

Project 15

Measurement of particle size and shape distribution of bipyramidal titania including deposition from liquid suspension

Objectives

- Determine and compare the particle size and shape distribution of bipyramidal TiO₂ nanoparticles by traceable measurement methods: electron microscopy (SEM, TEM, STEM-in-SEM), atomic force microscopy (AFM), and small angle X-ray scattering (SAXS);
- Determine the uncertainty induced by a deposition protocol from liquid suspension by comparison to known values from a prior Interlaboratory Comparison (ILC) in ISO 21363 with the same nanoparticles already prepared on TEM grids;
- Provide comparative validation of protocols for the techniques other than TEM.

Background

The recently published standard ISO 21363:2020 “*Nanotechnologies — Measurements of particle size and shape distributions by transmission electron microscopy*” specifies how to capture, measure and analyse TEM images to obtain particle size and shape distributions in the nanoscale. One of the seven case studies (#H *Nanoparticles with specific crystal habits*) is dedicated to the analysis of the same non-spherical nanoparticles as in this ILC as pre-deposited on carbon-coated TEM grids by BAM and distributed to all the participants.

In this project the primary objective is to quantify the additional uncertainty in particle size and shape introduced by the deposition procedure. ILC participants shall thus prepare their own samples for each measurement method according to provided preparation protocols. These protocols and the titania nanoparticles in liquid suspension will be distributed by BAM.

Standardisation needs

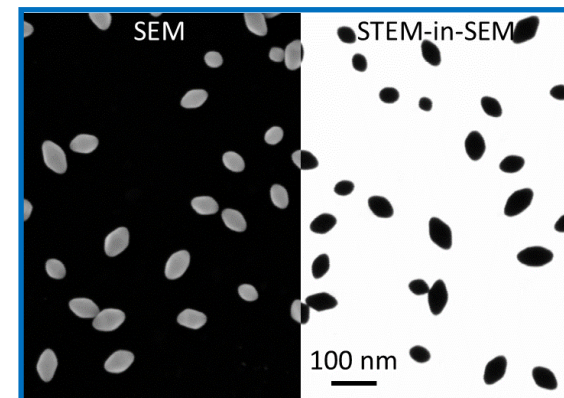
There is a need for standardized measurements of particle size and shape distribution for nanoparticles which are non-spherical. However, there are no standardized procedures for the preparation from liquid suspension of non-overlapped particles on a substrate suitable for accurate image analysis. Demonstrated reproducibility of methods for such deposition preparations will increase confidence and provide an estimate for the uncertainty in measured values attributable to sample preparation.

Work Programme

Ampoules with 2 mL liquid suspension are prepared within the EMPIR project 17NRM04 [nPSize](#) and will be provided to participants together with protocols for:

- sample preparation on a substrate,
- measurement and
- image analysis.

CALL FOR PARTICIPATION



Ampoules with the 2 mL liquid suspension of bipyramidal TiO₂ nanoparticles and electron micrographs (SEM and STEM-in-SEM) with the particles deposited on a carbon TEM grid according to the protocol.

Final data compilation and analysis will be carried out by BAM.

Deliverables and Dissemination

This interlaboratory study will be disseminated at scientific conferences and in a peer-reviewed scientific journal. Further, the [ISO 21363](#) (*Nanotechnologies — Measurements of particle size and shape distributions by transmission electron microscopy*) will be completed with measurement of nanoparticles from liquid suspension, including deposition protocols for imaging methods.

International Participation

Current participation includes volunteers from countries from all continents. More volunteers with methods other than electron microscopy, AFM and SAXS are also welcome.

Funding

Participants fund their own involvement in the project.

Project Status

The project is due to start in January 2022 for a duration of 12 months.

For more information:

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