SRI LANKA STANDARD 12006:2013
ISO/TS 12805:2011

NANOTECHNOLOGIES - MATERIALS SPECIFICATIONS - GUIDANCE ON SPECIFYING NANO-OBJECTS

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard

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SLS 12006:2013
ISO/TS 12805:2011

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Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

This standard does not purport to include all the necessary provisions of a contract.

Sri Lanka Standard
NANOTECHNOLOGIES - MATERIALS SPECIFICATIONS - GUIDANCE ON SPECIFYING NANO-OBJECTS

NATIONAL FOREWORD

This standard was approved by the National Mirror Committee on Nanotechnology and authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2013.11.27.

This Sri Lanka Standard is identical with **ISO/TS 12805:2011**, Nanotechnologies - Materials specifications -Guidance on specifying nano-objects, published by the International Organization for Standardization (ISO).

TERMINOLOGY AND CONVENTIONS

The text of the International Standard has been accepted as suitable for publication, without any deviation as a Sri Lanka Standard. However, certain terminology and conventions are not identical with those used in Sri Lanka Standards. Attention is therefore drawn to the following:

a) Wherever the words “International Standard” appear referring to this standard they should be interpreted as “Sri Lanka Standard”.

b) The comma has been used throughout as a decimal marker. In Sri Lanka Standards, it is the current practice to use a full point on the baseline as the decimal marker.

Wherever page numbers are quoted, they are “ISO” page numbers.

CROSS REFERENCES

International Standard	Corresponding Sri Lanka Standard
ISO/TS 27687, Terminology and definitions for Nano-objects - Nanoparticle, nanofiber and nanoplate	**SLS 12000 - Part 1**, Terminology and definitions for nano-objects - nanoparticle, nanofiber and nanoplate
ISO/TS 80004 - 1, Nanotechnologies - Vocabulary- Part 1: Core terms	**SLS 12000 - Part 2**, Nanotechnologies- Vocabulary- Part 1: Core terms

SLS 12006:2013

TECHNICAL SPECIFICATION

ISO/TS 12805

First edition
2011-11-15

Nanotechnologies — Materials specifications — Guidance on specifying nano-objects

Nanotechnologies — Spécifications de matériaux — Lignes directrices de spécification des nano-objets

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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.

ISO/TS 12805 was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Introduction

The need for this Technical Specification arose in response to the failure of specifications agreed between suppliers of manufactured nano-objects and their customers to ensure delivery of material that responds consistently to downstream processing or that is capable of generating consistent performance in the final product between batches and lots.

This observed inconsistent performance of batches or lots of material has led to the conclusion that the cause has to be related to one or more of the following scenarios.

a) The specification agreed between customer and supplier does not cover all material characteristics that have an influence on performance and/or processability, or it has been interpreted differently by the customer and supplier.

b) One or more material characteristic is currently being measured by an inappropriate technique.

c) One or more measurement technique is being applied in an incorrect manner.

This Technical Specification is intended to help address all of these issues. These same issues are also relevant to the process of initial material qualification, prior to specification and use, and some of the guidance provided can be used in this context.

Each broad category of manufactured nano-object has been considered in a separate clause: those at the nanoscale in all three (orthogonal) dimensions, those at the nanoscale in two (orthogonal) dimensions and those at the nanoscale in one dimension only. Many nano-objects are supplied in the form of dispersion in a liquid medium. For each broad category of nano-object, the characteristics relevant to dispersions have therefore also been identified.

For each category of manufactured nano-object:

- a list is provided of material characteristics that are considered relevant to initial material qualification in all areas of application;

- a list is provided of additional material characteristics that are considered relevant to material qualification in specific areas of application;

- where the use of these material characteristics in agreed specifications does not ensure batch-to-batch or lot-to-lot consistency, a further list of characteristics that might have an influence on product performance and/or downstream processing is provided for consideration;

- for all identified material characteristics, appropriate measurement methods are proposed, which separate into two categories:

  a) those generally utilising relatively low-cost equipment, which can be envisaged for use in routine batch or lot quality control in an industrial environment, and

  b) those which require specialist equipment and which might therefore only be viable for use in less frequent assessments;

- a brief description of the basis of each measurement method is provided and, wherever possible, reference is made to an appropriate source of guidance on good practice in carrying out the test (usually an established standard); where no viable or validated measurement method can currently be identified,

this is also stated. For some parameters, guidance on an appropriate measurement method is not quoted because no International Standard exists for measuring such a parameter. Often, however, International Standards do exist which refer to the measurement of the parameter for specific materials or applications. Such material- or application-specific standards can be identified by searching the ISO standards database (www.iso.org).

A decision tree is provided in Annex A as a guide to the use of this Technical Specification.

Users of this Technical Specification should be aware that packaging, labelling and transport of materials specified in this Technical Specification may be subject to statutory and regional regulations.

It has been assumed in the preparation of this Technical Specification that the execution of its provisions will be entrusted to appropriately qualified and experienced people, for whose use it has been produced.

TECHNICAL SPECIFICATION **ISO/TS 12805:2011(E)**

Nanotechnologies — Materials specifications — Guidance on specifying nano-objects

1 Scope

This Technical Specification provides guidance on the preparation of specifications for the characteristics of manufactured nano-objects and their measurement methods. This is intended to help ensure the delivery of products with consistent properties for subsequent processing and/or final product performance.

This Technical Specification includes guidance on specifying the physical and chemical characteristics of manufactured nano-objects, which might affect performance or subsequent processing. A list of applicable measurement methods is given in Annex B.

NOTE 1 The nano-objects can be supplied in a dry form or as dispersions in a liquid medium.

Guidance on specifying the environmental, health and safety (EHS) characteristics of manufactured nano-objects is outside the scope of this Technical Specification.

NOTE 2 Nanotechnology is a rapidly growing and evolving field. It is therefore good practice for users of this Technical Specification to maintain an awareness of the legislative environment and latest developments in EHS regarding nanotechnology (see References [1][2][3][4][15][16][38][39][40][41][42][43]). If the customer or supplier wishes to assess the environmental, safety or health risks of the material, they can refer to ISO/TR 13121 and ISO/TR 12885 for further guidance.

This Technical Specification also does not include guidance on specifying materials containing nanosized phases formed in situ by a transformation in the material, e.g. Guinier-Preston zones in precipitation hardening metals. Furthermore, it does not specify quantitative requirements for the object to be considered a nano-object, but lists appropriate examples of characteristics and properties and their measurement methods useful for specifying nano-objects. Characteristics and measurement methods for nano devices are not included.

Although this Technical Specification refers to parameters, which could be considered aspects of material quality, it is not intended to provide guidance on the establishment of quality management systems. For guidance on quality management systems, reference can be made to ISO 9000.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 27687, *Nanotechnologies — Terminology and definitions for nano-objects — Nanoparticle, nanofibre and nanoplate*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 27687, ISO/TS 80004-1 and the following apply.

3.1
nano-object
material with one, two or three external dimensions in the nanoscale

3.2
nanoscale
size range from approximately 1 nm to 100 nm

3.3
manufactured nano-object
nano-object intentionally produced for commercial purposes to have specific properties or composition

4 Specifying manufactured nano-objects

4.1 General

Product specifications are an important component of the information a supplier provides to the marketplace, which also includes items such as trade names, product grades, product information sheets, material safety data sheets, case histories, testimonials and advertising.

For the supplier, product specifications reflect production capabilities and are used to differentiate product grades. For the customer, product specifications are used to differentiate among alternative suppliers or materials. They are used to set specifications for the end-use product. For both supplier and customer, product specifications are used in firm-wide quality systems such as ISO 9001 to ensure consistent manufacture. They form the basis for commercial activities such as complaint resolution, product recall, guarantees and warranties.

There are instances where nano-objects can fail to generate a consistent response to downstream processing or consistent performance when incorporated into the final product, for reasons that are not fully understood by either supplier or customer. It is important, in these instances, that the two parties have a basis for working together to develop and agree a specification which is capable of eliminating these inconsistencies. The guidance in this Technical Specification follows standard industry practice of suppliers of powdered products. There is a general, all-applications clause focusing on product identity, shape and particle size, intended to determine what the product is, and whether it is nanoscale. These characteristics are provided for discrete solid nano-objects supplied in either dry or dispersion form and categorised by the number of dimensions at the nanoscale (3D nanoparticles; 2D nanofibres; 1D nanoplates**)**.

Clause 5 addresses those situations where greater information is required between the supplier and the customer. In 5.2, nano-object characteristics that are known to be influential in specific areas of application are identified, while 5.3 proposes further characteristics that could be investigated if those provided in Clause 4 and 5.2 have not been sufficient to ensure a reproducible processing response or consistent product performance.

4.2 Nano-objects having all three dimensions at the nanoscale, i.e. nanoparticles

The characteristics given in Table 1, provided as typical values or formal specifications, are useful in describing manufactured nanoparticles in all areas of application. The numbers in parentheses refer to proposed measurement methods, described in Tables B.1 and B.2. If there is no number, there is no generic specification or guidance appropriate to the measurement of the characteristic.

Table 1 — Characteristics useful in describing manufactured nanoparticles

Characteristic	Dry form	Dispersed form
Chemical composition, including surface functionalization, cross-section for particles with a core-shell structure (1.9,1.13, 1.14, 2.9 and 2.10)	Yes	Yes
Specific surface area (1.4)	Yes	Yes
Mean particle size and particle size distribution (1.1 and 2.1)	Yes	Yes
Mean primary crystalline particle size and size distribution (1.2 and 2.4)	Yes (if crystalline)	Yes (if crystalline)
Degree of agglomeration or aggregation (1.3 and 2.5)	Yes	Yes
Continuous phase of the dispersion	N/A	Yes
pH	N/A	Yes, for aqueous dispersions
Shelf life	Yes (if sensitive to storage conditions)	Yes
Specific gravity (or % solids)	N/A	Yes

4.3 Nano-objects having two dimensions at the nanoscale, i.e. nanofibres

Nanofibres may be either solid (nanorods, nanowires) or hollow (nanotubes). The characteristics given in Table 2, provided as typical values or formal specifications, are useful in describing manufactured nanofibres in all areas of application. The numbers in parentheses refer to proposed measurement methods, described in Tables B.1 and B.2. If there is no number, there is no generic specification or guidance appropriate to the measurement of the characteristic.

Table 2 — Characteristics useful in describing manufactured nanofibres

Characteristic	Nanorod or nanowire in dry form	Nanotube in dry form	Dispersed form
Chemical composition including chemical purity (and dopants if added), surface functionalization, cross-section for particles with a core-shell structure (1.9, 1.13, 1.14, 2.9, 2.10 and 2.11)	Yes	Yes	Yes
Mean length and length distribution (2.15)	Yes	Yes	Yes
Mean diameter and diameter distribution (2.14)	Yes	Yes	Yes
Mean aspect ratio and aspect ratio distribution (2.16)	Yes	Yes	Yes
Degree of agglomeration (1.3 and 2.5)	Yes	Yes	Yes
Specific surface area (1.4)	Yes	Yes	Yes
Number of walls, i.e. single, double or multi-walled (2.17)	N/A	Yes	Yes

Table 2 (*continued*)

Characteristic	Nanorod or nanowire in dry form	Nanotube in dry form	Dispersed form
Mean wall thickness and wall thickness distribution (2.17)	N/A (same as fibre)	Yes	Yes (for tubes only)
Catalyst residue (1.9 and 2.11)	N/A	Yes	Yes
Structural carbon purity	N/A	Yes, for carbon	Yes, for carbon
Continuous phase of the dispersion	N/A	N/A	Yes
Specific gravity	N/A	N/A	Yes
pH	N/A	N/A	Yes, for aqueous dispersions
Shelf life	Yes (if sensitive to storage conditions)	Yes (if sensitive to storage conditions)	Yes

4.4 Nano-objects having one dimension at the nanoscale, i.e. nanoplates

The characteristics given in Table 3, provided as typical values or formal specifications, are useful in describing manufactured nanoplates in all areas of application. Nanoplates occur in a free-standing form (e.g. flakes or exfoliated clays). The numbers in parentheses refer to proposed measurement methods, described in Tables B.1 and B.2. If there is no number, there is no generic specification or guidance appropriate to the measurement of the characteristic.

Table 3 — Characteristics useful in describing manufactured nanoplates

Characteristic	Dry form	Dispersed form
Chemical composition including surface functionalization and crystal structure (1.9, 1.13, 1.14, 2.9 and 2.10)	Yes	Yes
Specific surface area (1.4)	Yes	Yes
Mean particle size and particle size distribution (1.1 and 2.1)	Yes	Yes
Mean primary crystalline particle size and size distribution (1.2 and 2.4)	Yes (if crystalline)	Yes (if crystalline)
Degree of agglomeration or aggregation (1.3 and 2.5)	Yes	Yes
Surface morphology (1.15, 2.2 and 2.13)	Yes	N/A
Continuous phase of the dispersion	N/A	Yes
pH	N/A	Yes, for aqueous dispersions
Shelf life	Yes (if sensitive to storage conditions)	Yes
Specific gravity (or % solids)	N/A	Yes

5 Additional material characteristics that might influence end-product performance and/or downstream processability

5.1 General

Clause 4 has identified nano-object characteristics that should be included in material specifications for all areas of application.

Further characteristics are presented in 5.2 that should be added to specifications in particular areas of application.

Where the evaluation of the characteristics proposed in Clause 4 and 5.2 still does not ensure batch-to-batch or lot-to-lot consistency, in terms of processing response or final product performance it may be necessary to include further characteristics in specifications. Additional characteristics are suggested in 5.3, which might be considered in this context.

5.2 Characteristics known to be influential in specific areas of application

The characteristics in Table 4 should be included in material specifications related to specific areas of application. The numbers in parentheses refer to the guidance notes on measurement methods in Tables B.1 and B.2. If there is no number, there is no generic specification or guidance appropriate to the measurement of the characteristic.

Table 4 — Influential characteristics in specific areas of application

Characteristic	Nanoparticles	Nanofibres	Nanoplates
Dispersibility in solid matrices – in specifying nanoscale reinforcements in composite materials (1.8 and 2.8)	Yes	Yes	Yes
Dispersibility in liquids – polar and non-polar, (1.7)	Yes	Yes	Yes
Fuchs surface area – use of nano-objects in aerosols (2.3)	Yes	Yes	Yes
Symmetry – for electrical properties of nanotubes (2.18)	N/A	Yes	N/A
Strength of interface with matrix in specifying nanoscale reinforcements for composites	Yes	Yes	Yes
Crystallographic and mechanical anisotropy (1.6)	Yes	Yes	Yes

5.3 Other material characteristics with a possible influence on product performance and/or downstream processability

To ensure reproducible behaviour in processing and consistent product performance, it might be necessary to specify additional characteristics from the list in Table 5. The numbers in parentheses refer to the guidance notes on measurement methods in Tables B.1 and B.2. If there is no number, there is no generic specification or guidance appropriate to the measurement of the characteristic.

Table 5 — Other material characteristics which may influence product performance and/or downstream processability

Characteristic	Nanoparticles	Nanofibres	Nanoplates
Particle morphology (2.2)	Yes	N/A	N/A
Flow characteristics (1.10)	Yes	Yes	Yes
Tap density (1.11)	Yes	Yes	Yes
Apparent density (1.12)	Yes	Yes	Yes
Porosity (1.5 and 2.6)	Yes	Yes	Yes
Crystal structure and degree of crystallinity (1.6)	Yes	Yes	Yes
Colour	Yes	Yes	Yes
Transparency	Yes	Yes	Yes
Single agglomerate crushing strength	Yes	Yes	N/A
Structure at nanotube ends (2.17)	N/A	Yes, for tubes	N/A

6 Proposed measurement methods for determining the characteristics of manufactured nano-objects identified as important for specification purposes

Measurement methods separate into two categories:

a) those generally utilising relatively low-cost equipment for use in routine batch or lot quality control in an industrial environment (Table B.1), and

b) those which require specialist equipment and which might therefore only be viable for use in less frequent assessments (Table B.2).

Sampling of powders should be carried out in accordance with ISO 14488.

Many of the analytical methods require dispersion of powders in liquids. Sample preparation involving dispersion in liquids should be carried out following guidance given in ISO 14887.

As many nano-objects are reactive, their physical and chemical properties can be affected by the sampling point and their storage environment. Consequently, the supplier and purchaser should agree the sampling point and storage of the samples for comparability of results.

Material properties are either intrinsic to the material, or defined by the measurement method. Values of method-defined properties cannot be directly compared with values obtained with a different method. In addition, methods to assess an intrinsic property may be biased and lead to results that are different for other methods assessing the same property. Consequently, the results from one measurement method may not be directly comparable with results from a second measurement method.

As the guidance can be used for various types of materials and to obtain a large variety of information, this Technical Specification reviews a large number of types of analyses but remains non-exhaustive and will require updating on a regular basis.

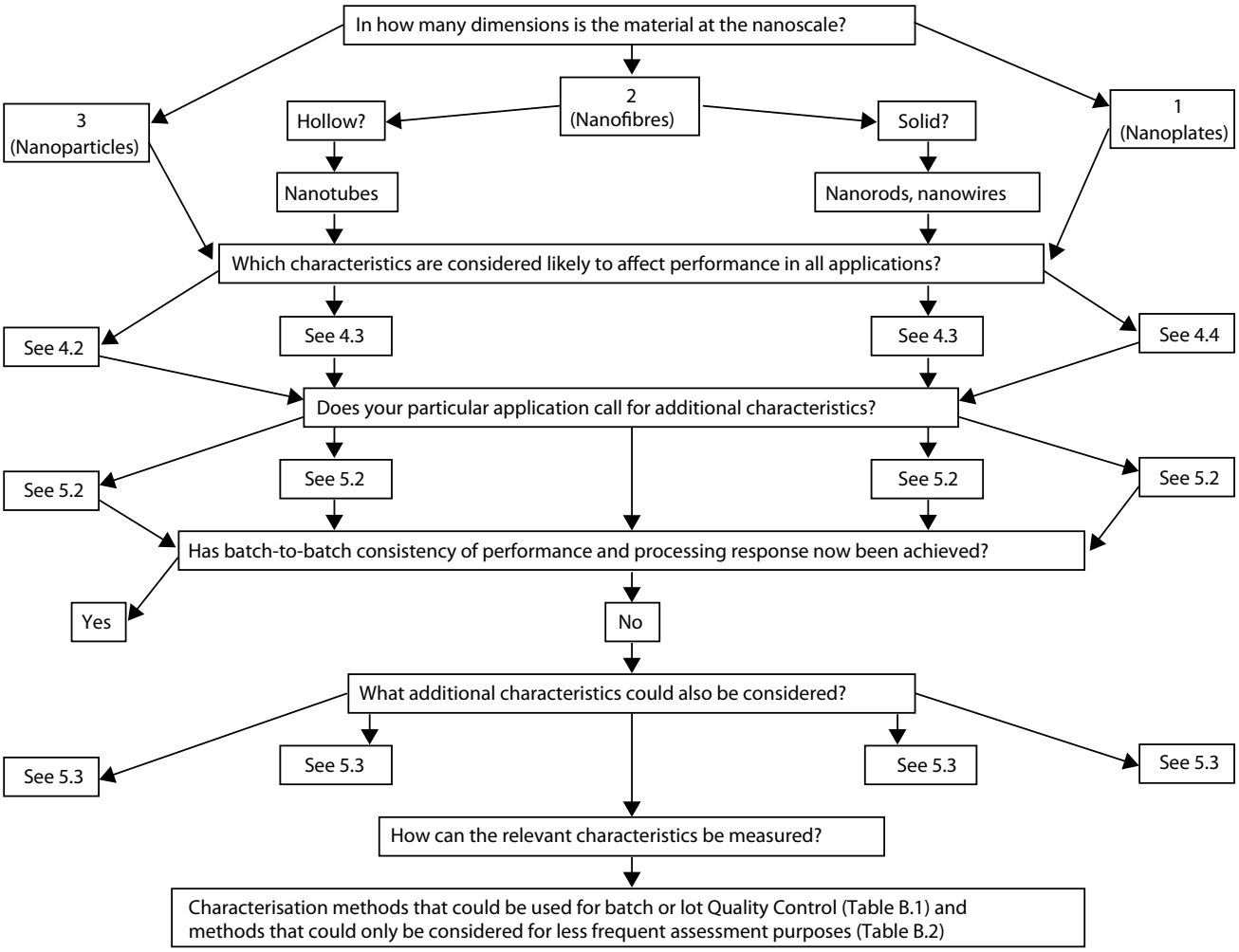
7 Possible impacts of contamination on the properties and performance of manufactured nano-objects and their mitigation

Due to their high surface area, and the associated high surface energy, many nano-objects have a tendency to agglomerate, to adsorb matter on their surface or to stick to the surface of larger objects. Consequently, their physical and chemical properties are easily affected by their environment. The mechanism and rate of deterioration will depend on several variables, including the composition of the nano-object, its morphology, the storage time and storage conditions.

Consequently, mitigation techniques should be specified considering the tendency of the material to deteriorate. Packing and storage conditions should be negotiated between the supplier and customer of the material. Specification of product storage conditions should consider the tendency of the material to be degraded by moisture or heat and the material's reactivity with other materials being stored in the same area.

Annex A
(informative)

Figure A.1 — Decision tree to assist with the use of ISO/TS 12805



Annex B
(informative)

Measurement methods

Table B.1 — Measurement methods for use in routine quality control in an industrial environment

	Characteristic	Method	Guidance
1.1	Mean particle size and particle size distribution	—	Different measuring techniques are likely to yield different results for the same material batch or lot. In specifying the material, supplier and purchaser should agree the technique used to measure the particle size distribution.
		Light scattering and diffraction methods	Applicable for particle diameters greater than about 40 nm, depending on the nature of the particle, and spherical or near spherical particles. Calibration of the equipment and measurement procedure should be in accordance with ISO 13320-1 or ISO 21501-2.
		Photon correlation spectroscopy	This method measures hydrodynamic diameter from Brownian motion. It is applicable to the measurement of particle diameters greater than 3 nm, depending on the test material. Guidelines on the application of the method can be found in ISO 13321 and ISO 22412.
		Electrokinetic sonic amplitude testing	A variant of zeta potential testing, in which an alternating voltage is applied to a suspension to produce a sound wave from the vibration induced in the suspended particles. Depending on the material properties, such as density, particle size distribution information for particles over 100 nm in diameter can be derived from the phase lag of the induced sound wave, whereas particle size distribution for finer nanoparticles (5 nm to 100 nm) can be derived from the attenuation of the amplitude of the sound wave.
1.2	Mean primary crystalline particle size and size distribution	X-ray diffraction line broadening (XRDLB)	XRDLB provides information about the size of small crystalline particles, irrespective of whether these are agglomerated or aggregated into larger particles. The technique measures the size and strain of individual crystals under about 100 nm, where the Debye rings (X-ray lines) generated by the procedure are broadened. It should be noted that line broadening can be caused by strain in the material as well as changes in crystallite size. Guidance can be found in EN 13925-1, EN 13925-2 and EN 13925-3.
1.3	Degree of agglomeration or aggregation	Calculation of agglomeration or aggregation index	Calculation from measurements of mean crystallite size, d, by X-ray diffraction line broadening (1.2) and mean particle size, D, by a particle sizing method (1.1). The agglomeration or aggregation index, T, is given by the ratio, D/d. Differential mobility analysis (DMA) techniques may be used to infer the electrical mobility diameter of aerosolized nanoparticles (primary particles and aggregates/ agglomerates). TEM is also routinely used for this task. NOTE he proposed X-ray diffraction method is only applicable to crystalline materials.

Table B.1 — Measurement methods for use in routine quality control in an industrial environment
(continued)

	Characteristic	Method	Guidance
1.3	Degree of agglomeration or aggregation	Calculation of agglomeration or aggregation index	Calculation from measurements of mean crystallite size, d, by X-ray diffraction line broadening (1.2) and mean particle size, D, by a particle sizing method (1.1). The agglomeration or aggregation index, T, is given by the ratio, D/d. Differential mobility analysis (DMA) techniques may be used to infer the electrical mobility diameter of aerosolized nanoparticles (primary particles and aggregates/agglomerates). TEM is also routinely used for this task. NOTE The proposed X-ray diffraction method is only applicable to crystalline materials.
1.4	Surface area and specific surface area	BET analysis	A technique based on the model developed by Brunauer, Emmet and Teller that allows the surface area of powders to be estimated by the amount of gas that is adsorbed. Typically, nitrogen or carbon dioxide is used, but gases such as krypton or argon may be used for low surface area materials because of their sensitivity (mass gain per unit area). The specific surface area is the ratio of surface area to mass. Guidance on this method can be found in ISO 9277 and ISO 18757.
1.5	Porosity	Pycnometry, for example using pressurized mercury or gas adsorption porosimetry.	Porosity can be measured using a pycnometer (a flask of known volume). A known mass and volume of powder is added to the pycnometer, which is then weighed, filled with a liquid or gas of known density, in which the powder is completely insoluble, and weighed again. The volume of the added liquid or gas can be determined and the difference between the combined volume of the powder and liquid/gas and the volume of the pycnometer gives the pore volume of the powder.
1.6	Crystal structure, degree of crystallinity, crystallographic anisotropy	X-ray diffraction	Guidance can be found in EN 13925-1, EN 13925-2 and EN 13925-3.
1.7	Dispersibility in liquids	Zeta potential methods	Zeta potential is the electrostatic potential at the slipping plane (which marks the region where the liquid molecules surrounding the particle first begin to move with respect to the surface) relative to the potential in the bulk solution. The value of the zeta potential is an indication for the mutual repulsion between particles, which promotes dispersion stability. Guidance on measurement and characterisation of particles by acoustic methods is available in ISO 20998-1.
		Rheometry	Guidelines on the determination of complex shear viscosity using a parallel plate oscillatory rheometer are given in ISO 6721-10.

Table B.1 — Measurement methods for use in routine quality control in an industrial environment
(continued)

	Characteristic	Method	Guidance
1.8	Dispersibility in solid matrices	X-ray diffraction	Guidance can be found in EN 13925-1, EN 13925-2 and EN 13925-3.
1.9	Chemical purity	Thermogravimetric analysis (TGA)	This is a test method performed on samples to determine changes in mass in relation to a change in temperature. TGA is commonly employed to determine degradation temperatures, adsorbed moisture content, levels of inorganic and organic components, decomposition points, e.g. of explosives and solvent residues. The TGA instrument usually consists of a high precision balance with a pan (generally platinum) loaded with the sample. The pan is placed in a small oven with a thermocouple to accurately monitor temperature. The atmosphere may be purged with an inert gas to prevent oxidation or other undesired reactions, or with pure oxygen to measure oxidation-related effects. The instrument is generally computer-controlled. Analysis is carried out by raising the temperature gradually and plotting weight against temperature. As many weight loss curves appear similar, transformation may be required before results can be interpreted. A derivative mass loss curve can be used to identify the point at which mass loss is larger. Several modern TGA devices can vent burn off to a fourier-transform infrared spectrophotometer or mass spectrometer to analyse composition.
1.10	Flow characteristics of powders	Annular shear cell	The available methods are those already established for the characterization of flow of coarser powders. The annular shear cell measures the shear stress and dilatancy of a granular material or a powder.
		Hall flowmeter	This method is based on measurement of the time taken for a given mass of powder to flow through a conical funnel with a specified cone angle and aperture diameter. For metallic powders, guidance on funnel design and experimental procedure is given in ISO 4490.
		Hausner ratio and Carr index	These can be calculated from determined values of tap density (the apparent density of a volume of powder obtained when its receptacle is tapped or vibrated) and apparent density (the weight per unit volume of a powder, in contrast to the weight per unit volume of the individual particles). Hausner ratio equals tap density/apparent density; high values of this ratio indicate poor flow behaviour. Carr index = 100 × (tap density−apparent density) / tap density. These indices are widely used measures of powder flow characteristics, particularly in the pharmaceutical sector.
1.10	Flow characteristics of powders (*continued*)	Jenike shear cell	The Jenike shear cell measures the unconfined strength, internal friction angle, effective internal angle of friction and wall friction angle as functions of consolidation or solid contact pressures.

Table B.1 — Measurement methods for use in routine quality control in an industrial environment *(continued)*

	Characteristic	Method	Guidance
1.11	Tap density of powders	—	The available method is that already established for the measurement of tap density (the apparent density of a volume of powder obtained when its receptacle is tapped or vibrated) of coarser powders. Relevant procedural standards are ISO 3953, which relates to metallic powders, and ISO 23145-1, which relates to fine ceramics. Guidance on measuring the absolute density of ceramic powders is given in ISO 18753.
1.12	Apparent density of powders	—	The available methods are those already established for the measurement of apparent density of coarser powders. These methods involve the filling of a cup of known volume with powder and then weighing the powder collected in the cup. ISO 3923 covers two procedures for the dispensing of the powder into the cup; the funnel method (ISO 3923-1) and the Scott volumeter method (ISO 3923-2). Guidance on measuring the apparent density of ceramic powders is given in ISO 18754
1.13	Surface chemical analysis	—	There are no techniques currently available for surface chemical analysis of manufactured nanomaterials, which can be proposed for batch or lot quality control in an industrial environment. Several techniques, which can be applied to the analysis of surface composition for audit check purposes, are listed in Table B.2.9.
1.14	Bulk chemical analysis	—	Manufacturers should consider the expected composition of material and likely impurities when determining the most appropriate analytical approach to be adopted. There are published standards covering the many aspects of chemical analysis, such as sample preparation, quality of water and analytical reagents.
1.15	Surface morphology	Confocal microscopy	A confocal microscope system provides measurement of the variation in height of the sample (z-axis) as it is moved across the detection plane using an xy translation stage. Such a system uses point illumination and a pinhole in an optically conjugate plane in front of the detector to eliminate out-of-focus information. As only one point is illuminated at a time in confocal microscopy, 2D or 3D imaging requires scanning over a regular raster (i.e. a rectangular pattern of parallel scanning lines) in the specimen.
1.15	Surface morphology *(continued)*	Confocal microscopy (*continued*)	The resolution of the measurements is < 1 μm in x- and y-axes and < 10 nm in the z-axis. Hence microfeatures and topographic variations can be monitored in detail. Sample sizes from a few mm^2 to 100 mm × 100 mm can be measured.
		Electron microscopy	Surface morphology can be measured using scanning electron microscopy and transmission electron microscopy. Guidance on static image analysis methods is given in ISO 13322-1.

Table B.2 — Measurement methods for less frequent assessment purposes

	Characteristic	Method	Guidance
2.1	Mean particle size and particle size distribution	—	Different measuring techniques are likely to yield different results for the same material batch or lot. In specifying the material, supplier and purchaser should agree the technique used to measure the particle size distribution.
		Condensation particle counter	This is the most widely used type of instrument for detecting and counting nanoparticles in aerosols. The instrument operates by condensing vapour, such as alcohol or water, onto sampled particles to grow them to a size range that can be detected by an optical counting technique. The technique is applicable to counting particles with diameters of 100 nm or below.
		Scanning mobility particle sizer	This technique detects and counts nanoparticles and is applicable to the measurement of aerosol size distribution in the range 3 nm to 800 nm. The technique operates by charging particles and separating them based on their mobility when passing between electrodes. The separated particles are detected by either a condensation particle counter or by measuring electrical charge on the particles. SMPS has a subsystem for size classification and a subsystem for particle detection (often a Condensation Particle Counter).
		Electron microscopy and image analysis	Guidelines for the calibration of image magnification can be found in ISO 16700. Guidance on static image analysis methods is available in ISO 13322-1.
2.2	Particle morphology	Electron microscopy	Particle morphology can be measured using scanning electron microscopy and transmission electron microscopy. Guidance on static image analysis methods is available in ISO 13322-1.
		Scanning probe microscopy	Scanning probe microscopy covers several related technologies for imaging and measuring surfaces on a fine scale. A sharp tip mounted on a flexible cantilever is scanned across the object profiling the surface. Atomic force microscopy, scanning tunnelling microscopy and near field scanning optical microscopy are types of scanning probe microscopy.
		Atomic force microscopy	Atomic force microscopy provides shape and structural information of nanoparticles. A very sharp tip (typically 10 nm diameter apex) is moved across the surface in a raster-like fashion while being moved up and down (z direction) to maintain the force between tip and sample constant. The three motions provide a topographical map of the surface being investigated. The technique provides direct measurement in the z direction of dimensions such as height, surface roughness and volume.

Table B.2 — Measurement methods for additional assessment purposes *(continued)*

	Characteristic	Method	Guidance
2.2	Particle morphology (*continued*)	Scanning tunnelling microscopy (STM)	A technique for revealing the apparent electron-density-related atomic structure of surfaces, using a needle-like probe near the object under observation. A tunnelling current, which is measured, is generated by altering the potential at the tip of the probe. A 3D representation of the sample surface is generated by rastering the surface of the object and mapping the position of the tip for constant current level at various points.
		Near field scanning optical microscopy (NSOM)	A technique for imaging surfaces in transmission or reflection by mechanically scanning an optical probe much smaller than the wavelength of light over the surface, while monitoring the transmitted or reflected light. The detector is placed very close to the specimen surface, which allows surface inspection with high spatial resolution.
2.3	Fuchs surface area	Aerosol diffusion charging	A method in which the Fuchs surface area of an aerosol is measured directly, by passing electrically neutral particles through a unipolar ion cloud and measuring the resulting aerosol charge. When the charging rate is low, aerosol charge is proportional to the Fuchs surface area.
		Epiphaniometer	Instrument used to measure the Fuchs surface area of aerosols directly. The aerosol is passed through a charging chamber where lead isotopes created from a decaying actinium source are attached to the particle surfaces. The particles are transported through a capillary to a collecting filter. The amount of radioactivity measured is proportional to the particle surface area.
2.4	Mean crystallite size and crystallite size distribution	Electron backscatter diffraction (EBSD)	This technique can be used to measure crystallite size. EBSD is conducted using a scanning electron microscope (SEM) equipped with a backscatter diffraction camera. Electrons interact with the atomic lattice planes of the crystalline structure of the sample, that satisfy Bragg conditions and undergo backscatter diffraction. Crystallite size is derived from the resulting image, which is formed because of the dependence of the image brightness on crystal orientation.
		Transmission electron microscope diffraction	This technique can be used to measure crystallite size. Electrons interact with the atomic lattice planes of the crystalline structure of the sample, which satisfy Bragg conditions and undergo diffraction. Different lattice orientations, corresponding with different crystallites, will result in different diffraction intensities. Crystallite size is calculated from the resulting pattern.
2.5	Degree of agglomeration or aggregation	Scanning electron microscopy	Guidance on static image analysis methods is available in ISO 13322-1.

Table B.2 — Measurement methods for additional assessment purposes *(continued)*

	Characteristic	Method	Guidance
2.6	Porosity	Electron microscopy and image analysis	Guidelines for the calibration of image magnification can be found in ISO 16700. Guidance on static image analysis methods is available in ISO 13322-1.
2.7	Compatibility with matrices	Raman spectroscopy	Spectroscopy in which the Raman effect is used to investigate molecular energy levels. The Raman effect involves the scattering of light with a change of frequency characteristic of the scattering material, representing a change in the vibrational, rotational or electronic energy of the substance that can be used to give information on the chemical bonding or mechanical stress state.
2.8	Dispersibility in solid matrices	Electron microscopy	Guidelines for the calibration of image magnification can be found in ISO 16700. Guidance on static image analysis methods is available in ISO 13322-1.
2.9	Surface chemical analysis		With the exception of EELS and AES, these techniques have spatial resolution at the nanometre scale in one direction (z-axis), but can only resolve at the micrometre scale in the other two orthogonal directions (x- and y-axes). In the context of this guide, therefore, the techniques are applicable in the majority of cases to nanoplates rather than nanofibres and nanoparticles.
		Auger electron spectroscopy (AES) and scanning Auger microscopy (SAM)	Techniques in which an electron spectrometer is used to measure the energy distribution of Auger electrons emitted from a surface. Guidance on AES can be found in ISO/TR 18394 and ISO 20903.
		Electron energy loss spectroscopy (EELS)	A technique where inelastic interaction of an electron beam with atoms in an electron transparent sample results in an energy distribution spectrum of transmitted electrons that contains compositional and chemical bonding information. EELS provides resolution at the nanoscale in the x- and y-axes.
		Ion beam analysis (IBA)	A method to elucidate composition and structure of the outermost atomic layers of a solid material, in which principally monoenergetic, single-charged probe ions scattered from the surface, are detected and recorded as a function of their energy or angle of scattering or both.
		Secondary ion mass spectrometry (SIMS)	A method in which a mass spectrometer is used to measure the mass-to-charge ratio and abundance of secondary ions emitted from a sample as a result of sputtering by energetic ions. Guidance on the method can be found in ISO 22048 or ISO 18114. SIMS measurements can be conducted in three analysis modes; namely static, dynamic and imaging SIMS. The difference between static SIMS and dynamic SIMS is in the ion dose of the primary beam. Static SIMS involves using sufficiently low primary ion doses so that sample measurements can be made without significant modification to the surface. Imaging SIMS represents an extension whereby the primary beam is rastered across the surface to allow measurement of the distribution of sputtered ion species from different regions of the surface.

Table B.2 — Measurement methods for additional assessment purposes *(continued)*

	Characteristic	Method	Guidance
2.9	Surface chemical analysis (*continued*)	Dynamic secondary ion mass spectrometry (D-SIMS)	Dynamic secondary ion mass spectrometry provides elemental and simple molecular information from the surface through to several micrometres. The technique can be used for depth profiling elemental or molecular distributions to sensitivities of ppb.
		Time of flight secondary ion mass spectrometry (ToF-SIMS)	This method is highly surface sensitive where the sampling depth is only 1 to 2 monolayers (often < 2 nm). It is analytically very sensitive (often trace detection levels) but it is not quantitative. Its chemical specificity, however, may be exploited in a semi-quantitative manner where variations in the surface concentrations of different chemical species can be followed by monitoring changes in the relative intensities of their diagnostic ToF-SIMS signals.
		Total reflection X-ray fluorescence spectroscopy (TXRF)	A method in which an X-ray spectrometer is used to measure the energy distribution of fluorescence X-rays emitted from a surface irradiated by primary X-rays under the condition of total reflection.
		Ultra-violet photoelectron spectroscopy (UPS)	A method in which an electron spectrometer is used to measure the energy distribution of photoelectrons emitted from a surface irradiated by ultra-violet photons.
		X-ray photoelectron spectroscopy (XPS)	A method in which an electron spectrometer is used to measure the energy distribution of Auger and photoelectrons emitted from a surface irradiated by X-ray photons. It is an extremely surface sensitive non-destructive technique that provides qualitative and quantitative chemical information of sample surfaces for all elements, except hydrogen and helium. Guidance on the method can be found in ISO 20903. XPS is also commonly referred to as ESCA (Electron Spectroscopy for Chemical Analysis).
2.10	Bulk chemical analysis	Glow discharge mass spectrometry (GDMS)	A method in which a mass spectrometer is used to measure the mass-to-charge quotient and abundance of ions from a glow discharge generated at a surface.
		Glow discharge optical emission spectrometry (GDOES)	A method in which an optical emission spectrometer is used to measure the wavelength and intensity of light emitted from a glow discharge generated at a surface. Guidelines of practices that should be followed in GD OES analyses are described in ISO 14707.

Table B.2 — Measurement methods for additional assessment purposes *(continued)*

2.11	Chemical purity	Ultra Violet/Visible/Near Infra Red (UV/Vis/Near IR) Spectroscopy	This is a combination of absorption spectroscopy techniques, which utilise electromagnetic radiation in the ultraviolet, visible light and near infrared wavelength ranges respectively. Absorption spectroscopy involves measuring and comparing the power of a beam of radiation before and after interaction with a sample and is often combined with a modulation technique, most often wavelength modulation but occasionally frequency modulation, in order to reduce the noise in the system. Instrumentation consists of a source of the radiation, a detector and a dispersive element (such as a prism or, more commonly, a diffraction grating) to allow the intensity at different wavelengths to be recorded. Many commercial instruments use the same detector for recording spectra in all three wavelength ranges. ISO/TS 10867 provides guidelines for the characterization of single-wall carbon nanotubes (SWCNTs) using near infrared (NIR) photoluminescence (PL) spectroscopy. This method can be expanded to estimate relative mass concentrations of semi-conducting SWCNTs in a sample from measured integrated PL intensities and from knowledge of their PL cross-sections.
2.12	Single agglomerate crushing strength	—	Techniques to measure the crushing strength of a single agglomerate are under development. For example, a relatively large (greater than 63 micrometres diameter) single agglomerate is placed on a transverse beam and the load versus deformation curve is measured until the agglomerate breaks into pieces.
2.13	Surface morphology	Atomic force microscopy (AFM)	A technique for imaging surfaces by mechanically scanning their contours using a microfabricated probe. A very sharp tip (typically 10 nm diameter apex) is moved across the surface in a raster-like fashion while being moved up and down (z direction) to maintain the force between tip and sample constant. The three motions provide a map of the surface being investigated. The technique provides direct measurement in the z direction of dimensions such as height, surface roughness and volume.
		Near field scanning optical microscopy (NSOM)	A technique for imaging surfaces in transmission or reflection by mechanically scanning an optical probe much smaller than the wavelength of light over the surface, while monitoring the transmitted or reflected light. The detection optic is located in the optical near field at the specimen surface, which allows surface inspection with high spatial resolution.

Table B.2 — Measurement methods for additional assessment purposes *(continued)*

2.13	Surface morphology *(continued)*	Scanning tunnelling microscopy (STM)	A technique for revealing the apparent electron-density-related atomic structure of surfaces, using a needle-like probe near the object under observation. A tunnelling current, which is measured, is generated by altering the potential at the tip of the probe. A 3D representation of the sample surface is generated by rastering the surface of the object and mapping the distance for constant current level at various points.
2.14	Mean diameter and diameter distribution of nanofibres	Raman spectroscopy	Spectroscopy in which the Raman effect is used to investigate molecular energy levels. The Raman effect involves the scattering of light with a change of frequency characteristic of the scattering substance, representing a change in the vibrational, rotational or electronic energy of the substance. The magnitude of the effect can be used to interpolate the diameter of carbon nanotubes.
		Scanning tunnelling microscopy	This technique can be used to measure the diameter of individual carbon nanotubes.
2.15	Mean length and length distribution of nanofibres	Scanning electron microscopy	Fibre length can be measured by imaging from fibres spun coated onto suitable substrates. Guidelines for the calibration of image magnification can be found in ISO 16700.
2.16	Mean aspect ratio and aspect ratio distribution of nanofibres		The aspect ratio of fibre is the ratio of length to diameter. See measurement methods in 2.14 and 2.15.
2.17	Mean wall thickness and wall thickness distribution, and end structures of nanotubes	Transmission electron microscopy	Wall thickness can be measured by imaging cross sections of fibres.
2.18	Symmetry	—	Measurement techniques for symmetry in carbon nanotubes are under development. For example, researchers report using scanning tunnelling microscopy, electron backscatter diffraction and Raman spectroscopy, but there is no established, widely applied technique. ISO/TS 10867 provides a measurement method for the determination of the chiral indices of the semi-conducting single-wall carbon nanotubes in a sample and their relative integrated photoluminence intensities.

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