



BSI Standards Publication

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

---

## National foreword

This Published Document is the UK implementation of ISO/TS 21361:2019.

The UK participation in its preparation was entrusted to Technical Committee NTI/1, Nanotechnologies.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2019  
Published by BSI Standards Limited 2019

ISBN 978 0 580 98785 4

ICS 07.120

**Compliance with a British Standard cannot confer immunity from legal obligations.**

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 30 April 2019.

### Amendments/corrigenda issued since publication

Date	Text affected
------	---------------

---

TECHNICAL  
SPECIFICATION

**ISO/TS**  
**21361**

First edition  
2019-04-17

---

---

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

*Nanotechnologies — Quantification du noir de carbone et de la  
silice amorphe nanométriques en suspension dans l'air en ambiance  
de production*



Reference number  
ISO/TS 21361:2019(E)

© ISO 2019



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2019. Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
copyright@iso.org  
www.iso.org

# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Principle</b> .....	<b>2</b>
<b>5 Abbreviations</b> .....	<b>3</b>
<b>6 Reagents</b> .....	<b>3</b>
6.1 Water, fibre-free.....	3
6.2 Chloroform, analytical grade.....	3
6.3 1-Methyl-2-pyrrolidone.....	3
6.4 Dimethylformamide.....	3
6.5 Glacial acetic acid.....	3
6.6 Acetone.....	3
<b>7 Apparatus</b> .....	<b>4</b>
7.1 Air sampling — Equipment and consumable supplies.....	4
7.1.1 Electrical low cascade pressure impactor (ELPCI).....	4
7.1.2 Limit of Detection.....	4
7.1.3 Real-time aerosol monitor.....	5
7.1.4 Vacuum pump.....	5
7.1.5 Polycarbonate substrate.....	5
7.1.6 Hydrocarbon grease.....	5
7.2 Analytical/microscopy laboratories.....	6
7.3 Sample analysis — Equipment and consumables.....	6
7.3.1 Transmission electron microscope (TEM).....	6
7.3.2 Energy dispersive spectrometry (EDS).....	6
7.3.3 Computer.....	6
7.3.4 TEM Grid.....	6
<b>8 Air sample collection</b> .....	<b>6</b>
8.1 Preparation for sampling.....	6
8.2 Determination of sampling time.....	7
8.3 Sample collection procedure.....	7
<b>9 Procedure for analysis</b> .....	<b>7</b>
9.1 General.....	7
9.2 Preparation of substrates.....	8
9.3 Sample analysis.....	8
9.3.1 Instrument conditions.....	8
9.3.2 Data collection.....	8
9.4 Calculation of air concentration.....	9
<b>10 Uncertainties and performance criteria</b> .....	<b>10</b>
10.1 Particle counting with the ELPCI.....	10
10.2 Particle analysis with TEM/EDS.....	11
<b>11 Test report</b> .....	<b>11</b>
<b>Annex A (informative) Case study overview</b> .....	<b>12</b>
<b>Bibliography</b> .....	<b>13</b>

## 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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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](http://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](http://www.iso.org/members.html).

## Introduction

Nanomaterials are widely used in industrial settings in the manufacture of consumer products. Carbon black and/or 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 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, have been challenging to date and has been identified as a hindrance to the development of nanomaterial-specific occupational exposure limits (Gordon, et al. 2014; Hansen, et al. 2012; van Broekhuizen, et al. 2013). This method outlines a technique whereby particles 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 analyzing 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 (Kreider, et al. 2015).

This document will provide guidelines to quantify and identify particles carbon black and/or amorphous silica in air samples collected in a mixed dust industrial manufacturing environment. The guidelines describe air sample collection and the characterization of the particles in the air samples by both particle size and elemental composition. 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. The concentration of particles of a specific nanomaterial in a given size range (#/cm<sup>3</sup>) is given by the product of the total particle count for size range (#/cm<sup>3</sup>), 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 of 6 nm to 2,5 µm and can be observed by TEM/SEM and chemically characterized by EDS.

At this time, this methodology represents the 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 appreciate 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. Furthermore, although other sampling methods may be amenable to the techniques described herein, none have been evaluated or validated for this purpose. Therefore, this methodology offers increased sensitivity for quantification of exposure to specific particle types in the nanoscale when such an interest arises. This methodology could be implemented as a higher tier step in an occupational exposure assessment sampling strategy for nanomaterials, particularly in the event 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 to health benchmarks, as they become available, to understand potential health risk of workers. In addition, it could be useful in selecting appropriate personal protective equipment (PPE) at a very early stage of the manufacturing process, when required.

Currently in preview, click buy full version

# 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 provides guidelines to quantify and identify air concentration (number of particles/cm<sup>3</sup>) of particles of carbon black and/or amorphous silica by size in air samples collected in a mixed dust industrial manufacturing environment.

The method is defined for air samples collected with an electrical low pressure cascade impactor (ELPCI) on a 25 mm polycarbonate substrate. The method is suitable 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 when confounders can be controlled (e.g. diesel sources). Other sampling methods can also be suitable, though this document is limited to describing methods associated with the electrical low pressure cascade impactor.

Samples collected with the electrical low pressure cascade impactor are analyzed via TEM and EDS to for particle morphology and elemental composition, respectively, to permit identification of particles by type. This information is then used, in conjunction with particle concentration by size range, as determined by the electrical low pressure cascade impactor, to determine concentration of the materials of interest by size.

## 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 amendment) applies.

ISO 10312, *Ambient Air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method*

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

ISO/TS 80004-1, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO 22309, *Microbeam analysis — Quantitative analysis using energy-dispersive spectrometry (EDS) for elements with an atomic number of 11 (Na) or above*

ISO/TS 10790, *Nanotechnologies — Characterization of single-wall carbon nanotubes using scanning electron microscopy and energy dispersive X-ray spectrometry analysis*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4225 and ISO/TS 80004-1 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/>