# ISO 21363:2020 (E)

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

## Contents

Foreword

#### Introduction

- Scope 1
- 2 Normative references
- 3 Terms, definitions and symbols
  - 3.1 Core terms — Particles
  - 3.2 Core terms — Image capture and analysis
  - 3.3 Core terms — Statistical symbols and definitions
  - 3.4 Core terms — Measurands
  - 3.5
  - Core terms Metrology Core terms Transmission electron microscopy 3.6
  - Statistical symbols, measurands and descriptors 3.7
  - Statistical symbols 3.7.1
  - 3.7.2 Measurands and descriptors
  - 3.7.2.1 Size descriptors and symbols — Category 1
  - Size descriptors and symbols Category 2 3.7.2.2
  - 3.7.2.3 Shape descriptor symbols — Category 1
- Stakeholder needs for TEM measurement procedures 4
- 5 Sample preparation
  - 5.1 General
  - Sample sources 5.2
  - Use a representative sample 5.3
  - 5.3.1 General
  - 5.3.2 **Powder samples**
  - 5.3.3 Nanoparticle dispersions in liquids
  - Minimize particle agglomeration in the sample dispersion 5.4
  - 5.5 Selection of the mounting support
- 6 Instrument factors
  - Instrument set-up 6.1
  - Calibration 6.2
  - 6.2.1 General
  - 6.2.2 **Calibration standards**
  - 6.2.3 General calibration procedure
  - Setting TEM operating conditions for calibration 6.3
- 7
- Image capture
- 7.1 General
- Setting a suitable operating magnification 7.2
- 7.3 Minimum particle area
- 7.4 Number of particles to count for particle size and shape distributions
- Uniform background 7.5
- 7.6 Measurement procedure
- 7.6.1 General
- 7.6.2 Developing a test sample
- Effects of magnification 7.6.3
- 7.6.4 Frames (micrographs)

7.7 Revision of image capture protocols

#### 8 Particle analysis

- 8.1 General
- 8.2 Individual particle analysis
- 8.3 Automated particle analysis
- 8.4 Example Automated particle analysis procedure
- Data analysis

9

- 9.1 General
- 9.2 Raw data triage Detecting touching particles, unselected particles, artefacts and contaminants
- 9.3 Data quality assessment Repeatability, intermediate precision and reproducibility
- 9.4 Fitting distributions to data
- 9.5 Assessing measurement uncertainty for samples under repeatability, intermediate precision or reproducibility conditions
- 9.5.1 Grand statistics for fitted parameters Three or more datasets
- 9.5.2 Measurement uncertainty of fitted parameters
- 9.5.3 Example Measurement uncertainty for a size descriptor
- 9.6 Bivariate analysis
- 10 Reporting
- Annex A (informative) Case studies overview
  - A.1 General
  - A.2 Discrete spheroidal nanoparticles (see Annex B)
  - A.3 Size mixture (see Annex C)
  - A.4 Shape mixture (see Annex D)
  - A.5 Amorphous aggregates (see Annex E)
  - A.6 Nanocrystallite aggregates (see Annex F)
  - A.7 Low aspect ratio particles (see Annex G)
  - A.8 Nanoparticles with specific crystal habits (see Annex H)
- Annex B (informative) Discrete spheroidal nanoparticles
  - B.1 Reference
  - B.2 Background and design objectives
  - B.3 Highlights
- Annex C (informative) Size mixture
  - C.1 Purpose
  - C.2 Background and design objectives
  - C.3 Highlights
  - C.3.1 General
  - C.3.2 Raw data triage
  - C.3.3 Differentiating between large and mid-sized particle clusters
  - C.3.3.1 General
  - C.3.3.2 Elongation–size correlation
  - C.3.3.3 Ruggedness-size correlation
  - C.3.4 Laboratory intermediate precision
  - C.4 Conclusions
- Annex D (informative) Shape mixture
  - D.1 Reference
  - D.2 Background and design objectives
  - D.3 Highlights
  - D.3.1 Method for identifying and separating touching particle complexes
  - D.3.2 Differentiating between nanorod samples
- Annex E (informative) Amorphous aggregates
  - E.1 Reference
  - E.2 Background and design objectives
  - E.3 Highlights
  - E.3.1 Measurement uncertainties of carbon black aggregate descriptors
  - E.3.2 Descriptor distributions re-assembled using four aggregate shape populations

#### Annex F (informative) Nanocrystalline aggregates

- F.1 Reference
- F.2 Background and design objectives
- F.3 Highlights
- F.3.1 Effects of protocol factors on crystallite data quality
- F.3.2 Primary crystallite size descriptors are best modelled using lognormal distributions
- F.3.2.1 General
- F.3.2.2 Data visualizations complement measurement uncertainty values

#### Annex G (informative) Nanofibres with irregular cross-sections

- G.1 Reference
- G.2 Background and design objectives
- G.3 Highlights
- G.3.1 General
- G.3.2 Data quality
- G.3.3 Comparisons of size descriptors for polygon tracing and cross-sectional analysis

Annex H (informative) Nanoparticles with specific crystal habits

- H.1 Purpose
- H.2 Background and design objectives
- H.3 Highlights
- H.3.1 Reference models and uncertainties for size and elongational shape descriptors
- H.3.2 Comparisons of data taken on three different instrument types (TEM, TSEM and miniTEM)
- H.3.3 Comparison of datasets taken with the same particles on the same images
- H.3.4 Reproducibility of data taken sequentially on the same grid, two laboratories

Page count: 83