Fraunhofer Institute for Microengineering and Microsystems IMM

Nanoanalytics

Determination of nanoparticle contents with high sensitivity

Quick Facts

- determination of nanoparticle content in colloidal suspensions
- quantitative and reproducible assessment of "Tyndall effect", as required by OECD test guideline # 105
- information about particle size and concentration
- highly sensitive device, easy to use
- full documentation for use in GLP laboratories
- adaptable to a wide range of new materials and application settings

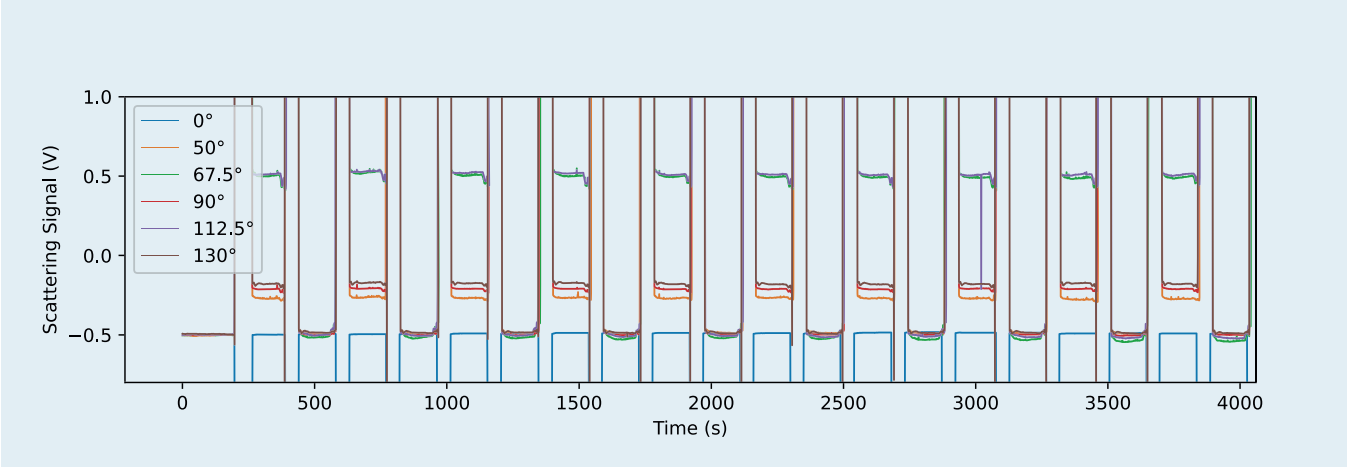
Nanomaterials have shown their great potential for improving the performance of industrial and consumer products in a multitude of cases. However, for most nanomaterials, the long-term effects to environment and to human health caused by an exposure to these materials are raising concerns. One important quality attribute of nano-enhanced materials is that individual particles are bound to the matrix material and therefore are not released into the environment during the product life cycle. A responsible risk assessment before market launch and a reliable, continuous quality control of such products during their manufacturing helps to avoid any risk for the environment or for individuals by the use of nano-enhanced products.

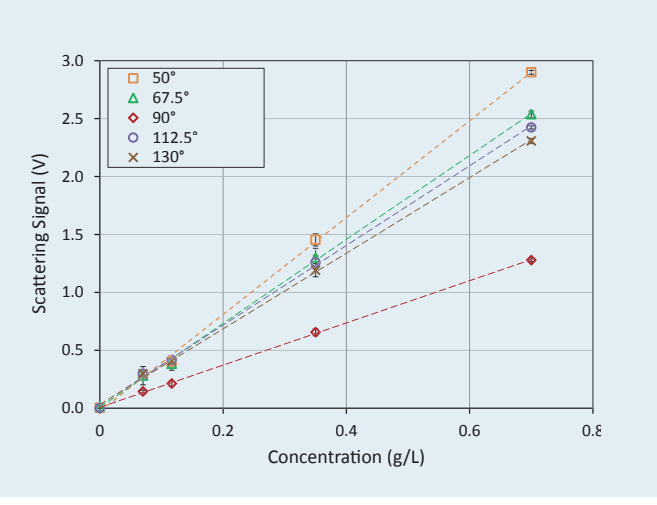
One aspect here is the check of water solubility corresponding to OECD test guideline # 105 to investigate the material quality regarding the tendency of the materials to disintegrate, possibly into nano-scaled fragments. The guideline describes sample preparation and testing for the determination of the amount of material dissolved or present as nanoparticles, small aggregates or agglomerates (< 100 nm) in water or other relevant fluids. The test requires, amongst others, "the check of the presence of colloidal matter by examination of the Tyndall effect", i.e. based on light scattering. This defines several challenges: Light scattering instruments that are currently available on the market either lack sensitivity for ultra-low concentrations of small particles or they are expensive and need a highly qualified operator to interpret the results.

For the proof-of-concept, a demonstrator instrument addressing the requirements of OECD TG # 105 has been developed. It is suitable for product monitoring and for the cost-efficient, easy to use and highly sensitive determination of nanoparticle contents of solutions, as obtained from standardized extraction tests.

Synthetic amorphous silica in commercial products

One relevant example of nanomaterials is pyrogenic (fumed) silica. This material is available as fine powder and can be found e.g. in paints and coatings, in cosmetics and in toothpastes, in the automotive sector it is used in tires and batteries, and silica is even approved for the application as food additive (E551). In a first step of the manufacturing process, the so-called primary





particles are formed which afterwards covalently bind to each other during aggregation. The resulting micrometer-sized aggregates are the product of interest for the applications, whereas a fraction of small, nanoscale primary particles may still be present. This potential fraction of non-aggregated primary particles is of concern in regard to consumer safety and environmental impact, and therefore generally undesired. As long as there is no specific OECD test guideline available that explicitly deals with nanomaterials, OECD TG # 105 is used to perform standardized extraction tests and check for the presence of colloidal matter, i.e. a potential nanoscale fraction.

Demonstrator instrument for proof-of-concept

In cooperation with an industry consortium, a demonstrator instrument has been developed by Fraunhofer IMM which is sensitive enough for the detection of small primary particles from pyrogenic silica products at concentrations near the solubility limit of synthetic amorphous silica (SAS). The demonstrator allows for the determination of the average particle size and the corresponding concentration of SAS nanoparticles with a diameter down to 5 nm which is a typical use-case for the testing of eluate fractions as obtained by OECD TG # 105. The instrument is suitable for both on-site material characterization as well as for continuous product monitoring; it can be operated stationary as well as in a mobile test laboratory setting. The demonstrator includes a violet laser (λ = 405 nm) and electronically amplified silicon photodiodes for signal detection. It allows a fully automated, time resolved data acquisition using six detectors at different angles. In-flow measurements are performed on alternating plugs of sample and reference (see graph above).

Validation of the demonstrator instrument

For validation and calibration, a commercial colloidal silica material consisting of individual particles (nominal diameter: 26 nm) was used, after detailed characterization with complementary methods. For this colloid, the limit of detection (LOD) was quantified to be 2 mg/l under GLP conditions. Correspondingly, for polystyrene latex with nominal dieameter of 50 nm, the LOD was 200 µg/l and for a suspension of liposomes with a nominal diameter of 85 nm, the LOD was 26 µg/l.

Currently, the instrument is in regular operation by a commercial analytical laboratory while at Fraunhofer IMM, the range of use is being extended to other materials and applications fields such as environmental analysis and testing of filter materials.

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