Application Note

One Step Extraction, Cleanup and Direct-to-Vial Concentration For PCBs in Soil and Sediment

Introduction

Polychlorinated biphenyls are a class of organic compounds with one to 10 chlorine atoms attached to a pair of bonded benzene rings. PCBs were widely used for many applications, especially as dielectric fluids in transformers, capacitors, and coolants. Due to PCB's toxicity and classification as a persistent organic pollutant, PCB production was banned by the United States Congress in 1979 and by the Stockholm Convention on Persistent Organic Pollutants in 2001.

PCBs have low water solubility and are largely chemically inert, making them extremely resistant to oxidation. These properties allow PCBs (commonly detected using EPA Method 8082) to resist environmental degradation so they tend to accumulate in soil and river sediment. This method determines the concentrations of various PCB congeners and Aroclors in extracts from solid and liquid matrices, including food products using gas chromatography with electron capture detectors (ECD). This new application speeds sample preparation by combining multiple processes into one. The FMS PLE (Pressurized Liquid Extraction) system automatically performs extraction and sample cleanup using the proprietary FMS InCell column and delivers the extract for final concentration directly to a vial for injection into the GC system. This is a first for analysis of PCBs.

Instrumentation

- FMS, Inc. PowerPrep™ PLE system
- FMS, Inc. 5 gm Acidic InCell Silica Column
- FMS, Inc. SuperVap™ Concentrator system
- Thermo Fisher Scientific Polaris Q GCMS

Method summary

PowerPrep PLE system

1. Extraction solvent: Hexane/Methylene Chloride (50/50)
2. Extraction temperature: 120 ºC
3. Extraction pressure: 1500 PSI
4. Extraction time: 15 minutes

SuperVap Concentrator system

1. Pre-heat temp: 55 ºC
2. Pre-heat time: 15 minutes
3. Heat in Sensor mode: 65 ºC
4. Nitrogen Pressure: 15 PSI

Procedure

1. 10 grams of are sample weighed out in 100 mL beaker, repeated for five replicates per matrix.
2. Samples were mixed and dried with Varian Hydro Matrix®.
3. The dried sample is transferred to a FMS extraction cell equipped with an InCell Acid Silica end cap.
4. Samples are spiked with 1 mL (acetone) of a 1 µg/mL PCB congener solution.
5. The void cell volume is filled with Ottawa Sand®, sealed and loaded on the FMS PLE system for extraction.
6. The sample is extracted and automatically transferred to the FMS SuperVap Direct-to-Vial concentrator system.
7. 5 µL ISTD is added to extract (PCB-209) and the extract transferred to GC/MS for analysis.

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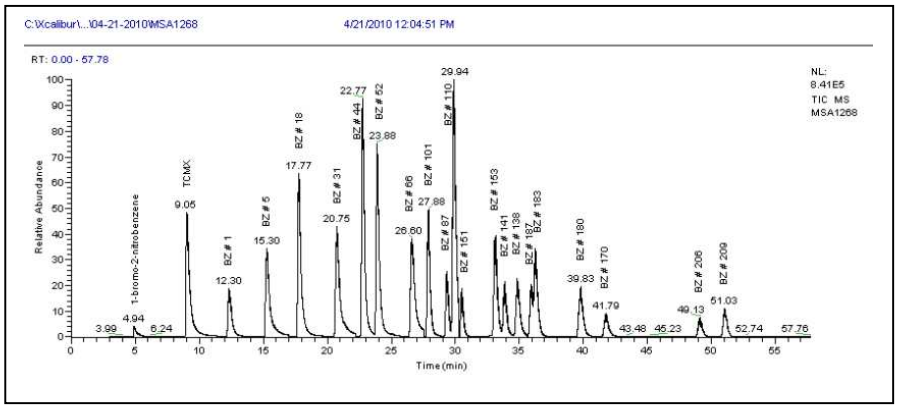
Results

Congener	Spiked µg/kg	Sand Mean Rec. µg/kg	%	Sediment Mean Rec. µg/kg	%
BZ #1	100	114.2	114.2%	75.2	75.2%
BZ #5	100	125.2	125.2%	81.3	81.3%
BZ #18	100	119.3	119.3%	93.3	93.3%
BZ #31	100	120.6	120.6%	85.1	85.1%
BZ #44	100	119	119.0%	85.2	85.2%
BZ #52	100	119.2	119.2%	91	91.0%
BZ #66	100	143.6	143.6%	89	89.0%
BZ #87	100	124.7	124.7%	84.7	84.7%
BZ #101	100	122.8	122.8%	89.8	89.8%
BZ #110	100	111.7	111.7%	84.8	84.8%
BZ #138	100	109.4	109.4%	87	87.0%
BZ #141	100	106	106.0%	92.7	92.7%
BZ #151	100	113.1	113.1%	91	91.0%
BZ #153	100	113.3	113.3%	91.4	91.4%
BZ #170	100	120.8	120.8%	86.2	86.2%
BZ #180	100	100.7	100.7%	95	95.0%
BZ #183	100	100.1	100.1%	95	95.0%
BZ #187	100	103.6	103.6%	87.9	87.9%
BZ #206	100	103.2	103.2%	79.3	79.3%
TCMX (IS)	100	102.1	102.1%	96.9	96.9%



Conclusions

The FMS PLE system and the SuperVap™ Direct-to-Vial Concentration system in combination with the FMS 5 gm Acidic Silica gel InCell column automatically performs the extraction, cleanup and concentration of PCB analytes at a high rate of speed producing consistent recoveries and reproducibility for both soil and sediment samples.

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