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English - Or. English

**ENVIRONMENT DIRECTORATE
JOINT MEETING OF THE CHEMICALS COMMITTEE AND
THE WORKING PARTY ON CHEMICALS, PESTICIDES AND BIOTECHNOLOGY**

**DOSSIER ON TITANIUM DIOXIDE
- PART 1 - NM 105
ANNEX 11**

**Series on the Safety of Manufactured Nanomaterials
No. 54**

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Standard operation procedure - Preparing Titanium Dioxide (TiO₂) – P25 nanoparticle suspension

Date: 31st January 2011

Version: 1.0 english

Scope

Aim of the SOP

Background

Preliminary results

Preparing suspension

Measuring procedure

Aim of the Standard Operating Procedure (SOP)

The aim of this Standard Operating Procedure is the preparation of a stable nanoscale Titanium dioxide suspension for environmental testing within the UFOPlan Project 3709 65 417¹ and afford reproducible results in different laboratories (Comprehensible proceedings).

The SOP describes the proceedings which are suitable for preparing a stable nanoparticle suspension in this project ("Mobility, fate and behaviour of TiO₂ nanomaterials in different environmental media" in framework of the UFOPlan 2010 FKZ: 3710 65 414, UBA-FB 001741/E by Nickel et al. 2013).

Background

Suspension Requirements

The suspension must be stable at least for 24h (a variance of 10 % is accepted).

An appropriate stability of a suspension is declared as a constant particle size distribution, concentration and zeta potential.

¹ Kuhlbusch et al. 2012: "Fate and behaviour of TiO₂ nanomaterials in the environment, influenced by their shape, size and surface area". Hrsg.: Umweltbundesamt, Report 25/2012, FKZ 3710 65 417, UBA-FB 001577, ISSN 1862-4804,

Stability criteria

- Optical observation (no visible sedimentation of the particles)
- size of the particles in the suspension
- zeta potential
- particle concentration
- pH value of the suspension
- conductance of the suspension

Necessary Instruments

A sensitive analytical balance.

Ultrasonication equipment with sufficient rated power.

Sensitive instrument detecting the particle size distribution and the zeta potential in aquatic media.

Used instruments

In this Project an ultrasonication equipment (Bandelin Sonoplus HD2200 ultrasonic homogenizer 200 Watt, Sonotrode VS70T) was used to disperse TiO₂ P25 nanoparticle in an aqueous suspension.

The particle size and the zeta potential of the suspension were measured using a DLS instrument (Delsa-Nano CS – Beckman Coulter / Zeta Sizer ZS - Malvern Instruments; Nanophox – Sympatec, size only).

Preliminary results

Preliminary results show that a sufficient stability is warranted if the suspension (100 mg P25 / 100 mL deionised water in a 250 ml beaker glass) was sonicated for 10 minutes.

Preparing suspension

- For preparing suspension deionised water was used (pH 5.0 - variance of 10 %)
- A defined amount of the Nanomaterial - here 100 mg of the dry powder was weighted in a 250 mL beaker glass (a variance of 1 % is accepted)
- After this 100 mL of deionised water was carefully added to the material

- The beaker glass with the nanoparticle suspension was sonicated with Bandelin Sonoplus HD 2200 ultrasonic homogenizer for 10 minutes*
- The horn of the ultrasonic homogenizer was dipped into the suspension and placed in the middle of the beaker glass with a distance between horn and bottom of the beaker glass of approximately 1 cm
- For sonication the beaker glass with the suspension was put in a bigger beaker glass with cold/ice water to minimize the heating of the suspension during the sonication
- After use the horn was cleaned with ethanol and afterwards with deionised water
- After sonication the suspension was characterised to its size distribution – using a DLS Instrument

* the sonication time must be adapted to the volume of the prepared suspension, diameter of the beaker glass, the concentration of the nanoparticles and the rated power of the ultrasonic instrument