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SLOVENSKI STANDARD
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01-marec-2022

Nanotehnologije - Meritve porazdelitve velikosti in oblike delcev s transmisijsko elektronsko mikroskopijo (ISO 21363:2020)

Nanotechnologies - Measurements of particle size and shape distributions by transmission electron microscopy (ISO 21363:2020)

Nanotechnologien - Messungen von Partikelgrößen- und Partikelformverteilungen mittels Transmissionselektronenmikroskopie (ISO 21363:2020)

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Nanotechnologies - Détermination de la distribution de taille et de forme des particules par microscopie électronique à transmission (ISO 21363:2020)

Ta slovenski standard je istoveten z: EN ISO 21363:2022

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ICS:

07.120	Nanotehnologije	Nanotechnologies

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 21363

January 2022

ICS 07.120

English Version

Nanotechnologies - Measurements of particle size and shape distributions by transmission electron microscopy (ISO 21363:2020)

Nanotechnologies - Détermination de la distribution de taille et de forme des particules par microscopie électronique à transmission (ISO 21363:2020)

Nanotechnologien - Messungen von Partikelgrößen- und Partikelformverteilungen mittels Transmissionselektronenmikroskopie (ISO 21363:2020)

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European foreword

The text of ISO 21363:2020 has been prepared by Technical Committee ISO/TC 229 "Nanotechnologies" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 21363:2022 by Technical Committee CEN/TC 352 "Nanotechnologies" the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2022, and conflicting national standards shall be withdrawn at the latest by July 2022.

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INTERNATIONAL STANDARD

ISO 21363

First edition
2020-06

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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Reference number
ISO 21363:2020(E)

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Published in Switzerland

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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).

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

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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 nm < a length dimension < 100 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.

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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.

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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.

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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.]

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3.1.3
particle
minute piece of matter with defined physical boundaries

[SOURCE: ISO 26824:2013, 1.1, modified — Notes 1, 2 and 3 to entry have been deleted.]

3.1.4
constituent particle
identifiable, integral component of a larger *particle* (3.1.3)

[SOURCE: ISO/TS 80004-2:2015, 3.3, modified — Note 1 to entry has been deleted.]

3.1.5
agglomerate
collection of weakly or medium strongly bound *particles* (3.1.3) where the resulting external surface area is similar to the sum of the surface areas of the individual components

Note 1 to entry: The forces holding an agglomerate together are weak forces, for example van der Waals forces or simple physical entanglement.

Note 2 to entry: Agglomerates are also termed secondary particles and the original source particles are termed primary particles.

[SOURCE: ISO/TS 80004-2:2015, 3.4]

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3.1.6
aggregate
particle (3.1.3) comprising strongly bonded or fused particles where the resulting external surface area may be significantly smaller than the sum of calculated surface areas of the individual components

Note 1 to entry: The forces holding an aggregate together are strong forces (for example, covalent bonds) or those resulting from sintering or complex physical entanglement.

Note 2 to entry: Aggregates are also termed secondary particles and the original source particles are termed primary particles.

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Note 3 to entry: Entries 3.1.6 to 3.1.10 define elements of agglomerates and aggregates, some of which are illustrated in Figure 1. Constituent particles in an aggregate are tightly fused into a discrete entity (the aggregate), while the constituent particles in an agglomerate are weakly bound and generally easily dispersed under shear or mechanical stress.

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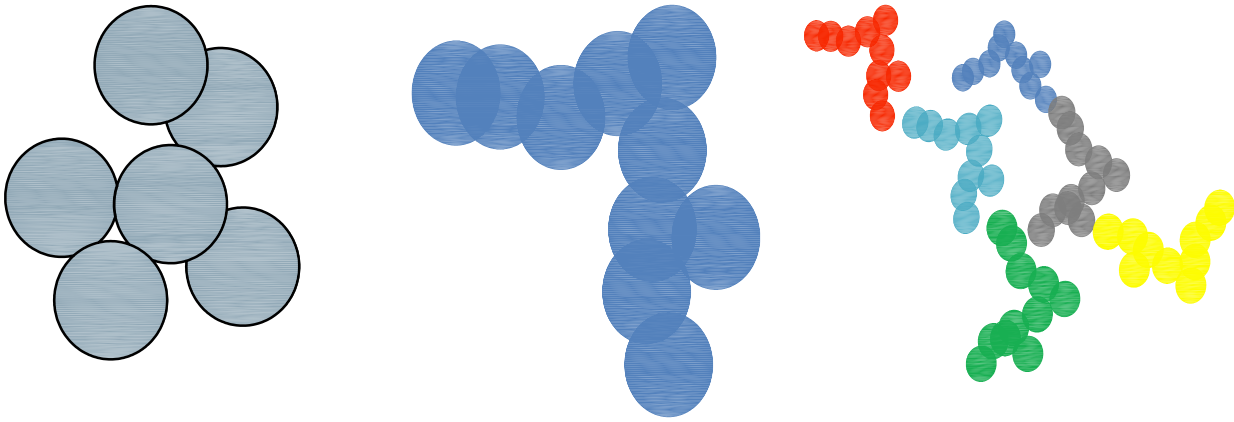
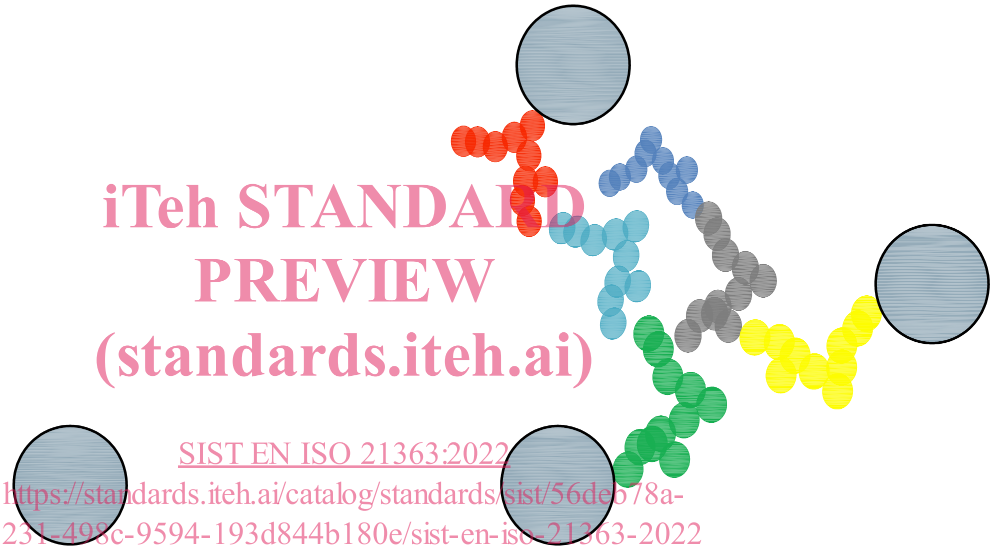


a) Primary particles in an agglomerate b) Primary particles in an aggregate c) Agglomerate of aggregates



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d) Nano-object (if less than 100 nm) or particle e) Agglomerate of both primary particles and aggregates

Figure 1 — Schematic showing elements of agglomerates and aggregates

[SOURCE: ISO/TS 80004-2:2015, 3.5, modified — In the definition, "may be significantly smaller" has replaced "is significantly smaller" and "calculated" has been added before "surface areas". In Note 1 to entry, "ionic bonds" in the example and the final phrase "or otherwise combined former primary particles" have been deleted. Note 3 to entry and Figure 1 have been added.]

3.1.7
nanoparticle
nano-object (3.1.1) with all three external dimensions in the *nanoscale* (3.1.2) where the lengths of the longest and shortest axes of the nano-object do not differ significantly

[SOURCE: ISO/TS 80004-2:2015, 4.4, modified — "three" has been added and Note 1 to entry has been deleted.]

3.1.8
nanorod
solid *nanofibre* (3.1.9)

[SOURCE: ISO/TS 80004-2:2015, 4.7]

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