DOSSIER ON TITANIUM DIOXIDE  
- PART 5 - NM 103  
ANNEX 2

Series on the Safety of Manufactured Nanomaterials  
No. 54

This document is only available in PDF format.
Standard operation procedure – Preparing titanium dioxide (stock) suspension for coating stability experiments

Date: 16th July 2012
Version: 1.0 english

Scope
Purpose
References
Requirements for the suspension stability
Instrument specification
Sample preparation
Data Evaluation / Reporting
Occupational safety
Waste disposal

Purpose
The aim of this Standard Operating Procedure is the preparation of a stable nanoscale Titanium dioxide suspension for environmental testing. The actual procedure based on an established SOP from a previous study (Kuhlbusch et al., 2012). This SOP affords reproducible results in different laboratories (comprehensible proceedings) and describes the proceedings which are suitable for preparing a stable TiO₂ nanomaterial suspension. The SOP was established in framework of the study “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.

References
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,
Requirements for the suspension stability

Suspension Requirements
- The suspension must be stable at least for 24 h (a variance of 10 % is accepted).
- An appropriate stability of a suspension is declared as a constant particle size distribution and/or zeta potential.

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.

Instrument specification

Necessary instruments
- Sensitive analytic balance.
- Ultrasonic homogeniser with sufficient rated power (min. 200 Watt – e.g. Sonoplus HD 2200 from Bandelin GmbH & Co. KG and UDS 751 from Topas GmbH)
- Sensitive instrument detecting the particle size distribution and the zeta potential in aqueous media (e.g. dynamic light scattering instrument - Delsa-Nano C from Beckman Coulter; HPPS from Malvern Instruments)

Necessary materials
- Tested nanomaterial
- Deionised water

Sample preparation
- Weigh a defined amount of the nanomaterial (e.g. 50 mg of the solid material) in a 100 mL beaker glass, a variance of 1 % is accepted.
- After this add deionised water (e.g. 50 ml) with pH 5.0 (variance of 10 %) carefully to the material.
- Wait until the nanomaterial has become wetted and is completely submerged in the liquid phase, 10 min.
• Afterwards homogenise the suspension by continuously, moderate agitation with an overhead or magnetic stirrer by around 350 rpm for at least 15 minutes.

• Disperse the nanomaterial suspension with an ultrasonic homogeniser with 200 W power for at least 1 minute (here 1 min for surface characterisation and 10 min for analysis of the carrier function).

• The probe of the ultrasonic homogeniser should be dipped into the suspension and placed in the middle of the beaker glass with a distance between probe and bottom of the beaker glass of approximately 1 cm.

• During the sonication the beaker glass with the suspension can put in a bigger container with cold/ice water to minimize the heating of the suspension during the sonication.

• After use the probe will be cleaned with ethanol and afterwards with deionised water.

• Sonication is followed by at least 60 min of moderate stirring (overhead stirrer 350 rpm or magnetic stirrer).

• Every stock 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 nanomaterials and the rated power of the ultrasonic instrument.

**Data Evaluation / Reporting**

The particle size distribution as well as the zeta potential of the TiO₂ suspensions was measured as stability criteria. The results were presented as intensity weighted z.average in nm and the zeta potential in mV. The measurement precision of the size measurements was proofed with PSL standard solutions of 20 nm ± 1 nm (Nanosphere NIST size standard) and 182 nm ± 5 nm (BS particle GmbH) as well as aluminium oxide solution for the zeta potential +50 mV ± 5 mV (Microtrac Reference material).

**Occupational safety**

Every measurement campaign must be conducted based on a risk assessment and the requirements of occupational safety regulations. 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 labeled. Furthermore, the laboratory regulations regarding these materials have to be followed. A fume hood has to be used whenever possible.
Waste disposal
Nanomaterial containing waste has to be collected and disposed of separately. It has to be differentiated between the kind of nanomaterial.
<table>
<thead>
<tr>
<th><strong>Title:</strong></th>
<th>Suspension preparation P25 nanomaterial</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>This SOP is valid from:</strong></td>
<td>16.07.2012</td>
</tr>
<tr>
<td><strong>Version:</strong></td>
<td>First Version</td>
</tr>
<tr>
<td><strong>Responsible person of the implementation of the SOP:</strong></td>
<td>user</td>
</tr>
<tr>
<td><strong>Previous changes:</strong></td>
<td>first version</td>
</tr>
<tr>
<td><strong>Information for:</strong></td>
<td>user safety administrator laboratory and division manager</td>
</tr>
<tr>
<td><strong>Author:</strong></td>
<td>Carmen Nickel</td>
</tr>
<tr>
<td><strong>Counterchecked:</strong></td>
<td>Dr. Frank Babick</td>
</tr>
<tr>
<td><strong>Approved:</strong></td>
<td>Dr. rer. nat. T. Kuhlbusch</td>
</tr>
</tbody>
</table>