

**Middle - Pressure - Liquid - Extraction (MPLE)
of selected Polychlorinated Biphenyls (Ballschmitter PCBs)
from Soil (MPLE II) ¹⁾**

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1. Introduction

Analyses of contaminated soils have been shown to depend mainly on the extraction method used ²⁾. The reason for this might be the inclosure of pollutants in the humic matter of the soil and the different interaction of the solvents with these complexes in the different methods ³⁾. Attempts to find a general scheme of analyses of contaminated soil led to different methods in respect to the pollutant or the matrix characteristics ⁴⁻⁶⁾. We proposed a new concept for extraction of contaminated soil in that the initial step of sample preparation is the grinding of the respective soil with silica gel to alter the matrix. The produced homogeneous powder can be used as a stationary phase of a middle pressure liquid chromatography system ⁷⁾. As we have shown lately, different pollutants can be eluted successively from this by different solvents of increasing polarity ⁸⁾. We will now report about the recovery of the six Ballschmitter PCBs from contaminated soils by this method.

2. Experimental

A solution of the six Ballschmitter PCBs in cyclohexane was prepared (1 µg / ml per PCB) for calibration and contamination purposes. 15 g of sterilised standardized soil (LUFA Speyer Sp 2.1 Charge No. 14292 and Sp2.2 Charge No. 24292) were spiked in an ISO-glass bottle by distributing 200 µl of each PCB solution onto it, followed by 12 hrs shaking in a shaking machine. After adding 15 g of silica gel, the spiked soil was ground in a 500 ml Retsch ceramic ball mill container with 10 ceramic balls of 20 mm diameter (Königliche Porzellan Manufaktur Berlin) for five minutes at 500 rpm to give a homogenous powder. These 30 g of denatured soil were filled into a Büchi MPLC column (23 / 1,5 cm) onto 2 g of silica gel (Lichroprep, Merck) and was treated like dry-filling of a stationary phase ⁹⁾. Cutting fractions of 50 ml, the contaminants were extracted by cyclohexane at a flow rate of 10-15 ml / min. The pressure varies between 2 and 4 bar. No clean up was necessary after elution. The resulting solutions were concentrated to about 1 ml under reduced pressure, and then transferred into a 2 ml volumetric flask.

The contaminants were determined using a Hewlett Packard 5890 II gaschromatograph with a J&W capillary column (DB 5 - 60N, 0.23 mm ID, 0.25 µm film), split/splitless injector and Electron-Capture-Detector (ECD). Quantification was done by means of the calibration curves.

PCB

3 Results and discussion

The modified soils were eluted with cyclohexane which had proved to be best for Hexachlorobenzene (HCB) contaminations⁸⁾. Unspiked soils were extracted first to determine the basic contamination of the matrices. Whereas the total quantity of the measured PCBs in Sandy Soil 2.1 came to about 1 µg/kg, the Soil 2.2, which contained much more humic matter, resulted in nearly the tenfold amount. The basic contamination levels were subtracted from the extraction results of the spiked soils to calculate the recovery rates. Using cyclohexane as mobile phase about 30-50 % of the PCBs eluted within the first two fractions, similar to Hexachlorobenzene, but at least 500ml (ten fractions), were necessary to get recovery rates of 80 % or better (table 1). Recovery rates were sufficient within the first 100ml of the elutant, if one shortened the time of equilibration after spiking (table 2).

Table 1: Relative recovery rate (RR) of the PCBs in forest soil 2.2; A) in the first two fractions (100 ml sample) and B) in ten fractions (500 ml sample).

PCB	28	52	101	153	138	180
RR A (%)	48	44	36	40	29	29
RR B (%)	94	96	81	81	83	93

Table 2: Relative recovery rates of the PCBs in the Soils 2.1 and 2.2 without aging after the spiking procedure.

PCB	28	52	101	153	138	180
soil 2.1 (%)	79	75	87	80	78	74
soil 2.2 (%)	77	68	75	78	87	79

Compared to the binding of the molecules to the organic matter, it seems likely that the solvency of the PCBs by cyclohexane is not strong enough. Unlike the polycyclic aromatic hydrocarbons, the elutionprofile of PCBs showed no significant effect on the modification of the solvent polarity by adding different amounts of acetone¹⁰⁾ In order to economize the solvent use, different solvents and solvent mixtures are still under study to elute successively PAHs and PCBs.

4. References

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