Technical Specification

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Nanotechnologies — Method to quantify air concentrations of carbon black and amorphous silica in the nanoparticle size range in a mixed dust manufacturing environment

Nanotechnologies — Méthode de quantification des concentrations dans l'air de noir de carbone et de silice amorphe à l'échelle nanométrique dans un environnement de fabrication industrielle contenant des mélanges de poussières

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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 document 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*.

This second edition cancels and replaces the first edition (ISO/TS 21361:2019) which has been technically revised.

The main changes are as follows:

— references have been updated;

— minor clarifications have been made to the text.

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

Nanomaterials are widely used in industrial settings in the manufacture of consumer products. Carbon black and amorphous silica are commonly used in consumer products, such as rubber products, insulating materials, and others. Although these materials typically exist as agglomerates in dimensions larger than the nanoscale, there is also the potential for worker exposure to these materials in the nanoscale size range. In spite of the widespread use of nanomaterials such as these, quantification of air concentrations of specific nanomaterials in mixed dust settings, such as a manufacturing environment, has been challenging to date and has been identified as a hindrance to the development of nano-specific occupational exposure limits (see References [3], [4] and [6]). This method outlines a technique whereby particles of carbon black and amorphous silica can be identified, distinguished, and quantified (in terms of air concentrations) by size in such manufacturing settings. It is anticipated that although this method is specific to carbon black and amorphous silica, the general principles of the method can be applied to many materials in a variety of manufacturing environments. This method advances beyond existing techniques for analysis in that it provides quantitative information regarding exposure to specific materials by size; many other methods provide quantitative information on nanoparticle exposures that are incapable of differentiating by material type. This method includes both a defined methodology for collecting air samples in the manufacturing settings as well as a methodology for analysing the sample to obtain appropriate information for quantifying air concentration of the materials of interest. Application of this methodology has recently been published in the peer-reviewed literature (see Reference [5]).

This document specifies a method to quantify and identify particles of either carbon black or amorphous silica, or both, in air samples collected in a mixed dust, industrial, manufacturing environment. It describes air sample collection and the characterization of the particles in the air samples by both particle size and elemental composition. The method is defined for air samples collected with an electrical low pressure cascade impactor (ELPCI). However, the method is suitable for sampling in manufacturing environments where there are a variety of particle types contributing to the overall atmosphere. The particles in the air sample are collected in the various stages of a cascade impactor with cut-offs for median particle size between 6 nm and 10 µm. This impactor determines the number particle size distribution in real-time based on the particle aerodynamic diameter. Particles collected on each stage are collected for off-line analysis using transmission electron microscopy (TEM) and energy dispersive spectrometry (EDS) to identify amorphous silica and carbon black particles. The TEM-EDS measurement provides the elemental composition and source of the particles in each stage. Scanning electron microscopy (SEM) is also an option to TEM in the electron microscopy/dispersive spectrometry combination. While TEM and SEM can also be used to measure particle size directly (see References [8] and [9]), in this method the concentration of particles of a specific nanomaterial in a specific size range (#/cm^3) is given by the product of the total particle count for size range (#/cm^3) obtained from the cascade impactor and the fraction of particles identified as the specific material of interest (e.g. carbon black or amorphous silica) from the TEM-EDS results. Though this technique is described for carbon black and amorphous silica, the technique can be applied to the measurement of other particle types, provided they are in the size range and can be observed by TEM/SEM and chemically characterized by EDS.

At this time, this methodology represents one of the methods available to quantify chemical-specific exposures to nanoparticles by size with this degree of sensitivity. Many of the other existing methods that can speciate and quantify chemical exposure in this size range are mass-based, and thus are limited by mass-based detection limits that are high when compared to the mass of particles in this size range. Other air sampling methods, like the microorifice uniform deposit impactor (MOUDI, see Reference [1]) can be amenable to the techniques described herein but none of them have been evaluated or validated for this purpose and are not included in this document. This methodology offers increased sensitivity for quantification of exposure to specific particle types in the nanoscale when such an interest arises. The sample collection methods and analysis are more labor intensive and more involved than practical for most industrial hygiene risk assessment uses. This methodology can be implemented as a higher tier step in an occupational exposure assessment sampling strategy for nanomaterials, particularly in the event that hot spots for exposure are identified using other methods and there is an interest in understanding the nature of the exposure. Results from this analysis can be used to compare health benchmarks, as they become available, to understand potential health risk for workers and help in selecting appropriate personal protective equipment (PPE).

Nanotechnologies — Method to quantify air concentrations of carbon black and amorphous silica in the nanoparticle size range in a mixed dust manufacturing environment

1 Scope

This document specifies a method to quantify and identify air concentration (number of particles/cm^3) of particles of either carbon black or amorphous silica, or both, by size in air samples collected in a mixed dust, industrial, manufacturing environment.

This method is applicable to air samples collected with an electrical low pressure cascade impactor (ELPCI) for sampling in manufacturing environments where there are a variety of particle types contributing to the overall atmosphere. This method is applicable only to environments with chemically and physically distinct particles contributing to aerosols or where confounders can be controlled (e.g. diesel sources).

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 10312, *Ambient air — Determination of asbestos fibres — Direct transfer transmission electron microscopy method*

ISO 4225:2020, *Air quality — General aspects — Vocabulary*

ISO 80004-1:2023, *Nanotechnologies – Vocabulary — Part 1: Core vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4225 and ISO 80004-1 and the following apply.

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

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

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

3.1
particle aerodynamic diameter
diameter of a sphere of density 1 g m^{-3} with the same settling velocity in calm air as the particle, under the prevailing conditions of temperature, pressure and relative humidity

[SOURCE: ISO/TS 20593:2017, 3.10, modified — "g/cm^3" has been replaced by "g m^{-3}", the phrase "terminal velocity due to gravitational force" has been replaced with "settling velocity".]

3.2
cascade impactor
sampler using impaction, that can simultaneously collect particulate matter separately in a number of size ranges, based on particle momentum

[SOURCE: ISO 4225:2020, 3.3.2.3]

3.3
cut-off
size of airborne particle (aerodynamic diameter) for which the sampling efficiency is 50 %

3.4
nanoscale
length range approximately from 1 nm to 100 nm

[SOURCE: ISO/TS 80004-1:2023, 3.1.1]

3.5
nanomaterial
material with any external dimension in the nanoscale or having internal structure or surface structure in the nanoscale

[SOURCE: ISO/TS 80004-1: 2023, 3.1.4]

3.6
particle
minute piece of matter with defined physical boundaries

[SOURCE: ISO 14644-1:2015, 3.2.1]

3.7
sampling time
interval of time over which a single sample is taken

[SOURCE: ISO/TS 20593:2017, 3.15]

4 Abbreviations

EDS	energy dispersive spectroscopy
ELPCI	electrical low pressure cascade impactor
HEPA	high efficiency particulate arrestance
LOD	limit of detection
PM10	airborne particles with an aerodynamic diameter less than 10 µm
SEM	scanning electron microscopy
STEM	scanning transmission electron microscopes
TEM	transmission electron microscopy

5 Principle

Air sampling is conducted using an ELPCI. The ELPCI is a cascade impactor with multiple stages representing different size bins, distinguished based on aerodynamic diameter. The cascade impactor is connected to a vacuum pump, which pulls air (and subsequently airborne particles) into the impactor. As particles enter the cascade impactor, they are charged and then subsequently separated by aerodynamic size onto different stages. As the particles come into contact with the surface of their appropriate stage, they transmit an electrical current to the ELPCI. This electrical current is then converted into a particle count for that stage, thus permitting the ELPCI to conduct real-time particle counting. This particle count is reported as the number of particles per cm^3 of air. In addition to the particle counting function, the ELPCI also allows for collection of samples on each stage, using a 25 mm polycarbonate substrate.

Following completion of air sampling, these substrates are analysed via TEM to identify the particles on each stage by morphology coupled with EDS to identify the particles on each stage by elemental composition. A minimum of 100 distinct particles per stage are analysed using TEM-EDS to identify the particle type, based on the chemical signature of the particles. Carbon black, made up primarily of elemental carbon, elicits a strong carbon signal (and absence of other signals) on the EDS, whereas silica (SiO_2) elicits a strong silicon and oxygen signal. These profiles are used to designate particles as carbon black or amorphous silica. Where there are potential confounders present in the sample, such as soot (for carbon black) or crystalline silica (for amorphous silica), either particle morphology or diffraction pattern, or both must be relied upon to identify the particles. Morphology is the main attribute that distinguishes carbon black from soot, whereas morphology and diffraction can be used to distinguish crystalline silica from amorphous. Furthermore, comparison to the morphology of the source materials (e.g. carbon black or amorphous silica) can be evaluated under TEM to ensure appropriate assignation of the particle type. The relative proportion of particles (e.g. number of particles by type out of 100 total particles identified per size fraction) identified by type of particle (carbon black, amorphous silica, or other) is the key outcome of this analysis.

To determine air concentration of the materials of interest (e.g. carbon black or amorphous silica), the proportion of particles identified by type is multiplied by the total number of particles in each size bin determined during the real-time air sampling. The outcome of this analysis is the air concentration of each material type by size in number of particles/cm^3.

Though the method described herein will specifically focus on the analysis of carbon black and amorphous silica, this methodology is transferrable to other materials, provided they either have a unique signal under EDS or are distinguishable via morphology under TEM, or both.

6 Reagents

During the analysis, use only reagents of recognised analytical grade. All agents listed are required to execute the transfer of the polycarbonate substrate content onto a TEM grid in accordance with ISO 10312. No additional reagents are required for execution of this specification.

WARNING — It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

6.1 Water, fibre-free

A supply of freshly distilled, fibre-free water, or another source of fibre-free, pyrogen-free water shall be used.

6.2 Chloroform, analytical grade.

Prepared by distillation in glassware, preserved with 1 % (W/V) ethanol; used to dissolve polycarbonate substrate.

6.3 l-Methyl-2-pyrrolidone.

Used to dissolve polycarbonate substrate.

6.4 Dimethylformamide.

Used to dissolve polycarbonate substrate.

6.5 Glacial acetic acid.

Used to dissolve polycarbonate substrate.

6.6 Acetone.

Used to dissolve polycarbonate substrate.

7 Apparatus

7.1 Air sampling — Equipment and consumable supplies

7.1.1 Electrical low cascade pressure impactor (ELPCI)

An ELPCI, consisting of a minimum of 14 size bins, should be used for collection of air samples. Of the size bins, at least 3 should be within the nanoscale, defined as less than 100 nm. The ELPCI should be operated in accordance with the manufacturer's instructions.

An example of measurement capability for a commercially available electrical low pressure impactor operating at a 10 L/min flow rate is presented in Table 1.

Table 1 — Measurement range for commercially available electrical low pressure cascade impactor (ELPCI)

D50 % nm	Di nm	Number min 1/cm^3	Number max 1/cm^3	Mass conc. min µg/m^3	Mass conc. max µg/m^3
10 000	Unavailable	Unavailable	Unavailable	Unavailable	Unavailable
6 800	8 200	0,1	2,40E+04	30	10 000
4 400	5 500	0,1	2,40E+04	10	3 000
2 500	3 300	0,15	5,40E+04	3	1 000
1 600	2 000	0,3	1,10E+05	1,4	450
1 000	1 300	0,5	1,90E+05	0,7	210
640	800	1	3,50E+05	0,3	100
400	510	2	6,40E+05	0,1	50
260	320	3	1,20E+06	0,07	20
170	210	5	2,10E+06	0,03	10
108	140	10	3,70E+06	0,02	5
60	80	20	7,30E+06	0,005	2
30	42	50	1,70E+07	0,002	0,5
17	22	100	3,40E+07	0,001	0,25
6	1	250	8,30E+07	0,000 4	0,13
D50 % = Aerodynamic diameter cut-offs for 50 % efficiency; Di = Geometric mean aerodynamic diameter of stage					

[SOURCE: Reference [2], page 12, adapted with permisision of the authors.]

7.1.2 Limit of detection (LOD)

Based on the table above and the expected volumetric flow rate of the sampling equipment (10 l/min), the practical LODs for both carbon black and amorphous silica (based on a one- second sample) by particle size are found in Table 2.