



BSI Standards Publication

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

National foreword

This Published Document is the UK implementation of CEN ISO/TS 23302:2022. It is identical to ISO/TS 23302:2021. It supersedes PD CEN/TS 17010:2016 and PD ISO/TS 23302:2021, which are withdrawn.

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 committee manager.

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English Version

**Nanotechnologies - Requirements and recommendations
for the identification of measurands that characterise
nano-objects and materials that contain them (ISO/TS
23302:2021)**

Nanotechnologies - Exigences et recommandations
pour l'identification des mesurands qui caractérisent
les nano-objets et les matériaux les contenant (ISO/TS
23302:2021)

Nanotechnologien - Anforderungen und Empfehlungen
zur Identifizierung von Messgrößen zur
Charakterisierung von Nanoobjekten und von
Werkstoffen, die welche enthalten (ISO/TS
23302:2021)

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The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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European foreword

The text of ISO/TS 23302:2021 has been prepared by Technical Committee ISO/TC 299 "Nanotechnologies" of the International Organization for Standardization (ISO) and has been taken over as CEN ISO/TS 23302:2022 by Technical Committee CEN/TC 352 "Nanotechnologies" the secretariat of which is held by AFNOR.

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Endorsement notice

The text of ISO/TS 23302:2021 has been approved by CEN as CEN ISO/TS 23302:2022 without any modification.

Contents

Page

Foreword.....	vii
Introduction.....	viii
1 Scope.....	1
2 Normative references.....	1
3 Terms and definitions.....	1
3.1 General core terms.....	1
3.2 Measurand related terms.....	3
4 Abbreviated terms.....	6
5 Approaches to identify measurands to characterize nano-objects and their agglomerates and aggregates, and materials containing nano-objects.....	9
5.1 Procedure.....	9
5.2 Types of measurands.....	10
5.3 State of nano-objects.....	10
6 Measurands related to size and shape measurement of nano-objects and their agglomerates and aggregates.....	11
6.1 General.....	11
6.2 Measurands related to size and shape measurement.....	11
6.2.1 Overview.....	11
6.2.2 General relevant standards.....	12
6.3 Measurands related to size and shape measurement in aerosols.....	12
6.3.1 Overview.....	12
6.3.2 General relevant standards.....	13
6.3.3 Electrical low-pressure impaction.....	13
6.3.4 Cascade impactors.....	14
6.3.5 Differential mobility analysing system.....	14
6.3.6 Relevant standards.....	15
6.3.7 Optical particle counter.....	15
6.3.8 Relevant standards.....	15
6.3.9 Aerodynamic particle sizing.....	15
6.3.10 TEM combined with TEM grid samplers.....	15
6.3.11 Relevant standards.....	16
6.3.12 Scanning electron microscopy.....	16
6.3.13 Relevant standards.....	17
6.4 Measurands related to size and shape measurement in powders.....	17
6.4.1 Overview.....	17
6.4.2 Relevant standards.....	17
6.4.3 Scanning electron microscopy.....	18
6.4.4 Relevant standards.....	18
6.4.5 Gas adsorption, the BET method.....	18
6.4.6 Relevant standard.....	18
6.4.7 Laser diffraction.....	18
6.4.8 Relevant standard.....	19
6.4.9 X-ray diffraction.....	19
6.4.10 Relevant standards.....	19
6.4.11 Raman spectroscopy.....	19
6.5 Measurands related to size and shape measurements of nano-objects in liquid dispersions.....	20
6.5.1 Overview.....	20
6.5.2 Centrifugal liquid sedimentation.....	21
6.5.3 Relevant standards.....	21
6.5.4 Dynamic light scattering.....	21
6.5.5 Relevant standards.....	22

6.5.6	Laser diffraction.....	22
6.5.7	Relevant standard.....	22
6.5.8	Small angle X-ray scattering.....	22
6.5.9	Relevant standard.....	22
6.5.10	Particle tracking analysis.....	22
6.5.11	Relevant standards.....	23
6.5.12	Electron microscopy.....	23
6.5.13	Field flow fractionation.....	23
6.5.14	Relevant standard.....	24
6.5.15	Single particle ICP-MS.....	24
6.5.16	Relevant standard.....	25
6.6	Measurands related to size and shape measurement on surfaces (microscopy techniques).....	25
6.6.1	Overview.....	25
6.6.2	Scanning electron microscopy.....	25
6.6.3	Atomic force microscopy.....	25
6.6.4	Relevant standards.....	26
7	Measurands related to chemical analysis of nano-objects and their agglomerates and aggregates.....	26
7.1	General.....	26
7.2	Measurands related to surface chemical analysis of nano-objects and their agglomerates and aggregates.....	27
7.2.1	Measurands.....	27
7.2.2	Auger electron spectroscopy.....	28
7.2.3	Relevant standards.....	28
7.2.4	Electron energy loss spectroscopy.....	28
7.2.5	Relevant standard.....	28
7.2.6	Secondary ion mass spectroscopy.....	28
7.2.7	Relevant standards.....	29
7.2.8	X-ray fluorescence spectroscopy.....	29
7.2.9	Relevant standards.....	30
7.2.10	X-ray diffraction.....	30
7.2.11	Relevant standard.....	30
7.2.12	X-ray photoelectron spectroscopy.....	30
7.2.13	Relevant standards.....	31
7.2.14	Energy dispersive X-ray spectroscopy.....	31
7.2.15	Low energy ion scattering.....	31
7.3	Measurands related to the chemical analysis of nano-objects as bulk samples.....	31
7.3.1	Measurands.....	31
7.3.2	Fourier transform infrared spectroscopy.....	32
7.3.3	Relevant standards.....	33
7.3.4	Thermal analysis with evolved gas analyser plus FTIR or QMS.....	33
7.3.5	Relevant standards.....	34
7.3.6	Ultraviolet-visible spectroscopy.....	34
7.3.7	Relevant standards.....	34
7.3.8	Raman spectroscopy.....	34
7.3.9	Inductively coupled plasma techniques.....	34
7.3.10	Relevant standards.....	34
7.3.11	Contact angle.....	35
8	Measurands related to mass and density.....	35
8.1	General.....	35
8.2	Aerosols.....	35
8.2.1	Measurands.....	35
8.2.2	Relevant standards.....	35
8.2.3	Aerosol particle mass analyser.....	36
8.2.4	Time of flight mass spectrometry.....	36
8.3	Powders.....	36

8.3.1	Measurands	36
8.3.2	Pycnometry	36
8.3.3	Relevant standards	36
8.4	Liquid dispersions	37
8.4.1	Measurands	37
8.4.2	Relevant standards	37
8.4.3	Centrifugal liquid sedimentation (isopycnic method)	37
8.4.4	Static light scattering	37
8.4.5	Resonant mass measurement	38
9	Measurands related to charge — Liquid dispersions	38
9.1	Measurands	38
9.2	Relevant standards	38
9.3	Electrophoretic light scattering	38
9.4	Electroacoustic phenomena measurements	39
10	Measurands related to crystallinity	39
10.1	Measurands	39
10.2	Small-angle/wide-angle X-ray scattering	40
10.3	X-ray diffraction	40
10.4	High-resolution transmission electron microscopy	41
10.5	Electron backscattered diffraction	41
10.6	Neutron diffraction	41
10.7	Reflection high-energy electron diffraction and low-energy electron diffraction	41
10.8	Differential scanning calorimetry	41
10.9	Relevant standards	42
10.10	Solid state nuclear magnetic resonance crystallography	42
10.11	Raman crystallography	42
10.12	Relevant standards	42
11	Optical properties measurands	42
11.1	General	42
11.2	Measurands	43
11.3	Spectroscopy techniques	43
11.4	Relevant standards	44
12	Electrical and electronic measurands	44
12.1	Measurands	44
12.2	Techniques	45
12.2.1	2- or 4-point conductance measurements	45
12.2.2	Angle-resolved ultraviolet photoemission spectroscopy	45
12.2.3	Scanning tunnelling microscopy	45
12.2.4	Conductive atomic force microscopy	45
12.2.5	Piezoforce microscopy	46
13	Magnetic measurands	46
13.1	General	46
13.2	Measurands	46
13.3	Techniques	47
13.3.1	Superconducting quantum interference device	47
13.3.2	Vibrating sample magnetometer	48
13.3.3	Mössbauer spectroscopy	48
13.3.4	Electron paramagnetic resonance spectroscopy	48
13.3.5	Magneto-optical Kerr-effect	48
13.3.6	Magnetic force microscopy	48
13.3.7	Scanning Hall effect microscopy	48
13.3.8	Spin-polarized scanning tunnelling microscopy	49
13.3.9	Relevant standards	49
14	Thermal measurands	49
14.1	Measurands	49

14.2	Techniques.....	49
14.2.1	Measurement of specific heat capacity	49
14.2.2	Scanning thermal microscopy.....	49
14.3	Relevant standard	50
15	Other performance related measurands.....	50
15.1	General.....	50
15.2	Powders — Dustiness	50
15.2.1	Measurands.....	50
15.2.2	Relevant standards	50
15.3	Liquid dispersions.....	51
15.3.1	Measurands.....	51
15.3.2	Viscosity.....	51
15.3.3	Dispersibility	53
15.3.4	Relevant standard	53
15.3.5	Solubility and rate of dissolution.....	53
15.3.6	Relevant standards	54
15.4	Mechanical properties.....	54
15.4.1	General.....	54
15.4.2	Measurement of elastic constants by static methods.....	55
15.4.3	Relevant standards	55
15.4.4	Measurement of elastic constants by dynamic methods	55
15.4.5	Relevant standards	56
15.4.6	Measurement of elastic and plastic properties by instrumented indentation methods.....	56
15.4.7	Relevant standards	56
15.4.8	Measurement of surface properties and wear.....	56
15.4.9	Relevant standard	57
	Bibliography.....	58

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).

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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*, 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 $1\,000\text{ kg m}^{-3}$ 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

ICP-OES	inductively coupled plasma optical emission spectroscopy
ICP-MS	inductively coupled plasma mass spectrometry
IR	infrared
LALS	low angle light scattering
LEED	low-energy electron diffraction
LEIS	low-energy ion scattering
MALS	multi-angle light scattering
MFM	magnetic force microscopy
MOKE	magneto-optical Kerr effect
NP	nanoparticle
NIR	near infrared
NMR	nuclear magnetic resonance
NOAA	nano-objects, and their agglomerates and aggregates
OPC	optical particle counter
PAS	photoacoustic spectroscopy
PFM	piezoforce microscopy
PL	photoluminescence
PSD	particle size distribution
PTA	particle tracking analysis
QMS	quadrupole mass spectrometer
RALS	right angle light scattering
RF	radio frequency
RBM	radial breathing mode
RHEED	reflection high-energy electron diffraction
RLC	resistor, inductor and capacitor
RMM	resonant mass measurement
SAXS	small angle X-ray scattering
SEM	scanning electron microscopy
SIMS	secondary ion mass spectrometry
SLS	static light scattering
SMLS	static multiple light scattering

SMPS	scanning mobility particle spectrometer
sp-ICP-MS	single particle ICP-MS
SPR	surface plasmon resonance
SP-STM	spin polarized scanning tunnelling microscopy
SThM	scanning thermal microscopy
STM	scanning tunnelling microscopy
STS	scanning tunnelling spectroscopy
SQUID	superconducting quantum interference device
TEM	transmission electron microscopy
TGA	thermogravimetric analysis
TOF	time of flight
TXRF	total reflection X-ray fluorescence
UHV	ultra-high vacuum
UV-Vis	ultraviolet-visible
USAXS	ultra-small angle X-ray scattering
VSM	vibrating sample magnetometer
WAXS	wide angle X-ray scattering
XRD	X-ray diffraction
XRF	X-ray fluorescence spectroscopy
XPS	X-ray photoelectron spectroscopy

5 Approaches to identify measurands to characterize nano-objects and their agglomerates and aggregates, and materials containing nano-objects

5.1 Procedure

The selection of measurands to characterize NOAAs is defined by the type and quality of information required by the user. Multiple methods or techniques are usually available for a specific measurand, and thus the method(s) or technique(s) chosen should be fit for purpose and compatible with the sample material.

In this document, measurands from different categories are described. Use one or more of the measurands to characterize NOAAs. The measurands that shall be used depend on the information that the user requires. Very often the user requires more than one measurand, for example size, shape, charge and chemistry therefore measurands from different subclauses of this document should be selected where required.

The user should not confuse 'measurand' with 'method', the latter being a means to obtain the former. The user should also be aware that different methods that nominally provide the same type of information, for example size, can in fact yield different results due to method-dependent difference and other factors. Therefore, an orthogonal approach (use of multiple orthogonal methods), when

practical, should be applied toward measurand determination when method dependencies are known or suspected. On the other hand, if different methods are targeting exactly the same measurand, where this measurand is specifically defined for example by fundamental or structural principles, then results should always be comparable and independent from the method used provided the methods are correctly calibrated.

5.2 Types of measurands

To aid the user in the selection of measurands and specific methods or techniques, this document describes the measurands for characterizing nano-objects based on commonly used techniques and separated into different 'types' of measurands. Tables and text in each subclause describe:

- a) Overarching measurand: this is the measurand intended to be measured, typically as the final output.
- b) Measurands specific to the method: these are the measurands measured by the instrument and are related to the overarching measurand. Results obtained by different techniques can be significantly different from one another due to differences in the techniques' underlying physical measurement principles.
- c) Range and resolution of particular techniques: these are given in units that depend on the particular measurand and are for typical current commercial instruments. State of the art research instruments can have higher resolution than those stated. The resolution of each measurand and technique is different and calculated via different means. Depending on the technique, it can be a percentage of the measurand size, the ability to resolve two peaks or ultimate performance of an instrument. Resolution is provided only as a guide and not discussed in detail here.
- d) Limitations and references: references to ISO standards are stated where they are available. These should be consulted for detailed information. Limitations refer to limitations of the technique.

5.3 State of nano-objects

Nano-objects exist in a number of different states, a summary of which is provided in [Table 1](#). For analysis, different techniques require the nano-objects to be in different forms or dispersed phases.

The material under test can be composed entirely of nano-objects or it can contain a percentage of nano-objects where the percentage can be measured via different measurands, for example number or mass, and hence different techniques. The degree and nature of the dispersion of the nano-objects is also important and should be considered, see [Clause 15](#).

Table 1 — Different states of nano-objects

Continuous phase	Dispersed phase		
	Solid	Liquid	Gas
Solid	Nano-composite, nano-(structured) powder mixture	Nano-porous material	Nano-porous material Solid nanofoam
Liquid	Nano-suspension / colloid	Nano-emulsion	Liquid nano-foam
Gas	Nano-aerosol (solid)	Nano-aerosol (liquid)	(does not apply)

Other factors that need to be considered include sample preparation. Here, the state of nano-objects before, during and after sample preparation and prior to analysis can vary considerably and effect the measured results. Examples include the type and duration of sonication and extraction and deposition parameters and methods.

6 Measurands related to size and shape measurement of nano-objects and their agglomerates and aggregates

6.1 General

The shape, mean size, and particle size distribution of NOAA have a strong influence on their intrinsic properties or the properties of composite materials that contain them. Polymers and elastomers reinforced by nano-sized fillers are examples.

During their life cycle, nano-objects and the materials that contain them need to be characterized. Several time identifiable steps can be identified where characterization can be appropriate. These include after synthesis, after shipping, after storage and processing before use. This characterization allows checking whether they exhibit the properties they are designed for. This is the case even if the relationship between intrinsic characteristics of the nano-objects and the desired application property (e.g. optical, electrical, and mechanical) is not fully understood. Regarding size and shape, their characterization should be made in dry form or in liquid dispersion, depending on the analytical technique used.

Due to the possibility of toxicological effects of some nano-objects, measurement methods in the air matrix, either at the workplace or in ambient air in the immediate vicinity of a production plant can be useful. In this case, in situ and real-time measurements are favoured since they allow monitoring emission fluctuations whereas classical laboratory techniques are used after sampling on suitable supports. During the use of a material containing nano-objects, some of them can be released from the material, as a consequence of a mechanical or atmospheric wear, for instance cutting or drilling of building materials, wear of paints or varnishes. Measurements in air are also needed in this case. Nano-objects can be coated on surfaces, to give for example antibacterial, optical or self-washing properties. Specific measurements of size and shape parameters on the surface of materials are then needed.

Overarching and specific measurands differ according to the techniques used and the continuous phase the nano-objects are in. These measurands and the techniques used to assess them are presented below.

6.2 Measurands related to size and shape measurement

6.2.1 Overview

PSD is a list of numerical values that represent the relative, or in rare cases the absolute number of particles present in different size classes or bins ranging from the smallest particle size to the largest particle size. There are a number of different amount evaluators (types), the most relevant of which are:

- a) the number of particles in a size class;
- b) the total surface area of particles in a size class;
- c) the total volume of the particles in a size class;
- d) the mass of the particles in a size class.

Evaluators like scattered light intensity are used to obtain PSD's in techniques using the scattering of light (e.g. DLS). These PSD's can be transformed into number or volume PSD's using specific mathematical models, often requiring assumptions on the spherical shape of particles, uniform particle density or the knowledge of other physical parameters like refractive index. Here, the term "particle" refers to NOAAs since many techniques used for size measurement are not able to differentiate between them and obtain separate PSD's for individual nano-objects and their agglomerates and aggregates.

Shape is the external geometric form of a particle. When direct observational techniques are used like microscopy, more specific measurands can be defined in image analysis software to describe the shape of an object. Commonly used measurands are, Feret elongation (the ratio between maximum Feret diameter and minimum Feret diameter where the Feret diameter is the distance between two parallel

tangential lines in a specific direction), aspect ratio (the ratio of length of a particle to its width) and convexity (the ratio between the projected area of the object and the area of its convex hull).

Particle shape distributions containing the amount of particles in different shape classes can be obtained by means of image analysis. The distribution amount is classified by particle number, surface area or volume.

NOTE Care needs to be taken when applying the definition of an aspect ratio (a 2D parameter) to different shaped materials (which are 3D). The relevant lengths are dependant on the morphology of the nano-object, for example, particle versus rod versus plate.

6.2.2 General relevant standards

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

ISO 9276-2, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

ISO 9276-3, *Representation of results of particle size analysis — Part 3: Adjustment of an experimental curve to a reference model*

ISO 9276-4, *Representation of results of particle size analysis — Part 4: Characterization of a classification process*

ISO 9276-5, *Representation of results of particle size analysis — Part 5: Methods of calculation relating to particle size analyses using logarithmic normal probability distribution*

ISO 9276-6, *Representation of results of particle size analysis — Part 6: Descriptive and quantitative representation of particle shape and morphology*

ISO 26824, *Particle characterization of particulate systems — Vocabulary*

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

6.3 Measurands related to size and shape measurement in aerosols

6.3.1 Overview

Nano-objects are prone to aggregation and agglomeration in all the matrices in which they are distributed. It is especially the case with aerosols where aggregates and agglomerates are present in most circumstances. As a consequence, measurement techniques have to be applicable not only in the nanoscale range but also for particles above 100 nm.

[Table 2](#) provides a summary of the measurands and methods used to determine size and shape of nano-objects in aerosols. In this table and the other tables that follow in this document, the term “overarching measurand” refers to the physical parameter that the technique intends to measure. The term “measurand(s) specific to method” refers to the physical parameter actually measured by the technique. For example, in electrical impaction, the inertia of particles is the specific measurand since it determines their trajectory in the device, therefore the plate which collects them. This plate corresponds to a particular equivalent aerodynamic diameter size class (the amount of particles on this plate – and on all others – gives a particle size distribution which is the overarching measurand). The term “resolution” does not refer to the minimum size achievable by the technique (this is indicated by the “typical size range”) but to the ability to distinguish two different sizes: it is therefore mainly linked to the number of measuring channels typically offered by the devices.

“Limitation” refers to the main drawback of the technique. It can be a concentration range, a measurement time or a minimum achievable size. Even if a particular technique gives number-weighted PSD, the concentration range is given in mass concentrations since it is easily known prior to measurement, for example, by sampling on filters.

[Table 2](#) contains techniques that measure in real-time with the exception of cascade impaction that detect the two microscopy techniques at the end of the table. The real-time techniques measure size only whereas the microscopy techniques can measure shape as well as size.

Table 2 — Measurands related to the size and shape measurement in aerosols

Overarching measurand	Measurand specific to the method	Method	Size range	Resolution	Limitations	References
Number-weighted size distribution, where "size" is the particle equivalent aerodynamic diameter	Electrical detection from impaction	ELPI	Medium equivalent aerodynamic diameter between 6 nm and 10 µm	Typically 13 to 500 size classes or bins split over the whole size range	Concentration range: 10 µg/m ³ to 100 mg/m ³ . Particle density shall be known. Particle bounce and charge transfer problems.	[1]
Mass-weighted size distribution	Inertia	Cascade impaction	10 nm to 18 µm (depending on specific design)	Typically 3 to 14 size classes or bins split over the size range		
Number – electrical mobility size distribution	Electrical mobility	DMAS	3 nm to 1 000 nm	sub-nm	Measuring takes at least one minute –not adapted for fast phenomena.	[2]
Number-weighted size distribution, "size" being the equivalent optical diameter of particles	Scattering cross-section	OPC	0,5 µm to 20 µm	0,1 µm	For NOAA, only usable for aggregates and agglomerates >0,5 µm (nanoplate issue). The refractive index shall be known.	[3]
Mass-weighted aerodynamic size distribution	Velocity	APS	0,5 µm to 20 µm	0,1 µm	The resolution is lower for non-spherical particles.	[4]
Size and shape of NOAA	2D-projected size and shape of NOAA. Number-weighted geometric size and shape distributions.	Particle sampler with TEM and image analysis	1 nm to 10 µm	0,2 nm	Typical sampling efficiency has a minimum (10 %) at 20 nm to 30 nm. TEM is not a real time technique.	[5],[6]
Size and shape of NOAA	2D-projected size and shape of NOAA. Number-weighted geometric size and shape distributions.	SEM on filters	5 nm to 1 mm	5 nm	Needs sample preparation onto a substrate. Slow, individual particle measurement. SEM is not a real time technique.	[7]

NOTE In [Table 2](#) and subsequent tables the parameters size range, resolution and limitations relate to the measurement method.

6.3.2 General relevant standards

ISO/TR 18196, *Nanotechnologies — Measurement technique matrix for the characterization of nano-objects*

ISO/TR 27628, *Workplace atmospheres — Ultrafine, nanoparticle and nano-structured aerosols — Inhalation exposure characterization and assessment*

6.3.3 Electrical low-pressure impaction

In ELPI, the aerosol particles are first electrically charged by a corona effect and fed into a low-pressure impactor fitted with several plates. Due to their inertial motion, the largest particles (or aggregates and agglomerates) accumulate on the upper plates whereas the smallest ones accumulate on the lower plates. Each plate is equipped with an electrical counting system delivering a signal proportional to the electric charge received by the plate. A number PSD containing a number of size classes equal to the number of plates in the impactor is thus obtained.

The specific measurand involved in this technique is the inertia of objects. This measurand is directly linked to density and aerodynamic diameter for a given gas flow. Therefore, each plate collects particles in a given aerodynamic diameter range, for a given density.

At each plate, the total number of collected particles in the corresponding mass or diameter range is derived from the measured electrical current signal. This is possible after the experimental determination of the charger efficiency function. For each aerodynamic diameter, this function takes into account particle losses inside the charger, the fraction of particles charged and their average charge. The density of particles shall be known. Number-weighted size distributions can be obtained by this technique.

Recent enhanced designs (high resolution electrical low-pressure impactors) can provide size distribution with up to 500 size classes. Their specific impactor technology enables post-measurement chemical analysis of size classified particles.

As the technique assumes the particles to be spherical, it is not able to give direct information about the shape of nano-objects. However, it is possible to examine individual particles on the plates using SEM to obtain shape information.

6.3.4 Cascade impactors

Other designs are 13- and 14-stage cascade impactors that classify particles according to their inertia into 13 or 14 size fractions in the range of 16 nm to 10 µm. Each stage is weighed before and after measurement: therefore, a mass-weighted particle size distribution is obtained.

Micro-orifice uniform deposit impactors are cascade impactors used to fractionate aerosol particles according to their aerodynamic size. They differ from other conventional cascade impactors, in the use of a large number of micro-orifice nozzles to reduce jet velocity and pressure drop, minimize particle bounce and reentrainment, and enhance collection efficiency. This results in sharper cut-size characteristics and more accurate mass-weighted size distributions.

NOTE Typical electrical impactors determine the size range in the order of 16 nm to 10 µm. The measurable mass concentration range depends on the size. For example, for an aerodynamic diameter of 100 nm, it is about 10 µg/m³ to 100 mg/m³.

6.3.5 Differential mobility analysing system

A differential mobility analysis system, also known as SMPS, is the combination of a DEMC and a CPC. Here, particles are first electrically charged by means of a radioactive source, according to the Fuchs equilibrium charge distribution law. An adjustable electric field is applied between two electrodes to deflect the path of charged objects entering the DEMC. Since this deflection is proportional to the electrical mobility, each voltage setting extracts a specific size class of objects from the system. The DEMC essentially acts as a band pass filter, allowing only objects of a specific mobility size to exit the column. Electrical mobility is determined by the charge-to-size ratio. For singly-charged spheres, the size is equivalent to the geometric spherical diameter of the object. For non-spherical objects, the size is the equivalent spherical mobility diameter exhibiting the same drag force as the measured object. A CPC placed at the outlet records the number concentration of each class using an optical-based technique. A mobility size distribution is thus obtained in a typical size range from 3 nm to 1 000 nm, although novel instruments are improving the size range down to 0,5 nm. Different columns (DEMCs) cover different size ranges in the classical DMAS and different CPCs exhibit different limits of detection. The specific measurand involved in this technique is the electrical mobility, directly linked to charge and aerodynamic diameter of objects since their charge law is known. Information on the shape of the objects cannot be directly obtained, but models exist to fit data to a known shape function to extract, for example, length distributions. For highly anisotropic objects like nanofibres, the charging law can be unpredictable, resulting in large uncertainties in the results.

Some alternate DEMC designs exist, where one of the electrodes is divided into a number of segments, each connected to highly sensitive electrometers. This allows the extension of the size range of the technique, typically from 10 nm to 10 µm. The fast mobility particle size spectrometer is an example of such a design and can measure particles in the range from 5,6 nm to 560 nm with a total of 32 channels.

These designs are much faster than a classical DMAS since they can perform measurements at more than 1 Hz.

6.3.6 Relevant standards

ISO 15900, *Determination of particle size distribution — Differential electrical mobility analysis for aerosol particles*

ISO 27891, *Aerosol particle number concentration — Calibration of condensation particle counters*

EN ISO 28439, *Workplace atmospheres — Characterization of ultrafine aerosols/nano aerosols – Determination of the size distribution and number concentration using differential electrical mobility analysing systems*

6.3.7 Optical particle counter

The aerosol containing the nano-objects flows into a cell illuminated by a laser beam. Objects crossing the beam scatter light according to their particle size and shape. This phenomenon is due to the physical effects of diffraction, reflection and refraction. A photodetector converts light flashes received at given angles into electrical pulses. Using the height and count rate of the pulses, the number concentration and the equivalent spherical diameter can be derived from Mie theory. For precise size distributions, the refractive index of particles shall be known. Usually the instrument is calibrated using a monodisperse aerosol of known refractive index such as polystyrene latex. The resulting size distribution displayed by the instrument is based on an equivalent diameter. Mie scattering calculations have been used to both model the response of the instruments and invert the resulting data but are limited by knowledge of the particle's refractive indices and shape. Number-weighted optical equivalent size distribution ranging from 0,3 μm to 20 μm can be measured using such counters. This size range can be seen as irrelevant for the nanoscale but is complementary to SMPS measurements since nanoparticles are often found in aerosols under the form of aggregates and agglomerates.

6.3.8 Relevant standards

ISO 13320, *Particle size analysis — Laser diffraction methods*

ISO 21501-1, *Determination of particle size distribution — Single particle light interaction methods — Part 1: Light scattering aerosol spectrometer*

6.3.9 Aerodynamic particle sizing

In APS, the aerosol is pumped and accelerated through a nozzle. Particles exiting the jet have a velocity which depends on their surface area and mass. They cross two laser beams separated by a known distance, which produces two pulses of scattered light. Measuring the time delay between the two pulses allows the determination of the velocity. Aerodynamic diameter of particles (overarching measurand) is derived from the measured velocity, assuming spherical shape and unity density. A mass PSD is obtained in the size range 0,5 μm to 20 μm so this technique is only suitable for measuring aggregates and agglomerates of nano-objects and not the nano-objects themselves.

6.3.10 TEM combined with TEM grid samplers

TEM is a microscopy technique in which a beam of electrons is transmitted through a thin specimen to form an image. Number-weighted geometric size and shape distributions can be obtained by means of image analysis of TEM micrographs, with some limits and restrictions. It is usually not possible to obtain separate PSD's for nano-objects since primary particles are very difficult to isolate in an aggregate or agglomerate especially with automatic criteria used by the software. Very long nanofibres, like carbon nanotubes, can be difficult to separate as individual fibres since they are often entangled inside complex agglomerates. When using sampling devices, the flow rate and sampling duration should be carefully set for the amount of collected particles to be optimized. Indeed, low concentrations can limit the counting statistics whereas high concentrations can hinder the counting, overlapping objects