Title: Metrology for manufactured nanomaterials in environmentally relevant matrices

Abstract

The impact of nanomaterials on environmental safety and human health is an increasing concern. In order to address this concern, there is a need for better measurement methods to support and improve the accurate risk assessment of nanomaterials, their use in product innovation and uptake, and their future regulation. Although the current EU recommendations and regulation for nanomaterials does not impose the measurement of the amount of nanomaterials in products or environmental compartments, it is expected that this will become a regulatory requirement, particularly for certain classes of nanomaterials, and those in environmentally relevant matrices, e.g. water and soil. Indeed, the first regulatory decisions have already been made in some EU countries, whilst in others this is still under development. Therefore, work needs to be done now to support such regulation, by developing traceable and improved measurement methods that can determine the properties of manufactured nanomaterials in environmentally relevant matrices.

Keywords

Manufactured nanomaterials, environmentally relevant matrices, sample preparation, uncertainty evaluation

Background to the Metrological Challenges

Most manufactured nanomaterials are engineered to have specific beneficial properties that cannot be obtained in bulk materials. Their production and application has only been relatively recent, due to technological advances, therefore the long term potential impact of manufactured nanomaterials on environment and human health is largely unknown.

Currently, EU regulation for some nanomaterials products requires environmental and health based risk assessments, in order to become eligible for authorisation. However, accurate risk assessment can only be based on reliable measurement data, and although the measurement of nanoparticle morphological and physical properties has largely been established in well prepared conditions without a surrounding matrix, reliable measurements of nanomaterial concentration and other properties in environmentally relevant matrices are still extremely challenging.

In 2012, ISO TC 229 'Nanotechnologies' defined a list of parameters and descriptors to characterise and identify a nanoparticle. These parameters are: size, size distribution, shape, chemical composition, crystal structure, agglomeration/aggregation state, surface charge (density), surface chemistry, surface area, solubility and dispersibility. However, the metrology related to most of these parameters is still in its early stages and certified reference materials and standard methods do not exist. For example, the dimensional metrology (size, size distribution, shape, agglomeration state) of manufactured nanomaterials can be measured using several techniques. Each measurement technique has its own advantages and drawbacks and hence so far traceable results have only been obtained for 'ideal' nanomaterials samples that preserve the state of dispersion, with low polydispersity (<= 20 %) and mono component systems. The more regulatory relevant challenge though, is to produce reliable measurements of the size, shape and concentration of nanoparticles in less ideal conditions, i.e. in matrices. To do this, requires the preservation of the nanoparticle's characteristics within its environmental during sample preparation, which is currently a major source of measurement uncertainty.

Establishing the traceability of measurement techniques for nanoparticles is vital for ensuring the comparability and usefulness of measurement results. Currently, measurement instrumentation only provides the measured values without an indication of a confidence level associated with these values and more often than not, end users do not have the capability to establish the uncertainty of the entire measurement process. Therefore algorithms to estimate the uncertainty of nanoparticle measurements are needed to provide end-users with a better understanding of their measurement results to provide confidence in them.

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 the traceable measurement and characterisation of manufactured nanomaterials in environmentally relevant matrices.

The specific objectives are

1. To develop traceable methods for the measurement of the geometrical properties (e.g. size, size distribution, shape), number concentration, and optical properties (e.g. refractive index and angular light intensity distribution) of representative manufactured nanomaterials (such as carbon (-nanotubes), silica, TiO2 and CeO) in environmentally relevant matrices.
2. To establish the traceability of existing methods used for sizing nanomaterials in complex environmentally relevant matrices. This should include the measurement of representative manufactured nanomaterials in suspension by e.g. dynamic light scattering (DLS), centrifugal liquid sedimentation (CLS), field flow fractionation inductively coupled plasma–mass spectrometry (FFF-ICP-MS/MALS), in aerosols by differential electrical mobility analysis (DEMA) and on substrates by e.g. atomic force microsopy (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM).
3. To develop validated sample preparation protocols for the measurement of nanomaterials in complex environmentally relevant matrices.
4. To develop and validate new algorithms for estimating uncertainties for nanomaterial detection and characterisation in environmentally relevant matrices. The algorithms and uncertainties should be used to support end users in the interpretation of measurement results, and to improve comparability between end-users and with industrial or regulatory requirements.
5. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain, by standards developing organisations (such as CEN/TC 352, ISO/TC 229 and ISO/DTS 19590) and end users.

These objectives will require large-scale approaches that are beyond the capabilities of single National Metrology Institutes and Designated Institutes. To enhance the impact of the research, the involvement of the appropriate user community such as industry, standardisation and regulatory bodies is strongly recommended, both prior to and during methodology development.

Proposers should establish the current state of the art, and explain how their proposed research goes beyond this and EMRP JRP NEW03 Nano ChOp.

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 2.0 M€, and has defined an upper limit of 2.3 M€ for this project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 35 % 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,
- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the nanotechnology and environmental 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

The following Standardisation request from the EC may be relevant:

M/461 Mandate Addressed to CEN, CENELEC and ETSI for Standardization Activities Regarding Nanotechnologies and Nanomaterials