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Nanotechnologies — Requirements and recommendations for the identification of measurands that characterise nano-objects and materials that contain them

Nanotechnologies – Exigences et recommandations pour l'identification des mesurandes qui caractérisent les nano-objets et les matériaux les contenant

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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*, and IEC/TC 113, *Nanotechnology for electrotechnical products and systems*.

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

The term “nano-object” applies to materials having one, two or three external dimensions in the nanoscale (therefore in the range of approximately 1 nm to 100 nm). Specific size dependent properties are usually exhibited in this size range, even if they do not disappear abruptly beyond these limits. Nano-objects, either natural or manufactured, can be found in the form of nanoplates (one dimension in the nanoscale), nanofibres (two dimensions, or the diameter, in the nanoscale), and nanoparticles (three dimensions in the nanoscale). Nano-objects exhibit higher specific surface areas than larger objects. They are particularly prone to aggregation and agglomeration phenomena due to attractive interactions during their life cycle.

There is increasing use of nano-objects in research and development, industry and commercial applications. Characterization of nano-objects, and their agglomerates and aggregates (NOAAs) plays an essential role in basic and applied research, through process and product quality control and commercialization to health and environmental protection. Characterization of nano-objects is key to determine their physical and chemical properties, performance and lifetime. The methods available for characterization of larger scale materials are often difficult to apply to nano-objects, sometimes due to restrictions of the test systems (e.g. low sensitivity, inadequate resolution of equipment). This has resulted in the development of new techniques and adaptation of existing ones.

The method selection is often strongly influenced by its initial cost and availability, time and sample compatibility. However, an aspect that is easily forgotten is whether the selected method truly targets the physical or chemical material property that is intended to be measured (“the measurand”). This can sound trivial, but in practice, insufficient knowledge or consideration about the actual measurement principle and/or the property measured can impede a correct assessment of the measurement results.

Measurement techniques and methods are typically classified according to the material properties they can measure. One definition of “measurand” used in many ISO standards is the “quantity intended to be measured”. In nanotechnologies popular material properties often considered as this “intended measurand” can be size, shape, chemical composition, surface charge. However, in reality, due to their different underlying physical measurement principles, results obtained by different techniques, for a common material property, can differ significantly. The logical reason for this is that these different techniques measure not the intended measurand but different measurands, which are specific to the technique but are closely related to the intended measurand.

For intended use in biological systems and therapeutic purposes, additional characterization beyond those mentioned in the document may be required.

This document describes measurands used to characterize nano-objects, and their agglomerates and aggregates. This document is split into 10 main clauses covering:

— Clause 6: size and shape measurands;

— Clause 7: chemical analysis measurands;

— Clause 8: mass and density;

— Clause 9: charge measurands;

— Clause 10: crystallinity measurands;

— Clause 11: optical properties measurands;

— Clause 12: electrical and electronic measurands;

— Clause 13: magnetic measurands;

— Clause 14: thermal measurands;

— Clause 15: other performance related measurands.

Nanotechnologies — Requirements and recommendations for the identification of measurands that characterise nano-objects and materials that contain them

1 Scope

This document specifies requirements and recommendations for the identification of measurands to characterize nano-objects and their agglomerates and aggregates, and to assess specific properties relevant to the performance of materials that contain them. It provides recommendations for relevant measurement.

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 20579-4, *Surface chemical analysis — Guidelines to sample handling, preparation and mounting — Part 4: Reporting information related to the history, preparation, handling and mounting of nano-objects prior to surface analysis*

ISO/TS 80004-1:2015, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO/TS 80004-2:2015, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

ISO/TS 80004-6:2021, *Nanotechnologies — Vocabulary — Part 6: Nano-object characterization*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1:2015, ISO/TS 80004-2:2015 and ISO/TS 80004-6:2021 and the following apply.

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

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

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

3.1 General core terms

3.1.1
nanoscale
length range approximately from 1 nm to 100 nm

Note 1 to entry: Properties that are not extrapolations from a larger size are predominately exhibited in this length range.

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

3.1.2
nano-object
discrete piece of material with one, two or three external dimensions in the *nanoscale* (3.1.1)

Note 1 to entry: The second and third external dimensions are orthogonal to the first dimension and to each other.

[SOURCE: ISO/TS 80004-1:2015, 2.5]

3.1.3
agglomerate
collection of weakly bound *particles* (3.1.5) or *aggregates* (3.1.4) or mixtures of the two where the resulting external surface area is similar to the sum of the surface areas of the individual components

Note 1 to entry: The forces holding an agglomerate together are weak forces, for example van der Waals forces, or simple physical entanglement.

Note 2 to entry: Agglomerates are also termed secondary particles and the original source particles are termed *primary particles* (3.1.6).

[SOURCE: ISO/TS 80004-6:2021, 3.10]

3.1.4
aggregate
particle (3.1.5) comprising strongly bonded or fused particles where the resulting external surface area may be significantly smaller than the sum of calculated surface areas of the individual components

Note 1 to entry: The forces holding an aggregate together are strong forces, for example covalent bonds, or those resulting from sintering or complex physical entanglement.

Note 2 to entry: Aggregates are also termed secondary particles and the original source particles are termed *primary particles* (3.1.6).

[SOURCE: ISO/TS 80004-6:2021, 3.11]

3.1.5
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* (3.1.2)

[SOURCE: ISO 26824:2013, 1.1]

3.1.6
primary particle
original source *particle* (3.1.5) of *agglomerates* (3.1.3) or *aggregates* (3.1.4) or mixtures 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.

Note 2 to entry: Agglomerates and aggregates are also termed secondary particles.

[SOURCE: ISO 26824:2013, 1.4]

3.1.7
constituent particle
identifiable, integral component of a larger *particle* (3.1.5)

Note 1 to entry: The constituent particle structures may be *primary particles* (3.1.6) or secondary particles.

[SOURCE: ISO/TS 80004-2:2015, 3.3]

3.2 Measurand related terms

3.2.1
measurand
quantity intended to be measured

Note 1 to entry: The specification of a measurand requires knowledge of the kind of quantity, description of the state of the phenomenon, body, or substance carrying the quantity, including any relevant component, and the chemical entities involved.

Note 2 to entry: In the second edition of the VIM and in IEC 60050-300:2001, the measurand is defined as the “particular quantity subject to measurement”.

Note 3 to entry: The measurement, including the measuring system and the conditions under which the measurement is carried out, might change the phenomenon, body, or substance such that the quantity being measured may differ from the measurand as defined. In this case, adequate correction is necessary.

EXAMPLE 1 The potential difference between the terminals of a battery may decrease when using a voltmeter with a significant internal conductance to perform the measurement. The open-circuit potential difference can be calculated from the internal resistances of the battery and the voltmeter.

EXAMPLE 2 The length of a steel rod in equilibrium with the ambient Celsius temperature of 23 °C will be different from the length at the specified temperature of 20 °C, which is the measurand. In this case, a correction is necessary.

Note 4 to entry: In chemistry, “analyte”, or the name of a substance or compound, are terms sometimes used for “measurand”. This usage is erroneous because these terms do not refer to quantities.

[SOURCE: ISO/IEC Guide 99:2007, 2.3]

3.2.2
particle size
linear dimension of a *particle* (3.1.5) determined by a specified measurement method and under specified measurement conditions

Note 1 to entry: Different methods of analysis are based on the measurement of different physical properties. Independent of the particle property actually measured, the particle size can be reported as a linear dimension, e.g. as the equivalent spherical diameter.

[SOURCE: ISO 26824:2013,1.5]

3.2.3
particle size distribution
distribution of the quantity of *particles* (3.1.5) as a function of *particle size* (3.2.2)

Note 1 to entry: Particle size distribution may be expressed as cumulative distribution or a distribution density (distribution of the fraction of material in a size class, divided by the width of that class).

Note 2 to entry: The quantity can be, for example, number, mass, or volume based.

[SOURCE: ISO/TS 80004-6:2021, 4.1.2]

3.2.4
particle shape
external geometric form of a *particle* (3.1.5)

[SOURCE: ISO/TS 80004-6:2021, 4.1.3]

3.2.5
aspect ratio
ratio of length of a *particle* (3.1.5) to its width

[SOURCE: ISO/TS 80004-6:2021, 4.1.4]

3.2.6
equivalent diameter
diameter of a sphere that produces a response by a given particle-sizing method, that is equivalent to the response produced by the *particle* (3.1.5) being measured

Note 1 to entry: Physical properties are, for example, the same settling velocity or electrolyte solution displacing volume or projection area under a microscope. The physical property to which the equivalent diameter refers should be indicated using a suitable subscript (see ISO 9276-1:1998), e.g. subscript "V" for equivalent volume diameter and subscript "S" for equivalent surface area diameter.

Note 2 to entry: For discrete-particle-counting, light-scattering instruments, an equivalent optical diameter is used.

Note 3 to entry: Other parameters, e.g. the effective density of the particle in a fluid, are used for the calculation of the equivalent diameter such as Stokes diameter or sedimentation equivalent diameter. The parameters used for the calculation should be reported additionally.

Note 4 to entry: For inertial instruments, the *aerodynamic diameter* (3.2.9) is used. Aerodynamic diameter is the diameter of a sphere of density 1 000 kg m^{-3} that has the same settling velocity as the particle in question.

[SOURCE: ISO/TS 80004-6:2021, 4.1.5]

3.2.7
light scattering
change in propagation of light at the interface of two media having different optical properties

[SOURCE: ISO/TS 80004-6:2021, 4.2.5]

3.2.8
hydrodynamic diameter
equivalent diameter (3.2.6) of a *particle* (3.1.5) in a liquid having the same diffusion coefficient as a spherical particle with no boundary layer in that liquid

Note 1 to entry: In practice, nanoparticles in solution can be non-spherical, dynamic, and solvated.

Note 2 to entry: A particle in a liquid will have a boundary layer. This is a thin layer of fluid or adsorbates close to the solid surface, within which shear stresses significantly influence the fluid velocity distribution. The fluid velocity varies from zero at the solid surface to the velocity of free stream flow at a certain distance away from the solid surface.

[SOURCE: ISO/TS 80004-6:2021, 4.2.6]

3.2.9
aerodynamic diameter
diameter of a sphere of density 1 g/cm^3 with the same terminal velocity due to gravitational force in calm air as the *particle* (3.1.5), under prevailing conditions of temperature, pressure and relative humidity

Note 1 to entry: Adapted from ISO 7708:1995, 2.2.

[SOURCE: ISO 23210:2009, 3.1.1]

3.2.10
mobility diameter
particle mobility diameter
particle mobility equivalent diameter
mobility equivalent diameter
diameter of a sphere carrying a single elementary charge with the same drift speed in an electric field as the *particle* (3.1.5) under prevailing condition of temperature and pressure

Note 1 to entry: The mobility diameter of a particle depends on its size, shape and electric charge level (which depends on the charging process involving its capacitance, i.e. its capacity to become electrically charged by bipolar air ions), but not of its density.

[SOURCE: EN 16966:2018]

3.2.11
mass specific surface area
absolute surface area of the sample divided by sample mass

Note 1 to entry: The mass specific surface area has units of m^2/kg.

[SOURCE: ISO/TS 80004-6:2021, 4.6.1]

3.2.12
photoluminescence
luminescence caused by absorption of optical radiation

[SOURCE: ISO/TS 80004-6:2021, 5.3]

3.2.13
Raman effect
emitted radiation, associated with molecules illuminated with monochromatic radiation, characterized by an energy loss or gain arising from rotational or vibrational excitations

[SOURCE: ISO/TS 80004-6:2021, 5.9]

3.2.14
lattice parameters
linear and angular dimensions of the crystallographic unit cell

[SOURCE: ISO 21432:2019, 3.19]

3.2.15
scattering angle
angle between the direction of the incident *particle* (3.1.5) or photon and the direction that the particle or photon is travelling after scattering

[SOURCE: ISO 18115-1:2013, 4.18]

3.2.16
zeta potential
electrokinetic potential
difference in electric potential between that at the slipping plane and that of the bulk liquid

Note 1 to entry: Electrokinetic potential is expressed in volts

[SOURCE: ISO/TS 80004-6:2021, 6.4.5]

3.2.17
fluorescence
phenomenon in which absorption of light of a given wavelength by a substance is followed by the emission of light at a longer wavelength

[SOURCE: ISO/TS 80004-6:2021, 4.5.12]

3.2.18
Curie temperature
temperature at which a ferromagnetic material passes from the ferromagnetic state to the paramagnetic state or vice versa

[SOURCE: ISO 11358-1:2014, 3.3]

3.2.19
thermal diffusivity
ratio of thermal conductivity to specific heat capacity per unit mass, which describes the rate at which heat flows through a material, expressed in m^2/s

[SOURCE: ISO 13826:2013, 2.1]

3.2.20
solubility
maximum mass of a nanomaterial that is soluble in a given volume of a particular solvent under specified conditions

Note 1 to entry: Solubility is expressed in grams per litre of solvent.

[SOURCE: ISO/TS 12901-2:2014, 3.17]

3.2.21
dispersibility
qualitative or quantitative characteristic or property of a particulate source material assessing the ease with which said material can be dispersed within a continuous phase

Note 1 to entry: Spatially uniform distribution (homogeneity) of the dispersed phase is considered an integral part of the desired end point.

Note 2 to entry: *Particle size* (3.2.2) or *particle size distribution* (3.2.3) is often used as an end point relative to defined criteria specific to the application.

Note 3 to entry: Dispersibility refers to a specific dispersion process and specific process time.

Note 4 to entry: Dispersion stability, though a related phenomenon, should not be confused with *dispersibility*.

[SOURCE: ISO/TS 22107:2021, 3.6]

4 Abbreviated terms

For the purposes of this document, the following abbreviations apply.

In the list of abbreviated terms below, note that the final “M”, given as “Microscopy”, may be taken equally as “Microscope” and “S”, given as “Spectroscopy”, may be taken equally as “Spectrometer” depending on the context.

1D	one dimensional
2D	two dimensional
3D	three dimensional
AES	Auger electron spectroscopy
AFM	atomic force microscopy
AGFM	alternating gradient-field magnetometer
APM	aerosol particle mass analyser

APS	aerodynamic particle sizing
ARPES	angle-resolved ultraviolet photoemission spectroscopy
ATR	attenuated total reflectance
BET method	Brunauer, Emmet and Teller method
CLS	centrifugal liquid sedimentation
CPC	condensation particle counter
CVC	colloid vibration current
DC	direct current
DCS	differential centrifugal sedimentation
DEMC	differential electrical mobility classifier
DLS	dynamic light scattering
DMAS	differential mobility analysis system
DRIFT	diffuse reflectance infrared Fourier transform spectroscopy
DSC	differential scanning calorimetry
EBSD	electron backscatter diffraction
EDX	energy dispersive X-ray spectroscopy NOTE EDS is another acronym for this method.
EELS	electron energy loss spectroscopy
EGA	evolved gas analyser
EL	electroluminescence
ELPI	electrical low-pressure impaction
EPR	electron paramagnetic resonance
ESA	electrokinetic sonic amplitude
ESR	electron spin resonance
FFF	field-flow fractionation
FIB	focused ion beam
FTIR	Fourier transform infrared spectroscopy
HRTEM	high-resolution transmission electron microscopy
LD	laser diffraction
ICP	inductively coupled plasma
ICP-AES	inductively coupled plasma atomic emission spectroscopy