Application Note

Automated One Step Solid Phase Extraction and Concentration of PCBs in Drinking Water

Introduction

PCBs are a group of synthetic organic chemicals that contain 209 individual compounds (known as congeners) with varying harmful effects. PCBs enter the environment in mixtures containing a variety of individual components. Seven types of PCB mixtures include 35% of all the PCBs commercially produced and 98% of PCBs sold throughout the world. PCBs don't burn easily and are good insulating materials. PCBs have been widely used as coolants and lubricants in transformers, capacitors and other electrical equipment. The manufacture of PCBs was stopped due to evidence of the harm they cause when they build up in the environment. Because of their durability and wide-spread industrial use, PCBs have found their way into drinking water supplies.

The PowerPrep™ SPE and SuperVap™ Concentrator systems speed up the sample preparation process for the analysis of PCBs by combining sample prep into a single, automated step.

Instrumentation & Consumables

- FMS, Inc. PowerPrep SPE (Solid Phase Extraction) System
- Waters Oasis® 1 gram HLB SPE cartridge
- FMS, Inc. SuperVap Concentrator
- Thermo Fisher Scientific Polaris Q GCMS

Method Summary

PowerPrep SPE system

1. Condition Cartridge: 10 mL MeOH
2. Condition Cartridge: 10 mL H_2O
3. Load Sample: 15 minutes
4. Rinse bottle: 5 seconds
5. Load rinse: 1 minute
6. Dry Cartridge: 30 minutes
7. Elute Sample: 20 mL DCM

SuperVap Concentrator

1. Pre-heat temp: 55 ºC
2. Pre-heat time: 30 minutes
3. Heat in Sensor mode: 65 ºC
4. Nitrogen Pressure: 15 PSI

Procedure

Five, 1 liter water samples were each spiked with a mixture of 19 individual PCB congeners at 1 µg/mL each. Samples were also spiked with a 1 µg/mL tetrachloro-m-xylene solution as an extraction surrogate. Using the PowerPrep SPE system, samples were then loaded on pre-wet Oasis HLB cartridges using vacuum to draw the samples across the cartridge. Sample bottles were then automatically rinsed with DI water, after which the rinse was loaded onto the cartridge. Cartridges were dried using a nitrogen stream blown across the cartridge to remove all remaining water (30 minutes). Once dried, the HLB cartridges were eluted with 20 mL of DCM, allowing the cartridge to soak wetted with DCM for 1 minute. DCM was then purged from the cartridge, directly to the FMS SuperVap Concentrator with direct-to-vial tubes (Figure 2). Extracts were blown down to 1 mL final volume using FMS direct-to-vial concentrator tubes. Extracts were then transferred for GC/MS analysis.

Fig. 1: PowerPrep SPE and SuperVap Concentrator systems.

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Fig. 2. FMS SuperVap™ Concentrator with direct-to-vial concentrator tubes.

Results

Congener	Spiked µg/mL	Mean Recovery µg/L	% Rec	STD Dev.
BZ #1	1	.911	91.1%	0.13152
BZ #5	1	.916	91.6%	0.16226
BZ #18	1	.903	90.3%	0.02089
BZ #31	1	.905	90.5%	0.02668
BZ #44	1	.898	89.8%	0.02869
BZ #52	1	.897	89.7%	0.02518
BZ #66	1	.907	90.7%	0.03209
BZ #87	1	.913	91.3%	0.03405
BZ #101	1	.905	90.5%	0.032893
BZ #110	1	.916	91.6%	0.03250
BZ #138	1	.900	90.0%	0.03206
BZ #141	1	1.005	106.0%	0.03216
BZ #151	1	.895	89.5%	0.03165
BZ #153	1	.907	90.7%	0.31756
BZ #170	1	.896	89.6%	0.03248
BZ #180	1	.915	91.5%	0.03151
BZ #183	1	.924	92.4%	0.03215
BZ #187	1	.914	91.4%	0.03139
BZ #206	1	.884	88.4%	0.03529
TCMX	1	.865	86.5%	0.02589

Conclusions

The results demonstrate that the FMS PowerPrep SPE system combined with the FMS SuperVap Concentrator and Waters Oasis cartridges can rapidly and accurately extract PCB samples that produce reproducible recoveries from water samples.

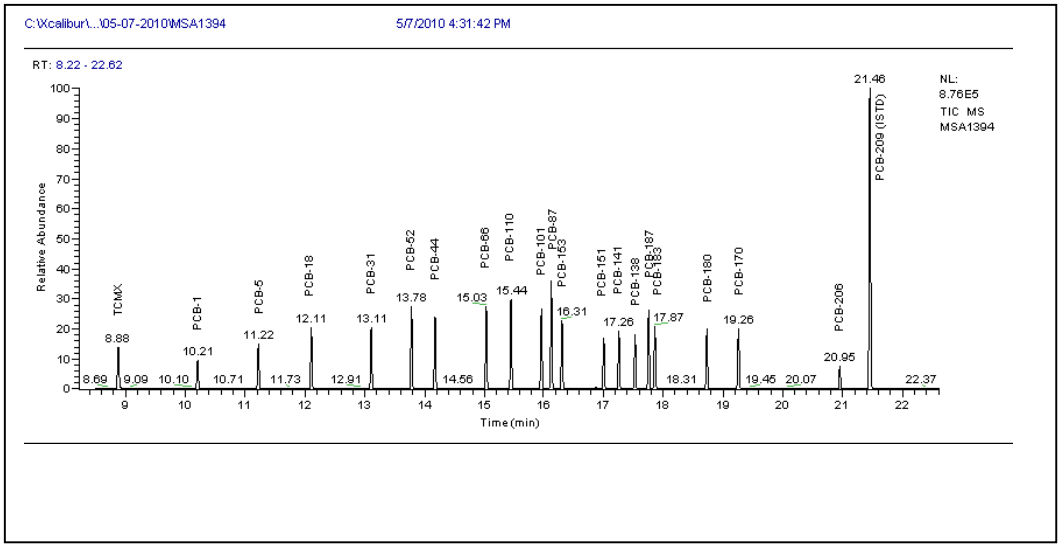

Fig. 3. Results of PCB congeners in sample extract.

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