Determination of PCBs in Baltic Sea sediments using Accelerated Solvent Extraction (ASE)

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Introduction

Polychlorinated biphenyls (PCBs) are ubiquitous pollutants present in all compartments in the environment. Due to their toxicity, carcinogenic potential and ability to interfere with reproductive systems (1) determination of PCBs in environmental samples is of major concern. Extractions are usually performed by means of Soxhlet or sonication. However, these extraction methods generally require several hours or days to perform and large amounts of hazardous organic solvents.

Therefore, new extraction techniques have been developed in order to reduce the volume of solvents required for extraction, reduce the time of extraction, improve the precision of analyte recoveries and reduce the sample preparation costs. Supercritical fluid extraction (SFE)(2,3), microwave-assisted extraction (MAE) (4,5) technique and Accelerated Solvent Extraction (ASE) (6,7,8) are all techniques that are gaining interest due to the above described characteristics.

Accelerated solvent extraction (ASE) is a relatively new technique that utilizes elevated temperatures and pressures to achieve fast and efficient removal of analytes of interest from various matrices. In this study, ASE was used to determine PCBs in Baltic Sea sediments and the results were compared to Soxhlet extraction. A review of the data indicates that ASE gives essentially equivalent data to Soxhlet extraction. However, ASE extractions are performed in less time and with less solvent than the conventional extraction techniques.

Materials and Methods

Sediment samples from the Baltic Sea with different origin and degree of contamination were used in this study. The sediments were homogenized and then centrifuged at approximately 1000 g for 15 min. After removal of the supernatant water, aliquots of each sediment sample were removed and stored at -20 °C until Soxhlet extracted. The sediment residues were air-dried for approximate 48 h. The dried sediment samples were then ground in an automatic grinding machine to homogenous powders, which were kept cool in glass bowls until extraction.

The ASE extractions were carried out using a Dionex ASE 200 (Dionex Corporation, Sunnyvale, CA, USA) at a temperature of 100 °C and a pressure of 14 MPa. A mixture of acetone/hexane (1:1 v/v) was used as extraction solvent. Prior to extraction four ^{13}C-labelled PCBs (nos. 52, 101, 138, 180) were added as internal standards.
Triplicates of each sample were placed in pre-extracted cellulose thimbles and extracted (wet) for 24 h in toluene using a Soxhlet apparatus. To remove the water from the samples a Dean-stark trap was attached to the Soxhlet extractors (9). Prior to extraction four ^{13}C-labelled PCBs (nos. 52, 101, 138, 180) were added as internal standards.

All extracts (provided after the Soxhlet and ASE extractions) were volume reduced and then eluted through a column packed with SiO_2 (deactivated with 10% water (w/w)). The extracts were then treated with copper for the removal of sulphur.

The extracts were then run through a nitropropylphenylsilica (nitro) column (Nucleosil, 5-mm particles, 250x4.6 mm, Macherey-Nagel, Germany) with n-hexane as mobile phase.

In short, three fractions were collected 1. aliphatic/monocyclic aromatic compounds, 2. dicyclic aromatic compounds (including the PCBs), 3. polycyclic aromatic compounds (PAHs with four rings or more) (10).

The extracts (fraction 2), containing the PCBs, was then further cleaned up with multi-layers of modified silica gel before the final GC/MS analysis.

Results and discussion

In the present study several sediments from the Baltic Sea were analysed for PCBs after extraction with ASE. The recoveries compared to Soxhlet in five of the sediments varied between 81 and 114 %. However, two of the sediments showed lower recoveries (48-85%) when ASE was used as extraction method. This might be because the sediments are more difficult to extract and/or high concentrations of pollutants. The ASE method showed equal or better precision than the Soxhlet extraction.

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