



VAMAS

Nanoparticle Populations

Technical Work Area 34

Project 16

Measurement of (relative) number concentration of bimodal silica nanoparticles including deposition from liquid suspension

Objectives

- Validate the performance of imaging methods electron microscopy (SEM, TEM) and atomic force microscopy (AFM) to measure the relative number concentration of two modes of bimodal (30 and 60 nm) silica nanoparticles (NP)
- Validate the performance of small angle X-ray scattering (SAXS) for the traceable measurement of the number concentration of the two modes.

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 NP size and shape distributions. Case study C refers to the analysis of bimodal silica NPs, but the number concentration of the two modes (even if relative) is not considered. In this Interlaboratory Comparison (ILC) the relative number concentration of the bimodal silica NPs shall be measured. Two different relative number concentrations of the two modes were prepared. Various standardisation activities on NP size and shape distribution and number concentration are currently in progress, e.g. [ISO 19749 “Nanotechnologies — Measurements of particle size and shape distributions by scanning electron](#)

microscopy”, the ILC “*Guidelines for Shape and Size Analysis of Nanoparticles by AFM*” on spherical silica NPs in TWA 2, or [ISO 17867 “Particle size analysis — Small angle X-ray scattering \(SAXS\)”](#) in ISO/TC 24. In the frame of CCQM/IAWG, a pilot study and a key comparison are in progress, both on measurement of the NP concentration of monomodal gold.

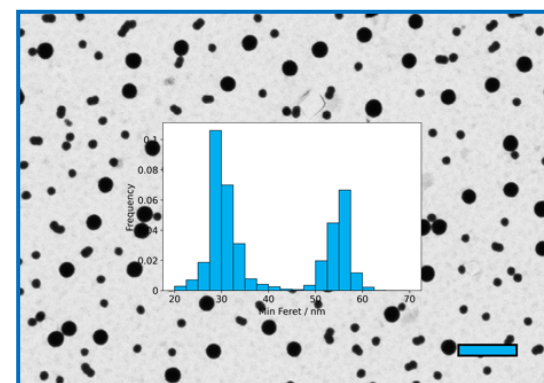
Standardisation needs

There is a need for standardized measurements of particle size and shape distribution for NPs which are non-monodisperse. There are no standardized procedures for the preparation from liquid suspension of non-overlapped particles on a substrate suitable for accurate image analysis. Only the TEM standard ISO 21363 treats the analysis of bimodal silica NPs with its challenges in detail, however, without touching the number concentration. The present ILC is intended to complete the entire sequence of NP analysis also with the evaluation of the (relative) NP number concentration step.

Work Programme

Ampoules with 1 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, and analysis.

CALL FOR PARTICIPATION



Ampoules with the 1 mL liquid suspension of bi-modal SiO₂ nanoparticles and an electron micrograph (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 TEM*) will be completed with measurement of concentration of bimodal NPs for bimodal from liquid suspension, including deposition protocols for imaging methods.

International Participation

Current participation includes volunteers from countries from all continents. Depending on the number of interested participants, more volunteers with methods other than electron microscopy, AFM and SAXS will be also considered.

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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