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

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

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Contents

Page

Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
3.1 General terms.....	2
3.2 Core terms: image analysis.....	4
3.3 Core terms: statistical symbols and definitions.....	4
3.4 Core terms: measurands and descriptors.....	6
3.5 Core terms: metrology.....	8
3.6 Core terms: scanning electron microscopy.....	10
4 General principles	11
4.1 SEM imaging.....	11
4.2 SEM image-based particle size measurements.....	12
4.3 SEM image-based particle shape measurements.....	13
5 Sample preparation	13
5.1 Sample preparation fundamental information.....	13
5.2 General recommendations.....	14
5.3 Ensuring good sampling of powder or dispersion-in-liquid raw materials.....	14
5.3.1 Powders.....	14
5.3.2 Nanoparticle dispersions in liquids.....	15
5.4 Ensuring representative dispersion.....	15
5.5 Nanoparticle deposition on a substrate.....	15
5.5.1 General.....	15
5.5.2 Nanoparticle deposition on wafers and chips of silicon or other materials.....	16
5.5.3 Nanoparticle deposition on TEM grids.....	17
5.6 Number of samples to be prepared.....	18
5.7 Number of particles to be measured for particle size determination.....	18
5.8 Number of particles to be measured for particle shape determination.....	19
6 Qualification of the SEM for nanoparticle measurements	19
7 Image acquisition	19
7.1 General.....	19
7.2 Setting suitable image magnification and pixel resolution.....	23
8 Particle analysis	24
8.1 Particle analysis fundamental information.....	24
8.2 Individual particle analysis.....	25
8.3 Automated particle analysis.....	25
8.4 Automated particle analysis procedure example.....	26
9 Data analysis	27
9.1 General.....	27
9.2 Raw data screening: detecting touching particles, artefacts and contaminants.....	27
9.3 Fitting models to data.....	27
9.4 Assessment of measurement uncertainty.....	27
9.4.1 General.....	27
9.4.2 Example: Measurement uncertainty for particle size measurements.....	28
9.4.3 Bivariate analysis.....	29
10 Reporting the results	29
Annex A (normative) Qualification of the SEM for nanoparticle measurements	31
Annex B (informative) Cross-sectional titanium dioxide samples preparation	36

Annex C (informative) Case study on well-dispersed 60 nm size silicon dioxide nanoparticles	38
Annex D (informative) Case study on 40 nm size titanium dioxide nanoparticles	46
Annex E (informative) Example for extracting particle size results of SEM-based nanoparticle measurements using ImageJ	55
Annex F (informative) Effects of some image acquisition parameters and thresholding methods on SEM particle size measurements	57
Annex G (informative) Example for reporting results of SEM-based nanoparticle measurements	61
Bibliography	71

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Foreword

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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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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

This document provides guidance for measuring and reporting the size and shape distributions of nanometer-scale particles using images acquired by the scanning electron microscope (SEM). This document applies to the SEM-based measurement of larger particles also. Nanoparticles are three-dimensional (3D) objects, but the SEM image is only a two-dimensional (2D) representation of the 3D shape from a certain viewing angle. The SEM image carries valuable information about the size and shape of particles. While the SEM image does contain a certain amount of 3D information, for sake of simplicity, this document does not deal with reconstructing 3D information. Rigorous three-dimensional characterization of nanoparticles would include size, shape, surface structure (e.g. texture), surface and internal material composition, and their locations in the investigated 3D volume. This document deals with two attributes of morphology, size and shape, for discrete and aggregated nano-objects (materials with at least one dimension in the nanometer-scale, i.e. within 1 nm to 100 nm). Suitable sample preparation is essential to obtaining high-quality electron microscope images and preferred techniques often vary with the sample material. It is equally important to make sure that the SEM itself is suitable to carry out the measurements with the required uncertainty. Typical guidance suggests that a large number, several hundreds or thousands of particles need to be measured for statistically sound size and shape distribution results. The actual number of nano-objects needed to be measured depends on the sample, the required uncertainty and on the performance of the SEM. Statistical evaluation of the data and the evaluation of uncertainty of the measurands are included as part of the measurement and reporting procedures.

This document contains measurement procedures, particle and data analysis and reporting clauses. In the Annexes, there are specific examples for measurements and guidance for the qualification of the SEM for reliable quantitative measurements. Automation of the image acquisition and data analysis can reduce cost and improve the quality of the results. Measurements of samples of discrete nanoparticles are generally easier to carry out with automated image acquisition and particle analysis systems. Measurements of complex discrete nanoparticles, and aggregates or agglomerates of nanoparticles may require operator-assisted image acquisition and analysis. Evaluation of particle shape is facilitated by many pertinent analysis software solutions that allow for automatic selection of various shape attributes as well.

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

1 Scope

This document specifies methods of determining nanoparticle size and shape distributions by acquiring and evaluating scanning electron microscope images and by obtaining and reporting accurate results.

NOTE 1 This document applies to particles with a lower size limit that depends on the required uncertainty and on the suitable performance of the SEM, which is to be proven first - according to the requirements described in this document.

NOTE 2 This document applies also to SEM-based size and shape measurements of larger than nanoscale particles.

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/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

ISO 9276-1, *Representation of results of particle size analysis — Part 1: Graphical representation*

ISO 9276-2, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

ISO 9276-3, *Representation of results of particle size analysis — Part 3: Adjustment of an experimental curve to a reference model*

ISO 9276-5, *Representation of results of particle size analysis — Part 5: Methods of calculation relating to particle size analyses using logarithmic normal probability distribution*

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

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

ISO 16700, *Microbeam analysis — Scanning electron microscopy — Guidelines for calibrating image magnification*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO/TS 24597:2011, *Microbeam analysis — Scanning electron microscopy — Methods of evaluating image sharpness*

ISO 26824, *Particle characterization of particulate systems — Vocabulary*

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

ISO/TS 80004-2, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

ISO/TS 80004-3, *Nanotechnologies — Vocabulary — Part 3: Carbon nano-objects*

ISO/TS 80004-4, *Nanotechnologies — Vocabulary — Part 4: Nanostructured materials*