Objectives

The proposed activity is comprised of a series of independent interlaboratory comparisons (ILCs) of methods that will assess whether more or less than 50% of the particles in a particulate material are smaller than 100 nm. Number-based and mass-based size distributions will be monitored depending on the measurement method. The objective will be to assess the between-laboratory reproducibility of the methods.

Background

Methods have been in-house validated as part of the NanoDefine framework, (http://nanodefine.eu/), a collaborative project to investigate methods for the implementation of an EU definition of a nanomaterial relevant to EU and non-EU countries. Validation of these methods through ILCs will support the determination of reproducibility necessary for international standardisation.

Standardization Needs

If the ILCs indicate that the results of the methods are reproducible, then the methods could be offered to ISO (TC 229 or TC 24/SC4) and/or CEN (TC 352) for standardisation.

Work Programme

ILCs will be implemented to assess multiple methodologies of particle size distribution determination. Test materials and detailed analytical protocols will be provided to each ILC participant, and must be strictly followed. Opportunity for commentary on the protocols will also be provided. Participants are invited to join one, or multiple, of the following interlaboratory comparisons:

1) Electron microscopy

Participants will be requested to use an ImageJ script to derive number based size distributions based on pre-recorded TEM images. The ImageJ plugin provides different splitting methods to handle agglomerates and aggregates, robust handling of different noise levels and adaptability to non-standard images.

2) Centrifugal liquid sedimentation (CLS)

Participants will be allowed to use one of three types of CLS instruments (cuvette-analytical centrifuge with optical detection, line-start disc centrifuge with optical detection, or analytical centrifuge with refractive index detection). For each technique, a specific protocol for the sample preparation and measurement of two grades of BaSO₄ samples will be provided.

3) Single particle ICP-MS:

Three specific protocols for the sample preparation and measurement of three samples (TiO₂ particles in suspension, TiO₂ particles in sunscreen, and Al₂O₃ particles in toothpaste) will be provided.

4) Field Flow Fractionation coupled to ICP-MS:

A specific protocol for the sample preparation and measurement of TiO₂ particles in sunscreen by AF4-MALS-ICP-MS will be provided.

Deliverables and Dissemination

The expected output is an assessment of the interlaboratory reproducibility of methods useful for the characterization of particle size distributions in the nanometer size range. Outcomes of the ILCs will be shared and discussed initially within the participants, and then published in open literature. If sufficient reproducibility is demonstrated, new work item proposals will be prepared for submission to relevant standards development technical committees in ISO and CEN.

Funding

Participants fund their own involvement in the project.

Status

The project is due to start in May 2017. Samples will be provided in June 2017. Results should be reported in August 2017.

For more information:
Dr. Ana BOIX
Joint Research Centre, EC, Belgium
ana.boix-sanfelix@ec.europa.eu
Dr. Robert KOEBER
Joint Research Centre, EC, Belgium
robert.koeber@ec.europa.eu
Dr. Jeffrey FAGAN
VAMS TWA 34, Chair
National Institute of Standards and Technology, NIST, USA
jeffrey.fagan@nist.gov

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