TECHNICAL SPECIFICATION

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Nanotechnologies — Nanoscale titanium dioxide in powder form — Characteristics and measurement

Nanotechnologies — Dioxyde de titane à la nano-échelle sous forme de poudre — Caractéristiques et mesurage

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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

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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order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn.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again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.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 11937 was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Introduction

Titanium dioxide, TiO_2, has been used extensively for circa 90 years as the main white pigment in paints, plastics, printing inks and many other products due to its ability to scatter visible light and provide white opacity to the products in which it is used. More recently, nanoscale titanium dioxide has been developed, here the smaller particle size does not provide pigmentary properties but gives a transparent product which can be used in different applications such as sunscreens or catalysis where the small particle size can enhance the activity. Accordingly, there is a need to better define the characteristics of the nanoscale material providing these alternative properties. This technical specification provides the methods to evaluate mass fraction of the rutile and anatase titanium dioxide as well as to measure four key parameters: crystal structure, average crystallite size, average primary particle size and specific surface area, which are commonly used to indicate characteristics of nanoscale materials.

Most of the manufactured nanoscale titanium dioxides are synthesized by the sulfate process, the chloride process or the sol-gel process, and the crystal structures of the products are almost entirely rutile and anatase. Therefore, brookite and amorphous forms are not specified in this Technical Specification. The X-ray diffraction (XRD) method is used to measure the crystal structure and the ratio of anatase to rutile.

Commonly, some of the nanoscale titanium dioxide products are coated with silica or alumina for specific applications. Alternatively, some of the nanoscale titanium dioxide products may include a dopant of another metal within their crystal lattice for other specific applications. These coatings and dopants are permanent. Buyer, seller and regulator should be aware the presence of any coatings. The XRD method and transmission electron microscopy (TEM) are used to measure crystal size and primary particle size/shape, respectively. The Brunauer, Emmet and Teller (BET) method is widely used for the evaluation of specific surface area. Theoretically, XRD just measures the core size of the coated nanoscale titanium dioxide but not the surface coating. TEM is used to measure the physical primary particle size including surface coatings.

Nanotechnology is a rapidly growing and evolving field. Users of this document should maintain an awareness of the legislative environment and latest developments in Environmental Health and Safety regarding nanotechnology. These references may be of interest[1-12]. Responsibilities of users of this document include the following: the seller is obliged to provide the buyer with such environmental health and safety information as required by law. If the seller or buyer wishes to assess the environmental, safety or health risks of the material, they may refer to ISO/TR 12885:2008[8] for further guidance.

This document may be used in conjunction with other application specific standards developed either by ISO or other standards development bodies.

Nanotechnologies — Nanoscale titanium dioxide in powder form — Characteristics and measurement

WARNING — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

1 Scope

This Technical Specification provides requirements to describe the basic characteristics of titanium dioxide in powder form relevant for applications in nanotechnology. It is intended to detail the materials specification necessary to use titanium dioxide in the applications related to nanotechonology.

It is limited to dry powders and does not include materials dispersed or suspended in water or solvents.

It does not cover characteristics for health and safety issue, and for specific application of titanium dioxide or for surface modification, if coated.

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 591-1, *Titanium dioxide pigments for paints — Part 1: Specifications and methods of test*

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

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

ISO 14887, *Sample preparation — Dispersing procedures for powders in liquids*

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*

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

3 Terms and definitions

For the purposes of this document, the terms, definitions and abbreviated terms given in ISO 14488, ISO/TS 27687 and ISO/TS 80004-1 and the following apply.

3.1
transmission electron microscope (TEM)
instrument that produces magnified images or diffraction patterns of the sample by an electron beam which passes through the sample and interacts with it

[ISO 29301:2010, definition 3.37]

3.2
X-Ray diffraction (XRD)
scattering in which the incident radiation is a beam of x-rays. The elastic scattering of the x-rays from the electron clouds of atoms in a system produces a diffraction pattern that gives information about the crystallographic structure

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

[ISO 9277:2010, definition 3.11]

3.4
crystal structure
arrangement of a regular and repeating internal unit of atoms in three dimensions in which the atoms are set in space in a fixed relation to each other

3.5
primary particle
particle not formed from a collection of smaller particles

Note 1 to entry: The term typically refers to particles formed through nucleation from the vapour phase before coagulation occurs.

[ISO/TR 27628:2007, definition 2.16]

4 Basic characteristics and measurement methods

For titanium dioxide in powder form conforming to this Technical Specification the following basic characteristics should be measured and reported. The necessary characteristics and corresponding measurement method are listed in Table 1.

The requirements for magnitudes of measured characteristics shall be agreed upon between interested parties and test results should be reported complying with the requirement of Clause 6.

Table 1 — Basic characteristics with corresponding measurement methods

Characteristics	Unit	measurement methods
Mass fraction of titanium dioxide	% (kg/kg)	Aluminium reduction method/ Chromium(II) chloride reduction method (ISO 591-1) or other chemical analysis methods upon the agreement between interested parties
Ratio of crystalline phases	%	XRD
Average crystallite size	nm	XRD (Scherrer formula)
Average primary particle size	nm	TEM
Specific surface area	m^2/g	BET method

NOTE 1 The set of basic characteristics is evaluated in order to represent the nanoscale titanium dioxide in powder form in terms of nano size-related features and its main ingredient.

NOTE 2 Additional characteristics relevant to specific applications may be specified depend on the intended application and other related international standards.

NOTE 3 The detailed procedures for these measurement methods are not provided in this Technical Specification. In order to obtain the measurement results required by the interested parties, the measurement methods should be applied and managed under a well recognized quality system.

5 Sampling

Take a representative sample of the product to be tested, as described in ISO 14488.

6 Reporting

The test report should contain at least the following information:

6.1 A reference to this Technical Specification, i.e. ISO/TS 11937.

6.2 Identification of material tested (product name, chemical name).

6.3 Samples description (manufacturer of nanoscale titanium dioxide, batch number or lot number, country of origin).

6.4 Laboratory (name of testing laboratory).

6.5 Results.

6.5.1 Measurement results of basic characteristics, and their measurement methods required in Table 1 (for TEM, also report the number of particles used in the determination of the average size, standard deviation of measurement results and details on measurement method for TEM method).

6.5.2 Measurement uncertainty (subject to the agreement between users, suppliers and regulators).

6.6 Additional information (if any).

Bibliography

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[4] EC Guidelines for the notification of Dangerous Consumer Products to the Competent Authorities of the Member States by Producers and Distributors in Accordance with Article 5(3) of Directive 2001/95/EC ec.europa.eu/consumers/cons_safe/prod_safe/guidelines_documents.pdf

[5] European Commission, *Risk Assessment Guidelines for Non-Food Consumer Products*, Draft for Consultation, August 2008

[6] http://ec.europa.eu/consumers/ipm/risk_assesment_guidelines_non_food.pdf

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[8] ISO/TR 12885:2008, *Nanotechnologies — Health and safety practices in occupational settings relevant to nanotechnologies*

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[10] ISO/IEC Guide 50 — *Safety aspects — Guidelines for child safety*

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[13] EN 13925-1:2003, *Non-destructive testing — X-ray diffraction from polycrystalline and amorphous materials — Part 1: General principles*

[14] ISO/TS 11931, *Nanotechnologies — Nanoscale calcium carbonate in powder form —Characteristics and measurement*

[15] EN 13925-2:2003, *Non-destructive testing — X-ray diffraction from polycrystalline and amorphous materials — Part 2: Procedures*

[16] ISO 29301:2010, *Microbeam analysis — Analytical transmission electron microscopy — Methods for calibrating image magnification by using reference materials having periodic structures*

[17] ISO/TR 27628:2007, *Workplace atmospheres — Ultrafine, nanoparticle and nano-structured aerosols — Inhalation exposure characterization and assessment*

[18] EN 13925-3:2005, *Non-destructive testing — X-ray diffraction from polycrystalline and amorphous materials — Part 3: Instruments*