

# Silylation: A Reproducible and Readily Applicable Method for Characterization of Non Extractable Residues (NER) of Chemicals and Pesticides in Soil and Sediment

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## Introduction

All chemicals and pesticides form NER in soils and sediments in varying amounts. Three NER types can be differentiated: type 1 comprise sequestered residues, type 2 those covalently bound and type 3 biogenic residues<sup>1</sup>. Silylation has been suggested as a methodology to differentiate type 1 and type 2 NER. But concern has been addressed that silylation is not suitable for routine analysis, e.g., in the frame of studies for the purpose of authorisation and registration. We here show a readily applicable and reproducible experimental procedure to use silylation for analysis of NER. Since also biogenic residues can be quantified<sup>2,3</sup> (not shown here), all three NER types can now be quantitatively assessed.

## Equipment needed

Beside normal laboratory equipment the only special equipment needed are Schlenk flasks and gas bags. Further, argon gas supply is needed.

The reaction needs to be conducted in a fume hood, which limits – due to its size – the number of simultaneously treated samples to 8 (see photo “silylation workstation”). A larger fume hood or the use of two fume hoods will allow more samples to be processed in parallel.

## Results and Conclusions

- ✓ For the proof of method reproducibility 42 <sup>14</sup>C-NER containing samples were silylated in duplicate. The amount of <sup>14</sup>C-radiolabel released was determined by LSC. The Mann-Whitney-U-Test (also known as Wilcoxon rank-sum test) was used to test, whether significant difference between the respective duplicates exist which was not the case (p-value 0.78 > 0.05). We conclude that silylating NER containing soil can be performed reproducibly under routine conditions.
- ✓ Silylation extracts obtained by this procedure, are in most cases non turbid, slightly colored solutions, which are easily to handle for further radio-TLC analysis. Due to high volatility of the solvents used (Chloroform, Acetone), application to TLC-plates is a standard procedure (for non-volatile test substances). Also HPLC-MS analyses can be performed after solvent exchange.
- ✓ The time necessary to get the final silylation extract is around 26 hours including washing steps that are not shown in detail in the step by step photo documentation shown on the right side.
- ✓ Stability of the parent test substance has to be verified beforehand in a pre-test with parent test substance spiked blank matrix. In case of instability EDTA extractions can be performed<sup>4</sup>.
- ✓ Hydrophilic test substances might not be extracted by the solvents used even after silylation of the matrix. Substance released by silylation should be extracted from the silylation residue with a more polar solvent like Methanol or Acetonitrile in this case.

## Silylation laboratory procedure step by step.

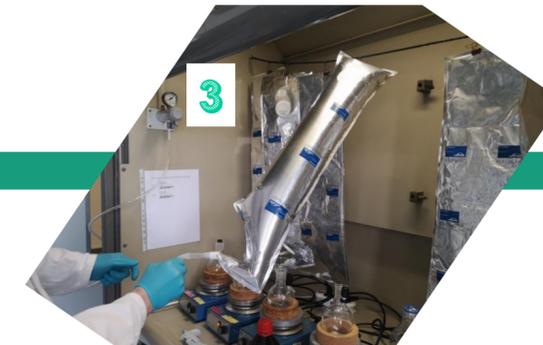
All photos on this page  
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1 Weighing of 1.5 g sample into Schlenk flask



2 Drying at 105 °C for 30 min



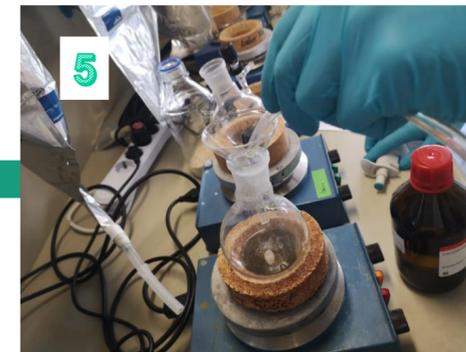
3 Filling of gas bag with Argon (Linde PLASTIGAS® bag 5.5 L)



Silylation workstation



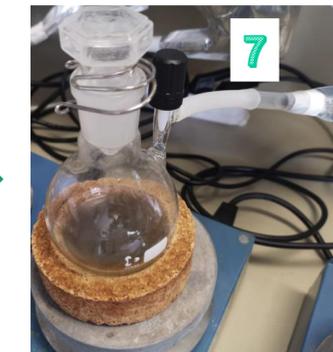
4 Addition of chloroform and NaOH



5 Flushing of Schlenk flask with Argon



6 Addition of reagent TMCS under Argon



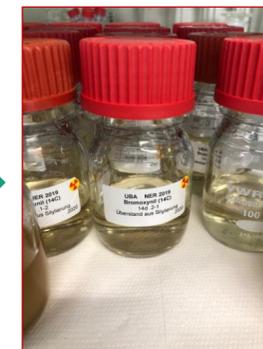
7 Connection of gas bag, stirring (100-200 rpm) over night at room temperature



8 Transfer to centrifuge tube,



9 Centrifugation



Silylation extract

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