TECHNICAL SPECIFICATION

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Nanotechnology — Nanoparticles in powder form — Characteristics and measurements

Nanotechnologies — Nanoparticules sous forme de poudre — Caractéristiques et mesures

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Foreword

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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. www.iso.org/directives

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The committee responsible for this document is ISO/TC 229, *Nanotechnologies*.

Introduction

As is commonly noticed for every technology concerned with the development of new materials, and for nanotechnology in particular, information sharing on material characteristics by sellers and buyers, and sometimes also by regulators, is important and is facilitated by the development of appropriate material specifications. For a comprehensive exchange of information, it is essential to agree on the description of the material characteristics. However, many characteristics of nanomaterials cannot be determined using general and well-established measurement methods. This may become the cause for inconsistency in experimental results and induce confusion in commerce and technology transfer. Furthermore, the rapid discovery of new materials from nanotechnology increases the number of characteristics that need to be specified for an appropriate dissemination of information.

In order to address this need, a systematic arrangement of characteristics has been carried out across different fields of application specific to each nano-object by identifying a list of fundamental characteristics commonly used in these circumstances and by developing tailor-made technical specifications for the list, as seen in ISO/TS 11931 and ISO/TS 11937.

Another approach that was followed for ISO/TS 12805 resulted in the development of a list of characteristics applicable to specifying nano-objects that are useful to the wider community of users of information on nano-objects.

To increase the reach of outcomes from these efforts, members of ISO/TC 229 have discussed and planned the systematic development of an ISO technical specification for defining a list of fundamental characteristics that are widely applicable to a broad range of nano-objects. This Technical Specification is intended to define the list of fundamental characteristics universally for nanoparticles in powder form, which covers a very broad range of nano-objects.

In order to develop a common understanding among sellers, buyers, and regulators, this Technical Specification uses the chemical composition, crystal structure, particle size, and surface area as fundamental measures for characterizing nano-objects from a chemical, physical, and surface scientific point of view that is of significant interest to users of the nano-objects. However, since measurement procedures used for determining the characteristics of nano-objects often rely on various idealized assumptions, the resulting characteristics of nano-objects with identical name may not guarantee the equivalence of measured results. This issue can be addressed by adopting well-recognized measurement methods that can provide reliable measurement results.

The measurement methods adopted in this Technical Specification are well known in the industry. Instruments used for measurement and data processing software are well developed and provide reliable measurement results when operated under a valid quality system.

The description of measurement methods is limited in this Technical Specification to important supplementary notices. For basic information about applying the methods, it is assumed that operating instructions are provided with any instruments, appropriate data processing software is available, and analysis has the required technical skills. The methods are applicable to situations where procedures are subcontracted to independent test laboratories. Since quantitative criteria concerning characteristics depend on the specific intentions among users, they are not described in this Technical Specification. These criteria are subject to agreement between users of this Technical Specification, namely, sellers, buyers, and regulators of nanoparticles in powder form.

Nanotechnology is a rapidly growing and evolving field. Users of this Technical Specification should maintain familiarity with the legislative environment and latest developments in human and environmental health and safety regarding nanotechnology.

If the seller or the buyer wishes to assess the environmental, safety, or health risks of the material, they may refer to ISO/TR 12885:2008 for further guidance. 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.

Nanotechnology — Nanoparticles in powder form — Characteristics and measurements

1 Scope

This Technical Specification lists fundamental characteristics which are commonly determined for nanoparticles in powder form. The Technical Specification prescribes specific measurement methods for each of these characteristics.

This Technical Specification does not specify acceptable quantitative criteria for the characteristics because they are subject to agreement between sellers, buyers, and regulators.

Excluded in this Technical Specification are characteristics specifically related to health, safety, and environmental issues, as well as characteristics that pertain to specific applications of nanoparticles in powder form.

2 Normative references

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

ISO 9277:2010, *Determination of the specific surface area of solids by gas adsorption — BET method*

ISO 13322-1, *Particle size analysis — Image analysis methods — Part 1: Static image analysis methods*

ISO 14488, *Particulate materials — Sampling and sample splitting for the determination of particulate properties*

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

3 Terms and definitions

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

3.1
transmission electron microscopy
TEM
method that produces magnified images or diffraction patterns of the specimen by an electron beam which passes through the specimen and interacts with it

[SOURCE: ISO 29301:2010]

3.2
X-ray diffraction
XRD
method to determine crystallographic and geometrical information about a sample by observing the diffraction pattern due to an X-ray beam scattered by a sample

3.3
specific surface area
absolute surface area of the sample divided by sample mass

[SOURCE: ISO 9277:2010, definition 3.11]

Note 1 to entry: In this Technical Specification, the absolute surface area is estimated by measuring the amount of physically adsorbed gas using the BET method.[14]

3.4
Feret diameter
distance between two parallel tangents on opposite sides of the image of a particle

[SOURCE: ISO 13322-1:2004]

4 Fundamental characteristics with corresponding measurement methods

The fundamental characteristics of nanoparticles in powder form are listed in Table 1, with unit and measurement method for each characteristic. The characteristics shall be measured using each measurement method listed and their results shall be reported as specified in Clause 7.

Characteristics 1, 2, 3, and 4 shall be measured for crystalline nanoparticles in powder form.

Characteristics 1, 2, and 5 shall be measured for non-crystalline nanoparticles in powder form.

NOTE If necessary, characteristic 5 may also be measured for crystalline nanoparticles in powder form.

Table 1 — Fundamental characteristics with units and corresponding measurement methods

Characteristics	Unit	Measurement methods (for detail, see Clause 6)
1) Chemical composition (in terms of the measurand, mass fraction of substance)	1 or g/g	analysis shown to provide metrologically traceable results
2) Specific surface area	m^2/g	BET method
3) Composition of crystal structure (in terms of the measurand, molar fraction of the substance)	1 or mol/mol	XRD method
4) Average crystallite size	nm	XRD (Scherrer formula) method
5) Average and standard deviation of the measured primary particle sizes	nm	TEM method

5 Sample preparation

The sample subjected to a measurement method shall be chosen as to be representative of the parent population of the nanoparticles in powder form. ISO 14488 applies to sampling and sample splitting procedure.

Any influences of the sampling process on the measured characteristics of the nanoparticles shall be estimated. Corrections for such influences shall be applied or appropriate components of uncertainty shall be incorporated. For example, mechanical stress may break up aggregates and/or agglomerates, changing the results of size distribution measurements. Such stress can also induce crystal strain, introducing deviations in average crystalline size measurements in the XRD (Scherrer formula) method.

NOTE 1 For more general information about sampling procedures, see also ISO 2859.

NOTE 2 Some measurement methods, sample preparation, and pre-treatment procedures may heavily influence characteristics other than those to be measured using those methods. Therefore, special care should be taken in designing test procedures. For example, dispersion processes that are used prior to TEM analysis may significantly influence specific surface area measurements. On the other hand, samples prepared for some test methods can be used for other test methods. For instance, a sample prepared for measurement of crystal structure using the XRD method can be used for measurement of average crystalline size using the XRD (Scherrer formula) method, without concern of bias introduced by the sample preparation procedure.

Handling and storage of the sample should be subject to the instruction given by the suppliers. The testing laboratory should consult the supplier regarding the optimum condition to prepare and treat samples.

6 Measurement methods

For surface-modified nanoparticles, including those having modified surface coating, and for nanoparticles with modified aggregates and/or agglomerates, such modifications may introduce significant deviations in the characteristics of a sample relative to those of the original nanoparticles. Therefore, it shall be clearly stated whether the reported characteristics pertain to the nanoparticles before or after modification.

Measurements shall be made under a well-recognized quality system, regardless of whether the testing laboratory is in-house or associated with an independent third party.

6.1 Chemical composition

Depending on the chemical substance of interest, one or more appropriate analytical methods, selected from the following list, shall be applied to the measurement of the chemical composition, together with necessary pre-treatment and quality control procedures, maintaining metrological traceability. The analytical methods are

— titrimetry,

— gravimetry,

— X-ray fluorescence spectrometry (XRF),

— inductively coupled plasma-mass spectrometry (ICP-MS),

— inductively coupled plasma-optical emission spectrometry (ICP-OES),

— high-performance liquid chromatography (HPLC),

— gas chromatography mass spectrometry (GCMS),

— nuclear magnetic resonance (NMR),

— atomic absorption spectrometry (AAS),

— X-ray photoelectron spectroscopy (XPS),

— Fourier transform infrared spectroscopy (FTIR),

— attenuated total reflectance-infrared spectroscopy (ATR-IR),

— attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR), and

— secondary ion mass spectrometry (SIMS).

Suitable certified reference materials, if available, shall be used for the required calibration and proficiency testing. When available, powder reference materials should be used for validation of the measurement method.

Examples of selected application can be found in ISO 3262-6:1998 for calcium carbonates and in ISO 591-1:2000 for titanium dioxides.

6.2 Specific surface area by BET method

Measurement method, ISO 9277:2010 applies to the measurement of specific surface area. ISO 18757 provides some useful detailed information but for specific materials. Measuring instruments for the BET method are commercially available.

Metrological traceability should be maintained. Reference materials are available for application of the BET method to nanoparticles in powder form.

6.3 Crystal structure by XRD method

Measurement methods, as described in EN 13925-1:2003 and JIS K 0131:1996, apply to crystal structure determination. Measuring instruments for X-ray diffraction (X-ray diffractometers), together with relevant data analysis software, are commercially available.

The wavelength of characteristic X-rays should be referred to a reliable database. The reference value for lattice spacing should be referred to a reliable database or to the certificates of any powder reference materials used.

6.4 Average crystallite size by XRD (Scherrer formula) method

Measurement methods, as described in EN 13925-1:2003 and JIS K 0131:1996, apply to average crystallite size. X-ray diffractometers, together with relevant data analysis software, are commercially available. Application of the Scherrer formula method is intended to follow the measurement of crystal structure because the same sample and instrumentation can be used for both measurements.

6.5 Average and standard deviation of the measured primary particle sizes by TEM method

TEM shall be performed using commercially available measuring instruments. References ISO 14488, ISO 14887, and ISO 2859 apply to sample preparation and ISO 13322-1, or its equivalent, to image processing. The primary particles should be identified by image processing. Their sizes should be estimated as an equivalent spherical diameter or as one or a combination of the Feret diameters of the nanoparticles on the TEM image. The average primary particle size and its standard deviation should be calculated from the distribution of the chosen diameters obtained over the sample.

The calibration of the scale of the TEM image shall be accomplished using nanoscale reference materials or a database containing well-known dimensions, such as lattice spacings or materials in the TEM view.

7 Test report

The test report shall include the following information:

a) all details generally necessary to identify the product tested (product name, chemical name);

b) a reference to this Technical Specification (i.e. ISO/TS 17200);

c) the measurement methods used for characteristic measurements;

d) for characteristics 1, 2, 3, and 4, the results of measurements (number of samples measured, averages, and standard deviation);

e) for characteristic 5, the results of measurement (number of particles measured, average, and standard deviation of primary particle size);

f) any deviation from the measurement methods;

g) the relationship between sample applied to the measurements and product tested, to which characteristics are assigned;

h) the date of test, name of testing laboratory, and statement on the quality system of testing laboratory;

i) uncertainty (especially if the agreement between sellers, buyers, and regulators requests it);

j) any other special information supporting the reliability of measurement results (for example, objective evidence of the quality of measurements performed by the testing laboratory, such as results of proficiency testing exercises or accreditation certificates).

The requirements for the test report in this clause should be observed and any other requirements in the normative references should be disregarded instead.