DOSSIER ON TITANIUM DIOXIDE
- PART 5 - NM 103
ANNEX 3

Series on the Safety of Manufactured Nanomaterials
No. 54

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JT03383142

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Standard operation procedure (SOP) – ICP-OES measurements of Aluminium and Silicon in solid samples

Date: 16th July 2012

Version: 1.0 english

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Necessary materials
Digestion method
Data Evaluation / Reporting
Quality control measures
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Purpose
The standard work specification must be applied for chemical measurements using Inductive coupled plasma optic emission spectrometry (ICP-OES) to detect the aluminium and silicon concentration in dry solid samples. 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.

Instrument specification
Manufacturer: Thermo Scientific
Version: ICAP 6000 Series

Necessary materials
- H₂O₂ (30%)
- HNO₃ (65%)
- NaOH (solid)
- PTFE Teflon Filter 0.45 µm
- Deionised water

Digestion method
Aluminium
100 – 200 mg of the solid materials was mixed with 9 mL HNO₃ (65% ROTH) and 1 mL H₂O₂ (30% ROTH) followed by a microwave digestion (temperature program - 3 min 20 to 130 °C, 5 min 130 °C to 210 °C and 7 min at 210 °C). Afterwards the sample were filled up to a volume of 25 mL

Silicon
100 – 200 mg of the dry powder were places in a platinum cup. 1000 mg NaOH (solid) were added and mixed with the powder. Afterwards the mixture was heated for 20 min at 600 °C. Afterwards 50 mL ultrapure water was added. If necessary the samples were filtered with a 0.45 µm PTFE - teflon filter.

Data Evaluation / Reporting
Measurement preparation and implementation as well as the data evaluation of the measurements must be documented in a test report.

Quality control measures
- The homogeneity of variance was verified according to DIN 38402, part 51.
- Comparison and (if necessary) adjustment of the instruments in the laboratory before each measurement campaign
- Reference material for the examination of the digestion method, chemical analysis and limit of detection of the applied technique was verified according to DIN EN ISO 11885.

According to the calibration, the limit of detection (LOD) of this digestion method is around 1 µg/L for aluminium and 1.5 µg/L for silicon.
Potential error sources when conducting measurements
- Digestion method → incomplete digestion of the material
- Loss of the substances at the vessel walls
- In any case a detailed record of measurements must be kept in which all possible disturbances are noted with time stamps

Occupational safety
Every measurement campaign must be conducted based on a risk assessment and the requirements of occupational safety regulations. This includes in particular the regulation of responsibilities and of the interfaces to occupational safety and health on the premises where the measurements shall be conducted. 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.
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<tr>
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<td>Carmen Nickel</td>
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<td>Fritz Luther</td>
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