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Nanotechnologies — Guidance for developing representative test materials consisting of nano-objects in dry powder form

Nanotechnologies — Directives de développement de matériaux d'essai représentatifs constitués de nano-objets sous forme de poudre sèche

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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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Contents

Page

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. www.iso.org/directives

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

Introduction

As new manufactured nano-objects are developed and find a wider range of industrial applications, the roles of physico-chemical, performance, and safety testing (hereinafter “testing”) of their powders (i.e. dry, unsuspended accumulations of such objects) have become more important than ever. However, many testing methods are still under development and have to be properly evaluated in terms of their reliability. Where possible, validation of new measurement methods is performed using certified reference materials, which have known and quantified properties. In the absence of certified reference materials, one often has to rely on non-certified reference materials, with assigned but not certified property values. However, in developing fields of measurement and testing, such as that of nanotechnology, even non-certified reference materials are scarce. In such cases, ‘test materials,’ which are evaluated for homogeneity and stability of one or several of their properties, will be helpful in efforts to improve the reproducibility of testing methods across testing laboratories and the comparability of test results across different test methods[9]. This document specifies that for dry powders of nano-objects the following minimum information can be gathered and provided in a verification report to qualify the material as a nanoscale representative test material:

— information describing the manufacturing process;

— information on the quality management of its manufacturing process;

— data from physico-chemical measurements representing the principal features of the representative test material; and,

— data on the stability and homogeneity of the above parameters.

Compliance with this document, expressed in the form of a verification report, will provide a level of assurance that the representative test material is homogeneous, statistically representative of the manufacturing process, and has stability. This will increase the likelihood that measurements that are undertaken on the representative test material, whether for safety or function, are comparable across testing laboratories, even for properties for which methods are being developed and for which homogeneity and stability have not been quantitatively assessed.

Nanotechnologies — Guidance for developing representative test materials consisting of nano-objects in dry powder form

1 Scope

This Technical Specification provides guidance for developing representative test materials consisting of nano-objects in dry powder form, to enable test method development and improve comparability of data for nanotechnology applications. This guidance includes the physico-chemical properties (specifically, size and shape, specific surface area, crystal structure, and bulk chemical composition) that are required to be measured and reported with the representative test material.

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

ISO/TS 80004-1:2010, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO Guide 31, *Reference materials — Contents of certificates, other documentation and labels*

ISO Guide 35, *Reference materials — General and statistical principles for certification*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1:2010 and the following apply.

3.1
representative test material
RTM
material, which is sufficiently homogenous and stable with respect to one or more specified properties, and is implicitly assumed to be fit for its intended use in the development of measurement and test methods that target properties other than those for which homogeneity and stability have been demonstrated

Note 1 to entry: An RTM may be a reference material[1] for other properties (i.e. properties for which homogeneity and stability have been demonstrated), and a candidate reference material for the target property.

Note 2 to entry: An RTM can be a useful tool in inter- or intra-laboratory developments of test methods for which reference materials cannot (yet) be produced.

3.2
primary particle
particle not formed from a collection of smaller particles

3.3
stability
ability of a representative test material, when stored under specified conditions, to maintain a stated property value within specified limits for a specified period of time

[SOURCE: Adapted from ISO Guide 30:1992]

4 Specific physico-chemical characterization requirements for representative test materials consisting of nano-objects in dry powder form

4.1 General

Following physico-chemical properties shall be evaluated in order to represent the principal features of the representative test material consisting of nano-objects in dry powder form.

4.2 Properties and measurement methods

4.2.1 Size and shape of nano-objects

4.2.1.1 Size distribution of primary particles and of their aggregates

A particle counting-based method shall be performed for measurement of the size distribution of primary particles and their aggregates. An example of measurement method is transmission electron microscopy (TEM). Scanning electron microscopy (SEM) can be used if the SEM resolution is sufficient to measure the particle size of interest. The method or calibrant used to calibrate the sizing method should be given, as an indication of the metrological traceability of the obtained data.

One critical issue is sample preparation, in particular, the dispersion of agglomerated particles and deposition onto the substrate without overlapping of particles. A defined dispersion procedure shall be followed and details thereof reported.

Another critical issue is statistical representativeness of the particles selected for analysis. Depending on the width of the size distribution and on the particle shape, a large number of particles might need to be counted to obtain sufficient statistical reliability.

Typically two different types of particle size distributions will be determined. The primary particle size determined from free primary particles and by measuring the primary particles making up agglomerates and aggregates. The types of particle size distribution shall be reported. The resulting size distribution shall be reported in a table and/or a graphical representation (histograms, density distributions or cumulative distributions) according to ISO 9276-1.

NOTE Use of ISO 13322-1 is recommended as a comprehensive guide for image analysis.[2] It also provides information about the number of particles to be measured.

4.2.1.2 Representative size of primary particles and of their aggregates

The representative size of primary particles and of their aggregates shall be derived from the size distribution (4.2.1.1). It may be reported as, for example, mean and standard deviation, and/or 10, 50, and 90th percentiles of the cumulative size distribution.

ISO 9276-2 to ISO 9276–3 should be used for the expression of the representative size.[3],[4]

4.2.1.3 Shape of primary particles and of their aggregates

The shape of primary particles and of their aggregates shall be deduced from imaging techniques, such as TEM, SEM or atomic force microscopy (AFM). A brief, qualitative description shall be done using clearly-defined or widely-acknowledged vocabulary.

NOTE 1 Attention should be paid that TEM provides 2D-projected image of nano-objects and SEM and AFM provide surface shape of nano-objects. Image acquisition with AFM requires special attention, because tip geometry can cause significant ambiguity of particle shape.

NOTE 2 Examples of the description are: spherical, spheroidal, polygonal, ellipsoidal, spherical aggregates, fractal aggregates. Some useful guidance is given in ISO 3252 and ISO/TS 80004-3.[5],[6] More detailed, quantitative description can be made using shape factors defined, for example, in ISO 9276-6.[7]

4.2.1.4 Photomicrographs showing representative primary particles and their aggregates

Representative TEM, SEM or AFM images that illustrate both primary particles and their aggregates shall be acquired. To ensure representativeness, take several photomicrographs of the specimen to show the size and shape of different nano-objects. Sample identification and magnification scale shall also be presented. Instrumental settings and preparation method should be reported if they are different from those employed in 4.2.1.1.

4.2.2 Specific surface area

The specific surface area of the representative test material shall be determined, for example, by gas adsorption using the BET method as described in ISO 9277.[11]

4.2.3 Bulk chemical composition

The bulk chemical composition of the representative test material shall be measured. Depending on the material, any suitable method(s) (e.g. titrimetry, gravimetry, X-ray fluorescence spectrometry, inductively coupled plasma-mass spectrometry, inductively coupled plasma-atomic emission spectrometry or atomic absorption spectrometry) can be used. The results shall be provided with a metrological traceability statement, where appropriate.

4.2.4 Crystal structure

The structure of the nano-objects constituting the representative test material shall be characterized. In the case that the nano-objects are crystalline, crystalline phase (e.g. rutile and anatase in titanium dioxide) shall be identified by X-ray diffraction. In the case that the nano-objects are not crystalline (i.e. 'amorphous'), it shall be reported.

4.2.5 Evaluation of stability

Stability of the physico-chemical properties specified in 4.2.1.2, 4.2.2 and 4.2.3 shall be evaluated according to ISO Guide 35 by several measurements in classical or isochronous stability studies.[8] In the measurements, and depending on the material, the influence of environmental temperature, humidity and/or electromagnetic radiation, and the influence of vibration, are studied and a tolerable environment for storage should be deduced. Representative test materials described in this document should be supplied in an appropriately packaged form (e.g. bottles, vials or test pieces) in order to minimize the potential for changes in environmental conditions that influence the physico-chemical properties.

4.2.6 Evaluation of homogeneity

The homogeneity of the physico-chemical properties specified in 4.2.1.2, 4.2.2 and 4.2.3 shall be evaluated between packages (e.g. bottles, vials or test pieces) as described in ISO Guide 35. Homogeneity within a single package ('Within-bottle homogeneity' in ISO Guide 35) should be evaluated if appropriate.

NOTE ISO 14488 may be consulted for guidance on sampling.[10]

5 Information related to quality management

Information on the manufacturing process of the representative test material and to the level of its quality control are required to support claims of homogeneity and representativeness of the representative test materials. The document claiming conformity to the quality control management requirement of the manufacturing process, the technical description of the management system, the description of the management of the measurement system and the record of the implementation of the management can be included as an informative reference. The quality management system shall apply ISO 9001 or its equivalent as the requirement standard.[12]