
Standard operation procedure – Transport experiments in unsaturated soil columns according to OECD 312 (2004)

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Version

1.0 English

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1 Aim of the Standard Operating Procedure (SOP)

The aim of this Standard Operating Procedure is intended to provide information for the performance of the leaching experiments of nanomaterials in soils within the Project nanoGRAVUR. The SOP describes the proceedings which are suitable for assessing the fate and behaviour of nanomaterials in a soil column.

2 Principle of the test

Columns of inert material (here glass) were packed with soil. The soil was saturated and equilibrated with a 0.01 M CaCl₂ solution (artificial rain) and allowed to drain. Afterwards the suspension was applied to the column. The mobility of the nanomaterial was detected by analysis of the eluate and different segments of the soil.

3 Description of the test method

- The glass column with an inner diameter of 4cm and a height of 20cm were filled with air dried, sieved soil (< 2 mm) to a height of 10 cm. The soil was added to the column in small portions with a spoon. Soiltype A01 – Dystric Cambisol was used (www.Refesol.de).
- Each column is wetted with the electrolyte solution (here 0.01M CaCl₂) from bottom to the top over night using a flow rate of 0.2 ml / cm² * h.
- After the water level reached a height of min. 10 mm over the soil surface the column was allowed to drain for 10 minutes.
- Afterwards 10 ml of the nanomaterial suspension or 10 ml DI water (deionized water) for the blank was directly applied to the soil surface using the peristaltic pump and a flow rate of 0.2 ml / cm² *h.
- Afterwards the column is topped with quartz fiber filter circles.
- The irrigation tubing is attached to the column top. Each column is irrigated with the electrolyte solution with a flow rate of 0.2 ml / cm² *h for 48 h.
- During irrigation eluate is collected in a time resolved manner, after 4h, 24h and 48h.
- After irrigation is finished 1 cm thick substrate layers are taken. For this, the substrate is carefully removed from the top of the column using a spoon.
- The substrate samples are air-dried (19-25 °C for min. 48h). Afterwards they are weighted and

grounded using a ball mill, to guarantee a homogeneous distribution of the Nanomaterial in the sample.

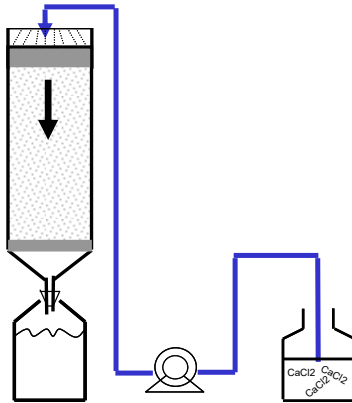


Fig1. Irrigation of the test column

The test was performed in duplicate with one blank.

4 Chemical analyses

The following analyses are performed:

Quantitative analysis of the eluate and substrate samples with ICP MS (Inductive Coupled Plasma Mass Spectrometry) after microwave digestion. SEM (Scanning Electron Microscopy) analyses of selected substrate samples in order to optically verify the presence of the nanomaterials on the substrate and to determine in which form the particles are present (single particles or agglomerates)

5 Data analysis

For data analysis, the nanomaterial concentration in the substrate is plotted against the column length. Moreover, the time dependent nanomaterial or ion concentration in the eluate as well as the SEM analyses is taken into consideration when evaluating the mobility.

The remaining samples are stored in dark at 4°C in case further analyses are necessary.

6 Quality assurance

In order to show the reproducibility of the experiments, for each nanomaterial and condition a minimum of two column-experiments as well as one blank experiment are performed. During the experiments, the conditions (e.g. pH value, pump rate) are controlled and documented.

7 Safety precautions

Handling of nanomaterials has always to be done with special care and according to the information of the material data sheets. This means e.g. that 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.

8 Waste disposal

Nanomaterial containing waste has to be collected and disposed separately.

<p>Title: Transport experiments in unsaturated soil columns</p>
<p>This SOP is valid from: 18th April 2017 This SOP replaces the SOP from: second version from 31st March 2015</p>
<p>Responsible for the implementation of this SOP: Measurement personal</p> <p>Previous changes: Primary and secondary version</p> <p>Distribution:</p> <ul style="list-style-type: none"> Management Head of department Measurement personal Quality management
<p>Date/Signature</p> <p>Author: _____ Dr. Carmen Nickel</p> <p>Reviewed: _____ Dr. Bryan Hellack</p> <p>Approved: _____ Dr. Christof Asbach</p> <p>Approved: _____ Dr. rer. nat. T. Kuhlbusch</p>