## TECHNICAL SPECIFICATION

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# Nanotechnologies — Materials specifications — Guidance on specifying nano-objects

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

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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; TANDARD PREVIEW
- 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order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn.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 tandards item.

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,

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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 (<a href="https://www.iso.org">www.iso.org</a>).

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.

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### 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 nanoobjects 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 strey can refer to ISO/TR 13121 and ISO/TR 12885 for further guidance.

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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. https://standards.iteh.ai/catalog/standards/sist/ab393717-6401-4cf9-a5e1-730ab6f9e832/iso-ts-12805-2011

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. http://phere.is.no.jonephere.is.no.jon

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
рН	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.

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Table 3 — Characteristics useful in describing manufactured hanoplates

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 (standard	Nanoparticles S.Iteh.al)	Nanofibres	Nanoplates
Dispersibility in solid matrices – in specifying nanoscale reinforcements in composite materials (1.8 and 2,8)/standards.iteh.ai/catalog/standards.	<b>Yes</b> <u>805:2011</u> ds/sist/ab393717-6401	Yes 4cf9-a5e1-	Yes
Dispersibility in liquids – polar and non-polar, (1.7)	-ts-12805 <b>γe's</b> 1	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.

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