
**Nanotechnologies — Measurements of
particle size and shape distributions
by transmission electron microscopy**

*Nanotechnologies — Détermination de la distribution de taille et de
forme des particules par microscopie électronique à transmission*

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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 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

Characterization procedures for nanoparticles often include, but are not limited to, size, shape, surface structure (or texture), and surface chemistry. These measurements, combined with phase information, such as crystalline phase, constitute the morphology of the material. This document focuses on two attributes of morphology, size and shape distributions, for discrete, agglomerated and aggregated nano-objects (materials with at least one dimension in the nanoscale, $1\text{ nm} < \text{a length dimension} < 100\text{ nm}$). Transmission electron microscopy, a standard tool for measurements on the nanoscale, provides two-dimensional images of particle projections. This generic workflow for measuring and evaluating particle size and shape distributions on the nanoscale includes sample preparation, instrument factors, image capture, particle analysis, data analysis, and reporting. Seven case studies have been included to illustrate how the generic protocol can be applied to different particle morphologies and sample types. Three discrete particle test samples are reported: spheroidal (gold nanospheres), a bimodal mixture of particle sizes (colloidal silicas), and a mixture of particle shapes (gold nanorods and gold nanocubes). Two aggregate test samples are reported: amorphous aciniform aggregates (carbon black) and aggregates of primary crystallites (titania). Measurements methods are also presented for low aspect ratio samples and nanoparticles with specific crystal habits. Several of the case studies are supported by interlaboratory collaborations conducted under the guidelines of the Versailles Project on Advanced Materials and Standards (VAMAS) for interlaboratory comparisons (ILCs)^[42].

Three types of size and shape descriptors are considered. Size descriptors include those determined by linear or areal measurements. Shape descriptors include elongational descriptors, such as ratios of two length descriptors, and ruggedness descriptors, which represent surface irregularities.

The protocol emphasizes qualitative and quantitative analysis of data quality by the user. Qualitative comparisons of datasets include determining the similarity or differences between single descriptor means or multivariate means. Quantitative comparisons of datasets are based on difference or similarities between the parameters of reference models fitted to descriptor distributions. At least two parameters (mean and spread) and their uncertainties are needed to define a descriptor distribution. In some cases, these two quantitative parameters and their uncertainties may not be sufficient for characterization of particle size and shape distributions. Data visualization techniques, such as residual deviation and quantile plots, and data correlations, such as pairs of size and shape descriptors or fractal analysis, can provide additional ways to evaluate and differentiate test samples. Taken together, qualitative and quantitative quality metrics plus visualization and correlation tools permit users to tailor the protocol to their qualitative and quantitative quality targets.

Nanotechnologies — Measurements of particle size and shape distributions by transmission electron microscopy

1 Scope

This document specifies how to capture, measure and analyse transmission electron microscopy images to obtain particle size and shape distributions in the nanoscale.

This document broadly is applicable to nano-objects as well as to particles with sizes larger than 100 nm. The exact working range of the method depends on the required uncertainty and on the performance of the transmission electron microscope. These elements can be evaluated according to the requirements described in this document.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 9276-3, *Representation of results of particle size analysis — Part 3: Adjustment of an experimental curve to a reference model*

ISO 9276-6:2008, *Representation of results of particle size analysis — Part 6: Descriptive and quantitative representation of particle shape and morphology*

ISO 29301, *Microbeam analysis — Analytical electron microscopy — Methods for calibrating image magnification by using reference materials with periodic structures*

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3 Terms, definitions and symbols

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <http://www.electropedia.org/>

3.1 Core terms — Particles

3.1.1

nano-object

discrete piece of material with one, two or three external dimensions in the *nanoscale* (3.1.2)

[SOURCE: ISO/TS 80004-2:2015, 2.2]

3.1.2

nanoscale

length range approximately from 1 nm to 100 nm

[SOURCE: ISO/TS 80004-1:2015, 2.1, modified — Note 1 to entry has been deleted.]