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Nanotechnology — Nanoparticles in powder form — Characteristics and measurements

Nanotechnologies — Nanoparticules sous forme de poudre — Caractéristiques et mesurages

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Foreword

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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).

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).

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.

This document was prepared by Technical Committee ISO/TC 229 *Nanotechnologies,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 352, *Nanotechnologies*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This first edition cancels and replaces ISO/TS 17200:2013, which has been technically revised. The main changes compared with the previous edition are as follows:

— ISO documents for primary particle size measurements by electron microscope have been updated;

— the descriptions of characteristics to be measured and their measurement methods based on the purpose of this document have been changed;

— the requirement for crystallite size measurement has been relaxed.

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.

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Introduction

As is commonly noticed for every technology concerned with the development of new materials, and for nanotechnology in particular, communication and mutual understanding of material characteristics are important among consumers, regulators and industries. In the case of nanoparticles, the stakeholders' basic interest is in the characteristics of nanoparticles in a material, i.e. what nanoparticles are present and what is the size distribution of nanoparticles. Such identification of nanoparticles in a material can be facilitated by the development of standards for nanoparticle characteristics and their measurement methods.

This document provides standardized methods for identifying and characterizing nanoparticles in powder form. Other standards have been developed for specific materials, i.e. ISO/TS 11931 and ISO/TS 11937 for calcium carbonates and titanium dioxides, respectively. This document is generic and may apply to nanoparticles composed generally of metal/metal ion and counter-ion, and to carbon materials (e.g. fullerenes and fullerene derivatives) and polymers (e.g. polystyrene). The applicability of this document includes calcium carbonate and titanium dioxide. This document is applicable to both coated and uncoated nanoparticles.

This document facilitates communication and mutual understanding among consumers, regulators and industries about the characteristics of nanoparticles. It supports consumers in purchasing and using nanoparticle-containing products, regulators in establishing legislative frameworks, and industries in setting up voluntary risk control systems.

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Nanotechnology — Nanoparticles in powder form — Characteristics and measurements

1 Scope

This document specifies the fundamental characteristics to be measured of a sample of engineered nanoparticles in powder form to determine the size, the chemical content and the surface area. This document also specifies measurement methods for determining each of the characteristics.

It is intended to facilitate communication among consumers, regulators and industries with the necessary characteristics.

It excludes characteristics that pertain to specific industrial applications of nanoparticles in powder form and detailed measurement protocols, as well as characteristics related to health, safety and environmental issues.

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 9276-1, *Representation of results of particle size analysis — Part 1: Graphical representation*

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

ISO/TS 80004-2, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1, ISO/TS 80004-2 and the following 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
area equivalent diameter
diameter of a circle having the same area as the projected image of the particle

[SOURCE: ISO 13322-1:2014, 3.1.1, modified — Note 1 to entry has been deleted.]

3.2
crystallite
small crystalline domain in the material

3.3
engineered nanoparticle
nanoparticle (3.6) designed for specific purpose or function

Note 1 to entry: In this document, the powder material containing engineered nanoparticles and provided for the measurement is called the "nanoparticles sample" and may be abbreviated to "sample".

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[SOURCE: ISO/TS 80004-1:2015, 2.8, modified — "nanoparticle" has replaced "nanomaterial" in the definition and Note 1 to entry has been added.]

3.4
engineered nanoparticles sample
sample in powder form that contains *engineered nanoparticles* (3.3)

3.5
Feret diameter
distance between two parallel tangents on opposite sides of the image of a particle

[SOURCE: ISO 13322-1:2014, 3.1.5]

3.6
nanoparticle
nano-object with all external dimensions in the nanoscale where the lengths of the longest and the shortest axes of the nano-object do not differ significantly

Note 1 to entry: If the dimensions differ significantly (typically by more than three times), terms such as "nanofibre" or "nanoplate" may be preferred to the term "nanoparticle".

[SOURCE: ISO/TS 80004-2:2015, 4.4]

3.7
particle size distribution
distribution of particles as a function of particle size

3.8
primary particle
original source particle of agglomerates or aggregates or mixture of the two

Note 1 to entry: Constituent particles of agglomerates or aggregates at a certain actual state may be primary particles, but often the constituents are aggregates.

[SOURCE: ISO/TS 80004-2:2015, 3.2, modified — Note 2 to entry has been deleted.]

3.9
scanning electron microscopy
SEM
method that examines and analyses the physical information (such as secondary electron, backscattered electron, absorbed electron and X-ray radiation) obtained by generating electron beams and scanning the surface of the sample in order to determine the structure, composition and topography of the sample

[SOURCE: ISO/TS 80004-6:2013, 3.5.5]

3.10
scanning transmission electron microscopy
STEM
method that produces magnified images or diffraction patterns of the sample by a finely focused electron beam, scanned over the surface and which passes through the sample and interacts with it

Note 1 to entry: Typically uses an electron beam with a diameter of less than 1 nm.

Note 2 to entry: Provides high-resolution imaging of the inner microstructure and the surface of a thin sample (or small particles), as well as the possibility of chemical and structural characterization of micrometre and sub-micrometre domains through evaluation of the X-ray spectra and the electron diffraction pattern.

[SOURCE: ISO/TS 80004-6:2013, 3.5.7]

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3.11
specific surface area
absolute surface area of the sample divided by sample mass

Note 1 to entry: In this document, the absolute surface area is determined by measuring the amount of physically adsorbed gas using the Brunauer–Emmett–Teller method.

[SOURCE: ISO 9277:2010, 3.11, modified — Note 1 to entry has been added.]

3.12
transmission electron microscopy
TEM
method that produces magnified images or diffraction patterns of the specimen by an electron beam which passes through the specimen and interacts with it

[SOURCE: ISO/TS 80004-6:2013, 3.5.6]

3.13
X-ray diffraction
XRD
method to obtain crystallographic information about a sample by observing the diffraction pattern due to an X-ray beam hitting a sample

[SOURCE: ISO/TS 80004-6:2013, 5.2.1, modified — Note 1 to entry has been deleted.]

4 Abbreviated terms

BET	Brunauer–Emmett–Teller
EDX	energy dispersive X–ray spectrometry
ICP–OES	inductively coupled plasma optical emission spectrometry
NMR	nuclear magnetic resonance
SEM	scanning electron microscopy
SIMS	secondary ion mass spectrometry
STEM	scanning transmission electron microscopy
TEM	transmission electron microscopy
TG	thermogravimetry
UV/Vis/NIR	ultraviolet, visible and near infrared absorption spectrophotometry
XPS	X–ray photoelectron spectroscopy
XRD	X–ray diffraction
XRF	X–ray fluorescence spectrometry

5 Characteristics to be measured and their measurement methods

5.1 General

The characteristics of the nanoparticles sample listed in Table 1 shall be measured. The measurement methods listed in Table 1 shall be adopted to determine the characteristics.

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When expressing the measurement results, the units should be either SI units or multiples or sub-multiples of them following ISO 80000-1. The unit symbols “nm” and “g” may be used in place of “10^{-9} m” and ”10^{-3} kg”.

Nanoparticles to which this document can be applied are listed in Annex A.

Table 1 — Characteristics and measurement methods

Characteristic		Measurement method
Chemical/crystalline content	Chemical composition	Chemical analysis (see 5.2)
	Crystallographic components content	XRD method (see 5.3)
Surface area	Specific surface area	Gas adsorption method (see 5.4)
Size	Diameter of primary particle	TEM, SEM or STEM (see 5.5)
	Crystallite size	XRD method (see 5.6)

5.2 Chemical composition

The sample may be composed of certain chemical compounds. The content of composing compounds is the ratio of the mass of the chemical compound in a powder sample to that of the dried whole sample. The content of major compound in the sample shall be measured and the results are usually expressed as % mass fraction.

An appropriate measurement method shall be adopted for the determination of the content. The measurement method can be taken from the following list of measurement methods. The standards listed provide useful measurement protocols for the measurement methods:

— titrimetry;

— gravimetry;

— XRF: ISO 9516-1:2003 and ISO/TS 80004-6:2013;

— ICP-OES: ISO/TS 80004-6:2013;

— NMR: ISO/TS 80004-6:2013;

— XPS: ISO 10810:2019 and ISO/TS 80004-6:2013;

— SIMS: ISO/TS 80004-6:2013;

— EDX: ISO/TS 80004-6:2013;

— UV/Vis/NIR: ISO/TS 10868:2017 and ISO/TS 80004-6:2013;

— TG, see ISO/TS 11308:2020 and ISO/TS 80004-6:2013.

International Standards specifying the measurement protocols for chemical composition of relevant material and relevant measurement method are listed in Bibliography. Other literature describing the measurement methods is also listed in the Bibliography.

Relevant certified reference materials, if available, shall be used for the measurements.

5.3 Crystallographic components content

The sample may be composed of different crystallographic components of a chemical compound. The crystallographic components contents of a chemical compound are the ratios of the mass of the components to that of the whole chemical compound. The crystallographic component contents shall be measured when the sample is composed of different crystallographic components, and the results of individual component contents expressed in the unit of unity or kg/kg.

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The crystallographic components content shall be measured by XRD. Measurement protocols for crystal structure determination are described in EN 13925-1:2003 and JIS K 0131:1996. The wavelength of characteristic X-rays should be referred to a reliable database. The reference value for lattice spacing should be referred to a reliable database or to the certificates of any powder reference materials used.

When the sample does not show a clear diffraction line, for example, in cases of amorphous and polycrystal structure, the crystallographic components content is out of the scope of the characteristics to be measured.

5.4 Specific surface area

The surface area of the sample is correlated to the size of composing particles. The specific surface area of the sample shall be measured and the results expressed in the unit m^2/g or its equivalent.

The specific surface area of the sample shall be measured by the gas adsorption method using the BET method. Certified reference materials for application of the BET method shall be used when they are available. ISO 9277 is applicable for the measurement of specific surface area. ISO 18757 provides some useful detailed information for specific materials.

5.5 Diameter of primary particle

The diameter of the primary particle is either the average of the longest and shortest Feret diameters or the area equivalent diameter of a primary particle based on the outermost dimension identified on a two-dimensional image taken by TEM, SEM or STEM. The applied diameter shall be reported.

The diameter of the primary particle may be obtained by measuring un-agglomerated discrete singular particles or particles comprising the structure within aggregates or agglomerates. The procedure used shall be documented as required in Clause 7.

The result of measurements of particle size distribution of particles shall be expressed in the form of a chart or table taking the numbers of particles at a certain diameter interval on the vertical axis and the diameter of particles on the horizontal axis as specified in ISO 9276-1. It shall be reported which is taken: Feret diameter or area equivalent diameter. Also, the average diameter of the particles shall be taken as the median of data. The diameter of particles should be expressed in the unit m or nm.

The diameter of the primary particle shall be measured by TEM. SEM or STEM can be used when its measurement precision is sufficient for the particles to be measured. References ISO 14488, ISO 14887 and ISO 2859 (all parts) apply to sample preparation or sampling and ISO 13322-1 applies to image processing. The primary particles should be identified by the image processing. The calibration of the scale of the TEM and SEM images should be made using certified reference materials or a database containing well-known dimensions, such as lattice spacing or materials in the microscopic view. The number and size of particles counted should be documented. The number selected will depend on the range of particle size and desired precision.

Standardization for the application of TEM, SEM and STEM to nanoparticle size and particle size distribution measurements is given in ISO 19749 and ISO 21363. They can apply to measurement protocols for the primary particle size and particle size distribution.

The practical interpretation of the definition of primary particle in 3.8 for the application of the measurement method can be dependent on the chemical compound and applied sample preparation related to the dispersing process. Documentation on the sample preparation is essential for reporting.

When an image of the primary particle is invisible in a material by microscopy, e.g. in heavily fused particles in an aggregate, measurement by microscopy should be conducted for the aggregates or agglomerates.

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5.6 Crystallite size

The line width of XRD from crystallite particles depends on the average size of the particles according to the Scherrer formula. The average diameter of the particles shall be measured by XRD and the results expressed in the unit m or nm.

When a powder sample does not show a clear diffraction line, for example, in the case of an amorphous or polycrystalline structure, measurement of crystallite size is inapplicable and can be excluded from the characteristics to be measured. Also, when the primary particle size has been measured at an acceptable accuracy level with the powder sample, it is not necessary to measure the crystallite size.

Measurement protocols applying to the crystallite size are described in EN 13925-1:2003 and JIS K 0131:1996. Usually the same sample preparation and instrumentation can be used for both measurements of crystallite size and crystallographic components content.

6 Sampling and sample preparation

The sample subjected to measurements should be chosen as representative of the parent population of the particles in powder form. ISO 14488 applies to sampling and the sample splitting procedure.

Any influences of the sampling process on the measured characteristics of the nanoparticles should be estimated. Corrections for such influences are applied or appropriate components of uncertainty are incorporated.

NOTE For more general information about sampling procedures, see ISO 28591.

A well-established method for dispersion should be applied in sample processing on the sample, e.g. ISO 14887.

7 Test report

The test report shall include the following information:

a) the information necessary to identify the sample measured (product name, chemical name and manufacturer's name and others, if any);

b) qualitative information about the existence of coating materials on the surface of a core material;

c) a reference to this document, i.e. ISO 17200:2020;

d) results of measurements;

e) documentation on sample preparation and measurement methods used for individual characteristics;

f) the date of measurement, name of measuring laboratory and statements on the quality system of measuring laboratory;

g) uncertainty of measurement results;

h) any other information supporting the reliability of measurement results;

i) any deviation from the measurement methods; if there are deviations from this document, the name of and detailed information on the measurement methods used and their justification.

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