## ANALYSIS OF SHORT-CHAIN CHLORINATED PARAFFINS (C<sub>10</sub>-C<sub>13</sub>) IN GERMAN RIVER SUSPENDED PARTICULATE MATTER, SEWAGE SLUDGE AND INDUSTRIAL SLUDGE SAMPLES

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## Introduction

Chloroparaffins are produced by chlorination of n-alkanes (C<sub>10</sub>-C<sub>30</sub>) and are used e.g. as high-pressure additives in lubricants in the metal-processing industry or as softeners in plastics such as polyvinyl chloride (PVC) <sup>1</sup>. Technical chloroparaffins consist of a mixture of straight-chain chloroparaffins of different chain length and degree of chlorination. Because of their physical and chemical features, chloroparaffins are hardly biologically degradable under environmental conditions. In examinations, the toxicity especially of short-chain chloroparaffins (C<sub>10</sub>-C<sub>13</sub>) in aquatic organisms as well as the carcinogenic features of this compound class in animal tests could be proved. Therefore, attempts are made on a national and international basis to ban the production and use of short-chain chloroparaffins.

Compared to other chloroorganic compounds only few data on chloroparaffin levels in the environment have been published up to now <sup>2-3</sup>. Because of the complex composition of chloroparaffins the quantification of these substances is very difficult. Thus, no recognised, validated method of analysis for the determination of chloroparaffins in environmental samples exists at the moment, which makes the published data difficult to compare. However, they admit to conclude that chloroparaffins are ubiquitously detectable.

A relatively simple and selective method of analysis for the determination of short-chain chloroparaffins in environmental samples (sludge, suspended particulate matter) has been developed and validated in case of which the quantification is done by GC/MS-NCI. With this method suspended particulate matter from different German rivers and creeks, sewage sludges from municipal sewage plants as well as sludges from industrial sewage plants have been examined for their content of short-chain chloroparaffins.

## **Methods and Materials**

First of all the samples were freeze-dried and 5 g each of the dry sample material were Soxhlet extracted with toluene for 8 hours (which corresponds to about 120 extraction cycles). An aliquot of the resulting raw extract (approx. 2 g of the dry weighed portion) was cleaned from organic matrix contents and sulphur compounds after addition of cis-chlordane as internal standard by means of column chromatography via silica gel/silver nitrate (10 %) and benzenesulphonic acid/silica gel/H<sub>2</sub>SO<sub>4</sub> (44 %). In a further cleaning step, the chloroparaffins could be separated by a fractionation with silica gel (1. fraction: n-hexane; 2. fraction: n-hexane/dichloromethane (1:1)) from interfering chloroorganic compounds such as PCBs. In the second fractionation no PCBs