EMPIR Call 2016 – Energy, Environment, Normative and Research Potential

Selected Research Topic number: **SRT-n06**
Version: 1.0

Title: An improved traceability chain for nanoparticle size measurements

Abstract

Nanoparticle size and size distribution play an important role in nanotechnology because both intended and unwanted effects are often dependent on the size of particles in the nanomaterial. In the regulatory context, the definition of a nanomaterial - as e.g. the recommended definition of a nanomaterial by the European Commission (EC) - is based on the number size distribution of the nanomaterial. However, metrology to reliably size nanoparticles and nanomaterials is still challenging, taking into account the variety of measurement methods used in industry and the broad range of nanomaterials in use. Proposers addressing this SRT should analyse existing measurement and characterisation methods for size and size distribution measurement of nanoparticles, improve the traceability and agreement of results from different measurement systems and address the research needs specified by CEN TC 352 “Nanotechnologies” via the CEN-EURAMET STAIR process.

Keywords

Traceability, EC nanomaterial definition, nanoparticle, signal modelling, particle size metrology, nanoparticle shape, morphology, particle size distribution (PSD)

Background to the Metrological Challenges

The EC recommendation for the definition of nanomaterial depends on reliable nanoparticle size metrology, which is expected to be traceable to SI units. In addition, a number of European regulatory documents refer to the particle size-based definition of nanomaterials. However, the agreement between the results obtained using different measurement methods for nanoparticle size and PSD is limited and differences of nanoparticle size measurements of up to 100 % can be observed on the same material using different methods in different laboratories. To a large extent this is due to fundamental differences between the available measurement methods, which actually target different aspects of the 'size' of nanoparticle populations. This means that currently the decision whether a particular material is a nanomaterial or not according to the recommended EC definition is largely dependent on the method used and on protocols followed by practitioners. However, the large spread of nanoparticle size measurement results is also due to i) insufficient knowledge of the dependency of measurement signals of specific measurement methods on the material and shape properties of nanoparticles, and to ii) how different instruments or laboratories, using the same method, treat the raw, measured signals.

Most often, several instrument or test parameters need to be calibrated or measured in a traceable way. This can be done at the level of the individual parameters, or at the level of the overall instrument response. In the latter case, the instruments for nanoparticle size analysis are calibrated with (certified) reference materials. Sometimes manufacturers provide their own size reference materials together with the size analysis instrument sold. Certified reference materials are also used to provide a verification of the overall accuracy of the method results, rather than a direct calibration of the instrument response.

Some instruments allow the user to provide relevant material input data when different nanomaterials are to be measured, but this is not the case for all instruments. For most equipment, suitable physical models for simulation of the measurement signal have already been developed that can be made used, even if further improvement of these models is needed.

Existing International, European or national standards for nanoparticle size metrology do not always explicitly address the issue of the material dependence of the threshold or fit signals used to determine the absolute size of nano-objects. CEN TC 352 “Nanotechnologies” has formulated a list of research needs, which has

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The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

been forwarded to EURAMET within the CEN-EURAMET STAIR process [1]. The research need no.4 is specified as 'Measurement, simulation and visualization at the nanoscale' as required by a specific EC Mandate M/461 (Mandate addressed to CEN, CENELEC and ETSI for standardization activities regarding nanotechnologies and nanomaterials) [2], while research need no.11 of CEN TC 352 focuses on 'Methods to characterize basic morphology and purity of manufactured nanoparticles and other nanoscale entities'.

Objectives

Proposers should address the objectives stated below, which are based on the PRT submissions. Proposers may identify amendments to the objectives or choose to address a subset of them in order to maximise the overall impact, or address budgetary or scientific / technical constraints, but the reasons for this should be clearly stated in the protocol.

The JRP shall focus on metrology research necessary to support standardisation in nanoparticle size measurements.

The specific objectives are

1. To analyse established methods for nanoparticle characterisation e.g. SEM, TEM, AFM, DLS, CLS, SP-ICPMS and SAXS regarding their respective sensitivity to the material, shape and quantity properties for nanoparticle size metrology. The material properties that are important should be determined, together with the degree to which the material and shape influence the size and size distribution measurement results.
2. To identify, investigate and, if necessary, further refine suitable physical models to describe the output signal of a meaningful subset of nanoparticle measurement systems and which adequately take into account the material, shape and quantity properties. This should enable the model-based determination of one or more size parameters of nanoparticles from the output signals of the measurement systems.
3. To develop procedures to transfer measurement results for nanoparticle size obtained on (certified) reference materials to other types of nanoparticle materials based on the model-based approach developed in objective 2 for different methods of nanoparticle characterisation, investigated under 2.
4. To collaborate with CEN TC 352 "Nanotechnologies" and to develop a Technical Specification (TS) or Technical Report (TR) based on the scientific results, ensuring that the outputs of the project are aligned with CEN TC 352's needs. In addition, to facilitate the take up of the procedures and models developed in the project by accredited laboratories, manufacturers and end users in the nanotechnology sector.

The proposed research shall be justified by clear reference to the measurement needs within strategic documents published by the relevant Standards Developing Organisation or by a letter signed by the convenor of the respective TC/WG. EURAMET encourages proposals that include representatives from industry, regulators and standardisation bodies actively participating in the projects.

Proposers should establish the current state of the art, and explain how their proposed research goes beyond this. In particular, proposers should outline the achievements of the nanoSTAIR and iMERA-Plus T3.J1.1 Nanoparticles projects and how their proposal will build on those.

EURAMET has defined an upper limit of 1.2 M€ for the EU Contribution to this project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 30 % of the total EU Contribution to the project.

Any industrial partners that will receive significant benefit from the results of the proposed project are expected to be unfunded partners.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the "end user" community, describing how the project partners will engage with relevant communities during the project to facilitate knowledge transfer and accelerate the uptake of project outputs. Evidence of support from the "end user" community (e.g. letters of support) is also encouraged.

You should detail how your JRP results are going to:

- Address the SRT objectives and deliver solutions to the documented needs, in particular CEN TC 352,
- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the industry and nanotechnology sectors.

You should detail other impacts of your proposed JRP as specified in the document “Guide 4: Writing Joint Research Projects (JRPs)”.

You should also detail how your approach to realising the objectives will further the aim of EMPIR to develop a coherent approach at the European level in the field of metrology and include the best available contributions from across the metrology community. Specifically the opportunities for:

- improvement of the efficiency of use of available resources to better meet metrological needs and to assure the traceability of national standards
- the metrology capacity of EURAMET Member States whose metrology programmes are at an early stage of development to be increased
- organisations other than NMIs and DIs to be involved in the work

Time-scale

The project should be of up to 3 years duration.

Additional information

[1] The documents from CEN outlining their priorities can be found at http://msu.euramet.org/current_calls/pre_norm_2016/documents/SRT_related_CEN_priorities/cen_priority_15_2016.zip

[2] http://ec.europa.eu/growth/tools-databases/mandates/index.cfm?fuseaction=search.detail&id=443