [Figures A.2](#) and [A.3](#)).

This document facilitates the communication between sellers and buyers and supports the growing trade of this new class of air filter media.

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Nanotechnologies — Air filter media containing polymeric nanofibres — Specification of characteristics and measurement methods

1 Scope

This document specifies the characteristics to be measured of air filter media containing polymeric nanofibres on the surfaces of a substrate. It also describes measurement methods for determining the individual characteristics.

This document does not cover characteristics specific for health and safety issues.

NOTE The properties/performance assessment of air filter media related to applications requires the use of relevant published standards. The physical properties and performances of filtration media, such as pressure drop, and the particle removal efficiency of air filter media are measured with test methods suitable for specific applications.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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/>

3.1

air filter filter

device for separating solid or liquid *particles* (3.6) or gaseous contaminant from an air stream passing through the device

Note 1 to entry: The device is generally formed of a layer or layers of porous, fibrous or granular material.

Note 2 to entry: Air being cleaned by a filter must pass through the filter, whereas an air cleaner can reduce air contamination by any method.

[SOURCE: ISO 29464:2017, 3.1.16, modified — The preferred term has been changed to “air filter”.]

3.2

air filter medium

porous permeable material employed in *filtration* (3.3) within which the filtrate is trapped or deposited

Note 1 to entry: The filter medium is composed of a *nanofibre* (3.5) layer(s) and *substrate* (3.7).

[SOURCE: ISO 9912-1:2004, 2.27, modified — “air” has been added to the term and Note 1 to entry has been added.]

3.3

filtration

separation of contaminants from a fluid stream in which they are suspended through retention of the contaminants (by extension, also the whole of the activities involved in the construction and commissioning of a filter installation)

[SOURCE: ISO 29464:2011, 3.5.29]

3.4

nanoscale

length range approximately from 1 nm to 100 nm

Note 1 to entry: Properties that are not extrapolations from larger sizes are predominantly exhibited in this length range.

[SOURCE: ISO/TS 80004-1:2015, 2.1]

3.5

nanofibre

nano-object with two external dimensions in the *nanoscale* (3.4) and the third dimension significantly larger

Note 1 to entry: The largest external dimension is not necessarily in the nanoscale.

Note 2 to entry: The terms nanofibril and nanofilament can also be used.

Note 3 to entry: If the dimensions differ significantly (typically by more than 3 times), terms such as nanofibre or nanoplate may be preferred to the term nanoparticle.

[SOURCE: ISO/TS 80004-2:2015, 4.5]

3.6

particle

minute piece of matter with defined physical boundaries

Note 1 to entry: A physical boundary can also be described as an interface.

Note 2 to entry: A particle can move as a unit.

Note 3 to entry: This general particle definition applies to nano-objects.

[SOURCE: ISO/TS 80004-2:2015, 3.1]

3.7

substrate

base layer for depositing *nanofibres* (3.5) on the surface

4 Abbreviated terms

AFM	atomic force microscopy
FESEM	field emission scanning electron microscopy
FTIR	Fourier transform infrared
IR	infrared
SEM	scanning electron microscopy
STM	scanning tunnelling microscopy

TEM	transmission electron microscopy
TGA	thermo gravimetric analysis
UV-Vis	ultraviolet-visible
XRD	X-ray diffraction

5 Characteristics to be measured and their measurement methods

5.1 General

This clause provides both mandatory and optional characteristics to be measured of air filter media containing nanofibres and their measurement methods. The purpose, definition and measurement methods are described for each characteristic in the following individual subclauses.

For all measurements of the characteristics, pieces of the air filter media sample combining a nanofibre layer(s) and a substrate are used as test specimens.

5.2 Mandatory and optional characteristics and their measurement methods

The characteristics of air filter media containing nanofibres listed in Table 1 shall be measured. The relevant measurement methods listed in Table 1 should be taken for the determination of the individual characteristics. The other listed measurement methods can be also used. The characteristic measurements listed in Table 1 shall be done on a nanofibre layer deposited on the substrate and reported in the example report format shown in [Annex B](#).

Table 1 — Mandatory characteristics to be measured and their measurement method(s)

Characteristics	Relevant measurement method(s)	Other measurement method(s)
Fibre diameter	SEM or FESEM	TEM
Morphology	SEM or FESEM	TEM, AFM or STM

The optional characteristics of air filter media containing nanofibres listed in Table 2 should be measured. The relevant measurement methods listed in Table 2 should be taken for determination of the individual characteristics. The other measurement methods listed in Table 2 can be also used. Measurement of the characteristics listed in Table 2 should be done for air filter media with nanofibres.

Table 2 — Optional characteristics to be measured and their measurement method(s)

Characteristics	Relevant measurement method(s)	Other measurement methods
Specific surface area	Gas adsorption method	—
Chemical composition content	FTIR spectroscopy and/or UV-Vis spectrophotometry	Raman spectroscopy, nuclear magnetic resonance, energy dispersive X-ray spectroscopy, gas chromatography, high performance liquid chromatography
Thermal stability	TGA	—
Crystalline structure	XRD	TEM or SAED
Crystallinity	XRD	DSC
Mean crystallite size	XRD	SEM or TEM

5.3 Descriptions on characteristics and their measurement methods

5.3.1 General

Descriptions of the characteristics as well as the measurement methods listed in Tables 1 and 2 are presented in [5.3.2](#) to [5.3.9](#).

5.3.2 Fibre diameter

The diameter of nanofibres deposited on the surface of substrate is one of the most basic characteristics affecting air filter performance.

There can be other fibres in addition to nanofibres of which the diameters are larger than 100 nm in the nanofibre layer of an air filter medium sample. The images obtained by microscopy are analysed to measure the diameters both of nanofibres and other fibres deposited on the substrate.

The diameter of a fibre is the distance on a two-dimensional image between the two edges on a cross-sectional line orthogonal to the longitudinal direction. A fibre may have bends and branches. One datum of diameter is obtained for each fibre. When the diameter varies along the fibre axis on an image, the largest diameter is measured and recorded.

The target fibres shall be representative of the fibres deposited on a substrate, i.e. all types of fibres on an image shall be equally selected. It is recommended to obtain about 100 diameter data points for an air filter medium sample^{[9][10]}.

SEM or FESEM should be used to provide the images. If SEM or FESEM is not available, TEM may be also used. The measurement results shall be displayed as a histogram of the number of fibres versus diameter at an appropriate interval of diameter. Also, the average (median) and standard deviation of diameter data shall be calculated and expressed in a unit of nm.

NOTE The measurement results can be qualitative with increased uncertainty when the observed microscopic images lack the representativeness of sample.

5.3.3 Morphology

The presence of desired nanofibres deposited on the surface of substrate can be confirmed by microscopic images. Different types of morphologies could affect the final performance of the air filter media containing nanofibres^{[9][11][12][13]}. Therefore, the determination of this qualitative characteristic provides important information. Morphology refers to the orientation, structure and shape of nanofibres and other solid objects deposited on the surface of substrate. [Annex A](#) provides example images of air filter media morphologies. Microscopic images of the top surface of the nanofibre layer should be obtained by SEM or FESEM at appropriate magnifications, providing that the target nanofibres can be clearly observed. When SEM and FESEM are not available, TEM, AFM or STM may be used. The magnification scale shall be shown on each picture. The number of microscopic images to be taken may be agreed between the seller and the buyer.

5.3.4 Specific surface area

The specific surface area of an air filter medium sample that contains nanofibres on the substrate is an important characteristic for air filter efficiency. It is defined as the absolute surface area of a test specimen divided by the test specimen mass. The test specimen for measurements is a piece of air filter medium sample.

The specific surface area of air filter media containing nanofibres should be determined using a gas adsorption method, see ISO 9277. This method depends on the measurement of the volume of gas adsorbed by the surface of the air filter media containing nanofibres^[14].

NOTE The reported specific surface area of the air filter media is a composite of the nanofibres and substrate, and can be substantially less than a sample of pure nanofibres.

5.3.5 Chemical composition content

The chemical composition content of polymers used to spin the fibres (mainly type and mass percentages, in % mass fraction, and impurities) can affect some of the nanofibre properties such as water repellancy, dimensional stability, flame retardancy, moisture sorption, breaking tenacity, chemical reactivity, etc^[15].

The reported chemical composition, considering any blending of polymers, may be based on the material specification or material safety datasheet provided by the polymer supplier to the air filter media manufacturer.

The chemical composition content of polymers can be measured by IR or UV-Vis absorption spectrophotometry. IR spectrophotometry is the subset of spectrophotometry that deals with the infrared region of the electromagnetic spectrum, which provides information regarding the type of chemical bonds and functional groups. In this respect, collected samples are mixed with KBr to make pellets. FTIR spectra in the absorbance mode are recorded using FTIR spectrometer. The obtained spectra are then analysed using IR software.

Ultraviolet and visible (UV-Vis) absorption spectrophotometry is the measurement of the attenuation of a beam of light after it passes through a sample. Absorption measurement can be at a single wavelength or over an extended spectral range (200 nm to 800 nm). Such data also provide information to determine the nanofibres composition. When FTIR spectroscopy or UV-Vis spectrophotometry is not available, other measurement methods including Raman spectroscopy, nuclear magnetic resonance, energy dispersive X-ray spectrometry, gas chromatography or high performance liquid chromatography may be used. The measurement results of chemical composition content shall be expressed in % mass fraction.

5.3.6 Thermal stability

The thermal stability indicates the quality and ability of the substances present in a filter medium to resist irreversible change in their chemical or physical structure by decomposition or depolymerisation when subjected to high temperatures. The measurement of thermal stability of a filter medium test specimen refers to the mass loss of the specimen in dried form during heating to a sufficiently high temperature. The thermal stability should be measured by TGA. The mass loss of test specimen should be measured for both dynamic and isothermal conditions. The measurement results should be expressed by a thermogravimetric curve composed of the sample mass and temperature with their interpretation.

Considering that the results depend on many experimental and instrumental variables, relevant measurement conditions are also reported including the atmosphere (e.g. air, N₂, O₂) and its flow rate, the method of sample drying (e.g. freeze-dried or air-dried) and the temperature programme used [e.g. heating ramp rate(s) and/or isothermal temperature(s)].

5.3.7 Crystalline structure

The nanofibres may be composed of amorphous or crystalline phases, which could have a significant effect on the physical properties. The spinning process could affect the crystalline structure of the polymer; therefore, analysis of the nanofibres coating the substrate is required. The crystalline structure is defined as a structure of molecules or atoms that are held together in an ordered three-dimensional arrangement. The XRD measurement is applied to analyse the crystalline structure of nanofibres on the substrate. The measurement results should be expressed by XRD patterns composed of 2θ (°) and intensity with their interpretation.

5.3.8 Crystallinity

Crystallinity affects the moisture absorption of air filter media. Usually moisture absorption happens in the amorphous regions of a fibre but not in the crystalline regions^[12]. Crystallinity refers to the presence of three-dimensional order at the level of molecular dimensions. The measurement results of crystallinity are usually expressed as a percentage of the volume/mass of the material that is

crystalline. The crystallinity can be measured by using XRD or DSC. DSC measures the heat flow into or from the air filter medium sample as it is heated, cooled or under isothermal condition.

5.3.9 Crystallite size

Crystallite size affects the crystalline structure and, therefore, the mechanical properties and moisture absorption. Crystallite is a small or even microscopic crystal. Crystallite size is usually measured by XRD. The mean crystallite size can be measured by using Scherrer's equation.

6 Reporting

6.1 The report shall include the items given in [6.2](#) and [6.3](#).

6.2 General information:

- sample name;
- manufacturer's name;
- lot number;
- storage conditions prior to testing;
- type of substrate.

6.3 Mandatory characteristics listed in Table 1 and their measurement results:

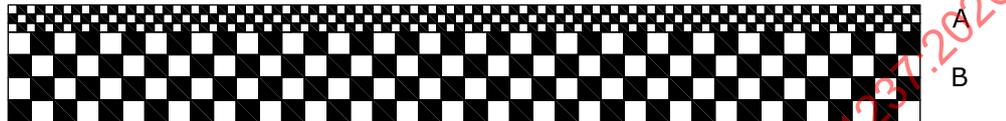
- characteristics measured;
- measurement method taken;
- name of testing laboratory;
- measurement date;
- measurement results.

[Tables B.1](#) and [B.2](#) give example formats for reporting the mandatory characteristics listed in [Table 1](#). [Table C.1](#) gives an example format for reporting the optional characteristics listed in [Table 2](#) that can be provided subject to agreement between the seller and the buyer.

Annex A (informative)

Typical structures of air filter media containing nanofibres

This annex provides a schematic of the cross-section of an air filter medium in [Figure A.1](#). SEM images relating to the morphology of the nanofibres are shown in [Figures A.2](#) to [A.3](#).

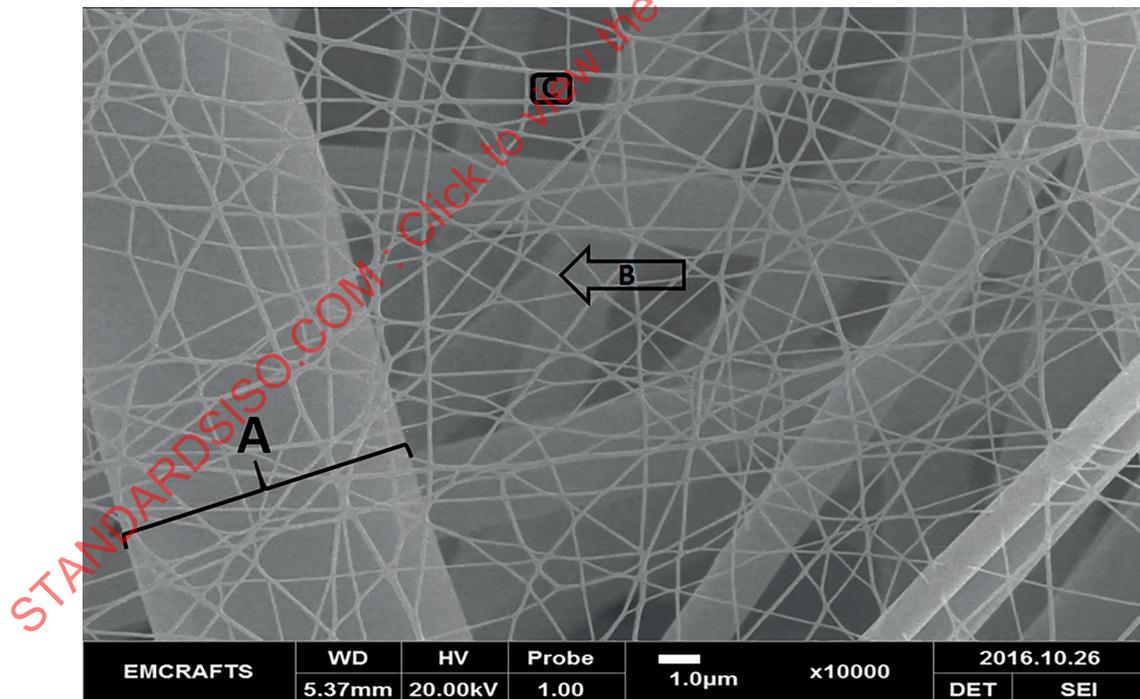


Key

- A nanofibres layer
- B substrate layer

Figure A.1 — Schematic of cross-section of an air filter medium containing nanofibres

[Figure A.2](#) illustrates a typical structure of the air filter media containing nanofibres prepared by depositing polyamide nanofibres on the surface of a meltblown polypropylene substrate.

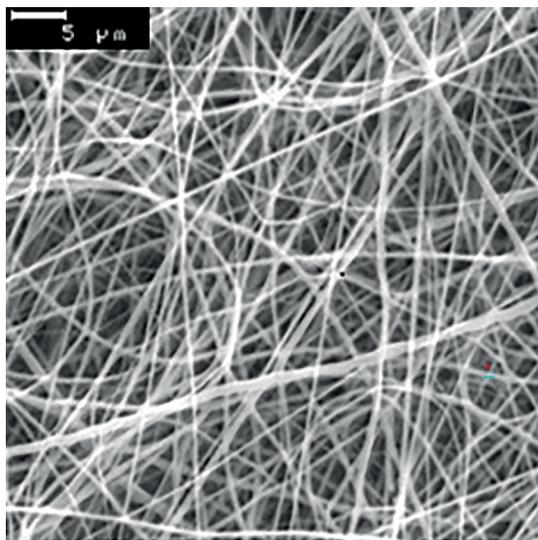


Key

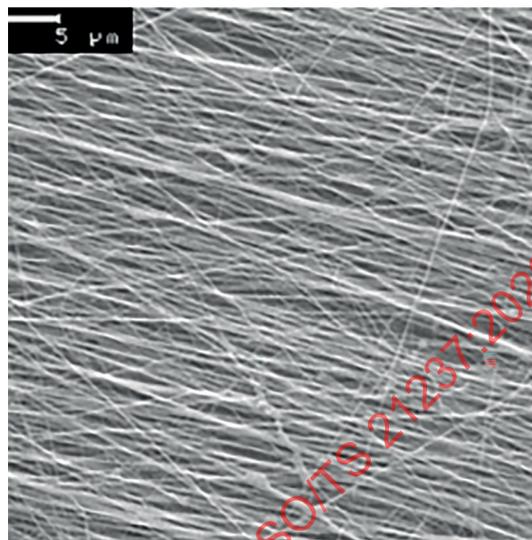
- A microfibre
- B nanofibre
- C pore

Figure A.2 — Demonstration of air filter media containing nanofibres

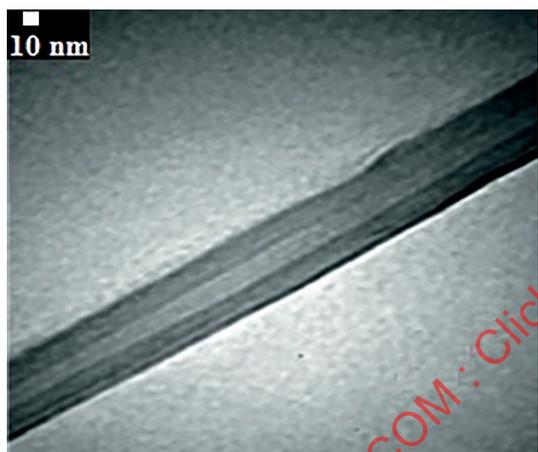
Figure A.3 illustrates types of nanofibres morphology and texture: random fibre-like, oriented fibre-like, core-shell fibre-like and beaded fibre-like.



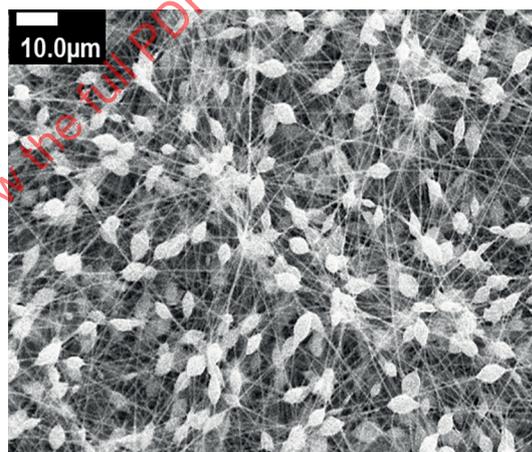
a) Random fibre-like



b) Oriented fibre-like



c) Core-shell fibre-like



d) Beaded fibre-like

Figure A.3 — Different morphologies and textures of nanofibres