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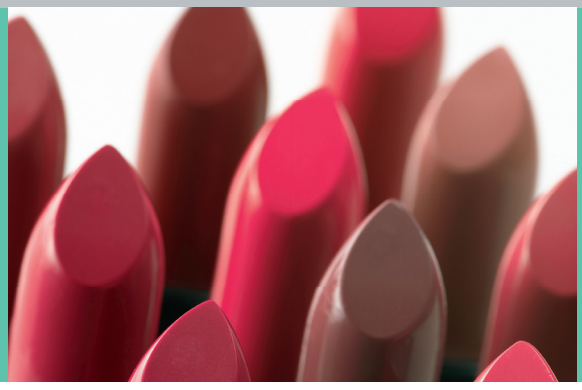

DETERMINATION OF NANOPARTICLE CONTENTS IN AQUEOUS SYSTEMS

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Nanomaterials (NM; see ISO/TS 27687:2008 and 2010) have a great potential for improving the performance of present industrial and consumable products. For certain nanoparticles (NPs), the long-term effect to environment and to human health caused by an exposure to these materials are recently under investigation.

One of the quality attributes of Nanostructured materials (NSM) is that individual NPs are not segregated and subsequently relieved into the environment. A responsible risk assessment before market launch and a reliable, continuous quality examination of such products during their production allows avoiding any risk for the environment by the use of NSMs. One aspect here is the check of water solubility corresponding to OECD Test Guideline No. 105 to control the material quality regarding their tendency to segregate nano-scaled fragments. The guideline describes sample preparation and testing for the determination of the amount of material dissolved or present as NPs, small aggregates or agglomerates (< 100 nm) in water. The test requires, amongst others, "the check of the presence of colloidal matter by examination of the Tyndall effect", meaning based on light scattering techniques. This defines several challenges: The light scattering devices that are actually available on the market either lack in sensitivity for low concentrations of small particles or they are very expensive and need a highly qualified operator to interpret the results. Therefore, a proof-of-principle demonstrator based on OECD No. 105 has been developed which is suitable for product monitoring and for the cost-efficient, easy-to-use and highly sensitive determination of nanoparticle contents in aquatic colloids.

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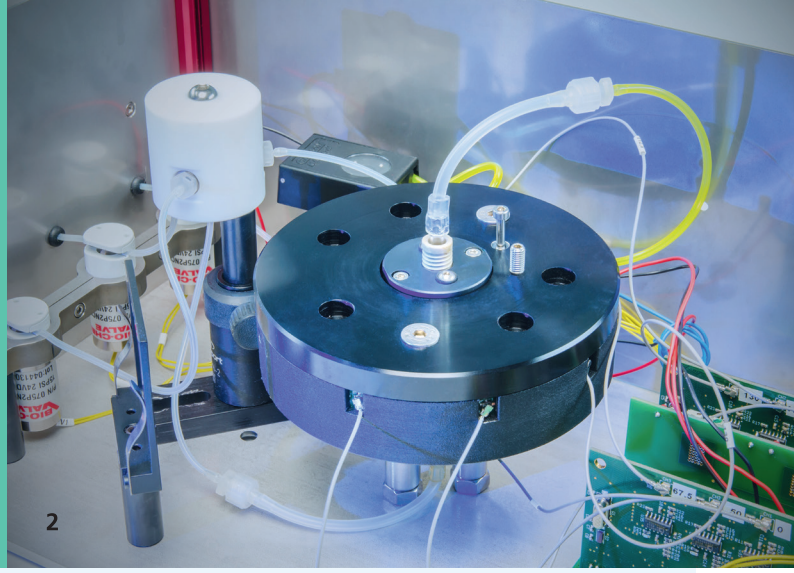

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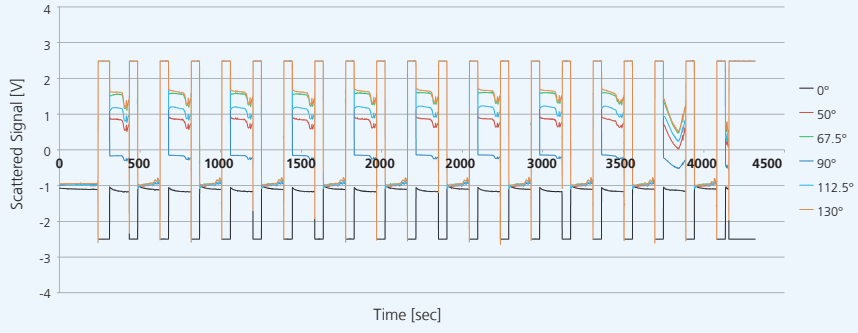
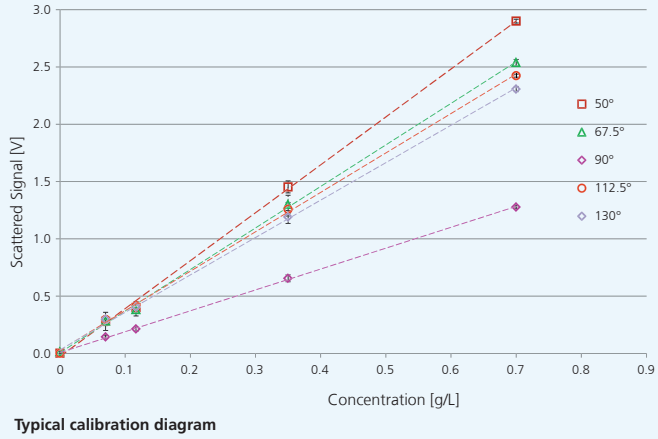
Synthetic Amorphous Silica (SAS) Products

One example of an existing, very well described NSM are inherently aggregated, pyrogenic (fumed) silica products with primary particle sizes < 100 nm. These materials can be found e. g. in paints and coatings, in cosmetics and in toners, they are used in accumulators for the automotive sector and for multimedia devices and they are even applied as additives to food.
In a first step of the manufacturing process, the so-called primary particles are formed which are afterwards covalently bonded during aggregation. Hence, no contents of NPs in colloids prepared according to OECD Test Guideline No. 105 from samples of these materials are to be expected. Nevertheless, NPs (primary particles or small particle fragments < 100 nm) could theoretically be released by water solubility from the aggregates.



Example for time and angle resolved, fully automated data acquisition



Typical calibration diagram

Proof-of-concept demonstrator

As a result of a feasibility study on behalf of an industry consortium, a proof-of-principle demonstrator has been developed by Fraunhofer IMM which had to be sensitive enough for the detection of NPs from pyrogenic silica products in concentrations near the solubility limit of 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 in aquatic colloids. It is suitable for in-site characterization and for product monitoring both in a certified stationary and in a mobile test laboratory. The demonstrator includes a blue laser (λ = 405 nm) and commercially available, electronically amplified silicon photodiodes. It allows a fully automated, time (2 Hz) and angle resolved data acquisition from 6 detectors (5 scattering angles plus transmission signal). In-flow measurements are performed on alternating plugs of sample and reference (water).

Validation of the demonstrator

For the validation and calibration of the demonstrator, a commercial colloidal silica (nominal size: 26 nm) was used after extensive characterization and comparison with complementary methods. For this colloid, the level of detection (LOD) was quantified to be 2 mg/L under GLP conditions. The particle size could be determined with an accuracy of approx. 15 % and for low concentrations, the concentration values have been calculated with an accuracy of approx. 30 %.

1 *Proof-of-concept demonstrator, general view*

2 *Proof-of-concept demonstrator, inner design*