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

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National foreword

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Foreword

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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.

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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]