**Ruggedness study investigating extractability of PCB and OCP from soil**

Ulrike Mülow1, Petra Lehnik-Habrink1, Stephanie Näther1, Wolfram Bremser2, Christian Piechotta1

1 BAM Federal Institute for Materials Research and Testing, FB 1.8 Umweltanalytik – Chromatographische Verfahren,

 Richard-Wilstätter-Str. 11, 12489 Berlin, ulrike.muelow@bam.de

2 BAM Federal Institute for Materials Research and Testing, FB 1.4 Prozessanalytik,

 Richard-Wilstätter-Str. 11, 12489 Berlin

The occurrence of persistent organic pollutants like polychlorinated biphenyls (PCB) and organochlorine pesticides (OCP) in the environment is still a matter of concern, even years after their use was abolished. Hence, threshold values are regulated by law which reference a number of national and international standards for determination of said contaminants. In this context, soil is an especially difficult matrix, because several defining parameters can vary in a wide range. Organic matter content in particular has a crucial influence on extractability of contaminants because it largely governs formation of non-extractable or bound residues.

For this study, four soils of different total organic carbon (TOC) content were generated from uncontaminated reference soil (RefeSol 01-A) and compost which were spiked to contaminant levels representative of the Bundes-Bodenschutz-Verordnung (BBodSchV). These test materials were extracted using accelerated solvent extraction (ASE), Soxhlet extraction, and liquid-liquid extraction. For the latter method, samples were initially extracted for 15 minutes employing acetone. The follow-up extraction step was carried out using cyclohexane and the extraction time varied between 15 minutes and 16 hours. After clean-up, extracts were analysed via gas chromatography-mass spectrometry (GC‑MS) and with a gas chromatograph equipped with an electron capture detector (GC‑ECD).

Statistical analysis of the experimental values showed that recovery was not dependent on matrix TOC content. Also, no clear preference for a single method could be detected. Concerning liquid-liquid-extraction, it is merely observed that prolonged extraction time results in slightly higher recoveries, while moderate extraction times appear to yield the most robust results. Although Soxhlet and ASE show high recoveries, they also lead to larger standard deviations and are not suitable for determination of thermolabile pesticides.

In addition to recovery by different extraction methods, the agreement of values obtained by ECD and MS was evaluated. For ECD, standard deviations are generally higher but overall the values obtained by MS and ECD are comparable.