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**Nanotechnologies — Nanoscale
calcium carbonate in powder form —
Characteristics and measurement**

*Nanotechnologies — Carbonate de calcium à la nano-échelle sous
forme de poudre — Caractéristiques et mesurage*



Reference number
ISO/TS 11931:2012(E)

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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order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn.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again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.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 11931 was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Introduction

Calcium carbonate, CaCO_3 , is widely used in the paint, ink, building, paper, pharmaceutical and food industries. The applications of this material are expected to increase with the development of nanoscale calcium carbonate. Accordingly, there is a need to better define the characteristics of this material contributing to its performance. This Technical Specification provides methods to determine chemical composition of nanoscale calcium carbonate and three key parameters commonly used to characterize nanoscale calcium carbonate: average crystallite size, average primary particle size and specific surface area.

Calcium carbonate has crystalline structures: calcite, aragonite and vaterite. However, only calcite is widely used in commercial applications. Thus nanoscale calcium carbonate described in this Technical Specification has the calcite crystal structure.

There are two kinds of commercial nanoscale calcium carbonate: ground calcium carbonate (GCC) and precipitated calcium carbonate (PCC). These two products have different characteristics such as particle shape and particle size distribution. The buyer and seller should be aware of the different characteristics required for different applications.

X-ray diffraction (XRD) and transmission electron microscopy (TEM) methods are used to measure crystallite size and primary particle size, respectively. The Brunauer, Emmet and Teller (BET) method is used to measure specific surface area.

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 wish to assess the environmental, safety or health risks of the material, they may refer to ISO/TR 12885:2008^[7] 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 calcium carbonate 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 nanoscale calcium carbonate in powder form relevant for applications in nanotechnology. It is intended to detail the material specification necessary to use CaCO₃ in the applications related to nanotechnology.

It does not cover characteristics specific for health and safety issues, and for specific applications of nanoscale CaCO₃.

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 3262-1, *Extenders for paints — Specifications and methods of test — Part 1: Introduction and general test methods*

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

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

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, definitions and abbreviated terms

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

precipitated calcium carbonate (PCC)

a synthetic calcium carbonate, consisting of trigonal crystals (like those of calcite) or rhombic bipyramidal crystals (like those of aragonite)

[ISO 3262-6:1998, definition 3.1]

3.5

ground calcium carbonate (GCC)

calcium carbonate produced by grinding (attrition) natural calcium carbonate

3.6

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 calcium carbonate 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 should be agreed upon between interested parties and the 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 calcium carbonate	% (kg/kg)	Titration method (ISO 3262-1) or other chemical analysis methods upon the agreement between interested parties
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 calcium carbonate 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 depending 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 shall contain at least the following information:

6.1 Reference to this Technical Specification, i.e. ISO/TS 11931.

6.2 Identification (product name, chemical name).

6.3 Samples description (source of nanoscale calcium carbonate, 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).

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