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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

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).

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.

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 nanoparticles in a powder sample 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 a generic document rather than being applicable to nanoparticles, which in general are composed of a metal or a metal and a counter-ion. It is applicable to carbon materials (fullerenes, fullerene derivatives) and polymers (polystyrene), including calcium carbonates and titanium dioxides. This document is applicable to both coated and uncoated.

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.

Nanotechnology — Nanoparticles in powder form — Characteristics and measurements

1 Scope

This document specifies the fundamental characteristics to be measured of an engineered nanoparticles sample 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".

[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 may contain *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]

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 spectrometry
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.