ISO 21363:2020 (E)

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

Contents

Foreword

Introduction

1 Scope

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
3.6 Core terms — Transmission electron microscopy
3.7 Statistical symbols, measurands and descriptors
3.7.1 Statistical symbols
3.7.2 Measurands and descriptors
3.7.2.1 Size descriptors and symbols — Category 1
3.7.2.2 Size descriptors and symbols — Category 2
3.7.2.3 Shape descriptor symbols — Category 1

4 Stakeholder needs for TEM measurement procedures

5 Sample preparation

5.1 General
5.2 Sample sources
5.3 Use a representative sample
5.3.1 General
5.3.2 Powder samples
5.3.3 Nanoparticle dispersions in liquids
5.4 Minimize particle agglomeration in the sample dispersion
5.5 Selection of the mounting support

6 Instrument factors

6.1 Instrument set-up
6.2 Calibration
6.2.1 General
6.2.2 Calibration standards
6.2.3 General calibration procedure
6.3 Setting TEM operating conditions for calibration

7 Image capture

7.1 General
7.2 Setting a suitable operating magnification
7.3 Minimum particle area
7.4 Number of particles to count for particle size and shape distributions
7.5 Uniform background
7.6 Measurement procedure
7.6.1 General
7.6.2 Developing a test sample
7.6.3 Effects of magnification
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

9 Data analysis

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