Procedure for solubility testing of NM suspension

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Version
1.0 English

Content

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1 Scope

This Standard Operating Procedure (SOP) describes the preparation procedure for solubility testing of NM suspension.
Note: Not described is the actual chemical element analysis.

2 Basics

In this SOP the preparation procedure for solubility testing of NM in aqueous solvent (deionized water and cell media) is described. The solubility is the property of a (nano)material to dissolve in a solvent. The solubility is a common characterization parameter for NM as dissolved ions of some NM can be more reactive as the solid material. Additionally the dissolution of NM has been shown as a characterization parameter linked to potential toxicity for some NM. In consequence, defined solubility tests are necessary to investigate how much NM remains as solids and how much is available in ionic form.

2.1 **Materials**

3 Materials & Instruments

3.1 **Materials**

The following materials, chemicals and instruments are required:

- HPLC Grade water, cell media
- Nanomaterial
- Spatula
- Pipette
- Centrifuge vial 50 ml
- Rack for centrifuge vials
- Pipette (10 ml)
- Disposable syringe (B. Braun Inject 10 ml)
- Syringe filter (0.22 microns Cameo 30N Syringe Filter Nylon, GE Water & Process Technologies, PA 0,20µm nylon, Roth KC84; 0,20µm syringe filter ∅ 33 mm , Rotilabo KY61.99)

3.2 **Instruments**

The following instruments are required:

- Overhead-shaker (60 revolutions / minute)
- Centrifuge (5000 G)
- Analysis balance (Resolution 0.01 mg)

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4 Experimental procedure

Briefly, NM is weighted in a vial with a certain concentration before overhead-shaken for a defined time period. Afterwards the sample is centrifuged and filtered before the chemical mass determination via Inductive Coupled – Optical Emission Spectrometry (ICP-OES),

4.1 Suspension preparation

The NM is weigh in a correspondingly labeled vial and the mass is documented. Target value for the concentration of the suspension is 1 mg / mL with a volume of at least 40 mL. This results in a test sample of 40 mg (± 1%) of the solid NM and 40 ml (± 1%) of the medium in a centrifuge tube.

4.2 Shaking

The sealed tubes are fixed in an overhead shaker and shaken for a certain period (e.g. 4 h, 24 h, 72 h) at 60 revolutions / minute. Meanwhile, the room temperature is recorded (1 hour-values), since the temperature also influences the solubility. After shaking, the sample is immediately transferred to the centrifuge.

4.3 Centrifugation and filtration

The centrifuge vials are placed in the instrument and centrifuged for 45 minutes at 5.000 G. After centrifugation the vials are carefully removed from the centrifuge and transported in an upright position until filtration, to minimize a mixing of the solid and aqueous fraction.
For the following filtration the supernatant (3 x 10 mL) of each sample is taken by a 10 mL pipette and filled into a disposable syringe (B. Braun Inject 10 ml), with a syringe filter (0.22 microns Cameo 30N Syringe Filter Nylon, GE Water & Process Technologies, PA 0,20µm nylon, Roth KC84; 0,20µm syringe filter ∅ 33 mm , Rotilabo KY61.99). The filtered supernatant is then filled in a labeled vial for quantification of the soluble fraction.

5 Further sample handling

The filtered sample is according to the suggested further analysis (for example chemical identification via ICP-OES) stored or processed.

6 Safety precautions

In general when handling the nanomaterials, protective clothing and suitable gloves have to be worn at any time and the working area, as well as the used materials and instruments, have to be labelled. Please follow the safety information of the instrument manufacturer and material provider.

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7 Waste disposal

Please follow the disposal advice of the material provider, if available.

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