A common European approach to the regulatory testing of nanomaterials

Improved measurement instruments, tools and methods Deliverable 3.06

Introduction

To assess and manage the risks related to the occupational-, consumer- and environmental exposure to nanomaterials (NMs), information is needed respectively on the actual exposure of workers, consumers and the environment to such materials. Work package 3 of the NANoREG project aims at (among others) providing such information by evaluating methods to determine the release of nano materials from products containing such materials (Deliverable 3.03), by evaluating methods to measure actual exposure to nanomaterials (Deliverable 3.06) and by applying such methods in real life situations (Deliverable 3.07).

Description of Work

The evaluation of methods to measure and monitor the exposure to NMs followed up on (and is complementary to) previous projects like NanoGEM and nanoIndEx. Both projects investigated the accuracy, comparability and field applicability of granulometers, counters and personal devices to characterize airborne NMs and produced several Standard Operation Procedures for commercial instruments.

The (additional) tools selected by task 3.3. of the NANoREG project were evaluated and compared to reference instruments. Improved sampling strategies were developed in cooperation with Task 3.2 (D3.03 and D3.07) to make the link between release and exposure. Some approaches towards conversion between metrics were evaluated. Attempts to discriminate background from actual (environmental) exposure were evaluated based on chemical composition (XRF, LIBS) and on isotope analysis.

Main results and evaluation

Fourteen instruments, tools and methods to measure exposure have been selected, evaluated and tested. They covered various route of exposure: inhalation, dermal contact and ingestion. In addition to atmospheric measurements for aerosol exposure assessment, biomonitoring tools were selected since they are essential to provide information to determine whether there is a real individual exposure situation.

Personal devices for exposure measurement	
Mini Particle Sampler	Samples the aerosol particles onto a TEM grid to allow further characterization of the NMs.
NANOBADGE	Personal sampler; provides information on the elemental composition, size distribution and morphology of material collected in the personal breathing zone of workers thanks to physical-chemical characterization by XRF and SEM-EDS.
Surface swab method; tape stripping technique	Complementary to air sampling; determines surface contamination by NMs at workplaces that may be released by cleaning activities, movement of the workers etc. Together, these methods (air and surface sampling) provide an overview of the hygienic situation in workplaces where nanomaterials are handled.

Figure: The NANOBADGE worn as personal sampler during field measurements

Used in a coherent strategy, those easy-to-use personal devices are able to provide a good picture of the potential release sources and emissions of NMs in occupational settings. Those devices could deliver mass concentration shift average with the advantage to enable the identification of specific morphological and chemical features. A limitation of these methods is the limit of detection that could require a longer sampling time than actual exposure duration. In such situations one might prefer personal monitoring using for instance commercial devices such as DiSCmini, NanoTracer or Partector. Moreover, depending on the analytical technique associated to the sampling method, information on the particle identity might be partial (shape, morphology, chemical composition …).

Biomonitoring tools	
Nasal paper flag and the Exhaled Breath Condensate method	Methods to measure biomarkers of exposure and biomarkers of effects in order to assess the actual personal exposure to occupational and environmental toxicants. They can be used for occupational health surveillance and human biomonitoring.

More sensitive, reliable and versatile instruments that requires highly qualified personnel were also investigated since they could provide valuable information on potential exposure.

More sensitive, reliable and versatile instruments	
Small Angle X-rays Scattering	Provides information on size, shape, aggregation state and structure of aggregates. This technique could be applied to samples with various physical states (i.e. solids, liquid suspensions, aerosols…). It has however a limited applicability for very heterogeneous samples in size and chemical composition.
X-ray computed tomography	Technique that provides semi-quantitative 3D chemical mapping for solids or frozen liquids (100 ppm concentration). Its main limitation is due to the long data acquisition period during which the sample has to remain stable (e.g. drying, deforming that can cause modifications).
Cryogenic – Transmission Electron Microscopy	An *in situ* analytical technical that allows imaging materials and biological samples in a frozen state, directly within the TEM. Samples and processes that previously required deposition on substrates (and presented aggregation), or could not be imaged in their native environment, could now be studied and observed in an amorphous frozen liquid and with high resolution.
Laser Induced Breakdown Spectroscopy	Another candidate for nanoparticle *detection in situ* in real time or semi real time. Applied in air or on substrate, this technique does not require sampling preparation and allows measuring to the entire list of atomic species of the periodic table.
Asymmetric-Flow Field Flow Fractionation	Separation technique that allow physical-chemical characterization of nanoparticles in suspension in complex matrices. An example is given on the detection, the characterization and the quantification of silver nanoparticles in an aqueous matrix.
Isotopic labelling approaches	Radioisotope labelling can be used as an aid to the detection and localisation of nanoparticle in environmental media, as well as kinetic studies of stability and bioavailability. It allows the detection of nanoparticle selectively from natural nanoparticles and the large variety of amorphous materials present in environmental media.

For all tools and methods mentioned, the developed SOPs will be available on the NANoREG web-site as of 1st March 2017.

For more details about NANoREG please visit the official website **www.nanoreg.eu**.

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