VAMAS

Nanoparticle Populations

Technical Work Area 34

Project 19

Analysis of total functional group content on aminated silica nanoparticles using quantitative NMR (qNMR)

Objectives

This ILC will provide data to assess a protocol for quantitative NMR (qNMR) data acquisition and quantification analysis of amine functional groups on silica nanoparticles (NPs).

ILC results will inform international standardisation efforts aimed at methods for quantifying surface chemistry under ISO/TC 229, ISO/TC201 and BIPM CCQM OAWG/SAWG, specifically validation of a qNMR protocol for surface chemical analysis under ISO TC229 PWI 19257.

Background

Functional groups on NP surfaces govern interactions with biological systems, catalytic activity, and colloidal stability. While ISO standards exist for specification of particle size and morphology, there is currently no normative international standard for assessing the surface chemistry of such NPs, particularly for quantifying the amount of either coating ligands or functional groups.

This project will test a protocol for quantification of total and surface localized and accessible surface functional groups on NPs as informed by previous bilateral comparisons of qNMR and XPS methods and the results of a European metrology SMURFnano project.

The proposed protocol involves hydrolysis followed by quantitative solution NMR assessment of functional group concentration(s). This method is anticipated to provide a traceable measurement of total functional group content in a sample.

Data collected by participants will be used to determine reproducibility and uncertainty statistics, as well as information on the clarity of the protocol document and ease of implementation. These goals align with emerging needs in regulation, nanomedicine, and materials characterisation. A set of silica NP test materials were selected for this study based on their industrial importance.

Standardization Needs

This ILC will support ISO TC/229 PWI 19257 (Characterization and quantification of functional groups and coatings on nano-objects) in developing and validating protocols to assess both total and surface/near surface functional group content on NPs. A sufficiently successful protocol will be contributed to a Technical Specification anticipated to include multiple methods to quantify total and surface functional groups. The ILC will run in parallel with a BIPM CCQM OAWG pilot study on nanoparticle surface analysis.

Call for Participation

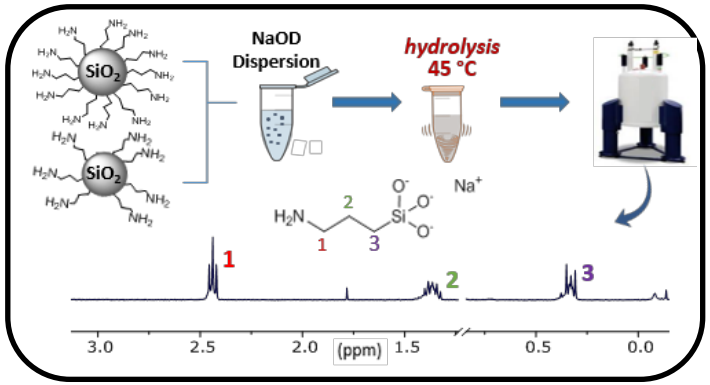

Figure.
Hydrolysis procedure and qNMR spectrum of the three aminopropyl methylene residues of aminosilane released from silica NPs

Work Programme

Organizers will provide:

- pre-bottled aminated silica NPs (nominal sizes of 50 nm, 80 nm, 100 nm) characterized for stability
- qNMR experimental method and analysis protocol
- data sheet for recording results

Participants will:

- Provide input on the method and text of the qNMR protocol
- Conduct measurements and report results, including statistical analysis of local reproducibility and uncertainty

Global analysis of all results will be conducted by the organizers.

Deliverables and Dissemination

The interlaboratory study will be completed within one year and is expected to lead to a publication. The results will provide pre-normative data for an ISO technical specification.

The ILC results will be presented at conferences and SMURFnano meetings.

Funding

Participants fund their own involvement in the project. The anticipated time commitment of participation is ≈ 5 days.

Status

Participants are being recruited. Estimated ILC start date is January 2026

For more information:

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