

EPA 608.3 Automated Solid Phase Extraction of Organochlorine Pesticides with In-line Florisil Cleanup Using FMS TurboTrace® ABN System

Introduction

Organochlorine Pesticides are among the more notorious organic pollutants. Gaining wide spread attention in the later part of the 20th century, and ultimately leading to their ban, OCPs remain with us as a legacy contaminant in the environment. In water samples, OCPs are monitored by the U.S. Environmental Protection Agency by various methods including EPA 508, EPA 608 and EPA 8081.

The use of Solid Phase Extraction (SPE) can rapidly increase both extraction efficiency and reduce lab solvent usage. By implementing the FMS TurboTrace® ABN system with its dual cartridge functionality, an in-line Florisil cartridge can be added to perform automated extract clean-up during the elution step.

Instrumentation

- FMS, Inc. TurboTrace® ABN SPE system
- FMS, Inc. SuperVap® 12 Concentrator
- 55 ml SuperVap Tubes vials
- Agilent 7890 GC with μ ECD detector

Consumables

- FMS Inc 1 gram C18 SPE cartridges
- FMS Inc Florisil SPE cartridges
- Acetone, pesticide grade or equivalent
- n-hexane, pesticide grade or equivalent
- Ultrapure DI water
- 6N HCL
- Anhydrous Sodium Sulfate, ACS grade or equivalent

Sample/Reagent Prep

1. Add 7 grams NaSO₄ to Florisil cartridges.
2. Affix C18 cartridge to position 1 and Florisil cartridge to position 2
3. Bring samples to pH 2 adding HCL drop wise
4. Add add methanol to samples (5mls per 1 Liter)
5. Add respective surrogate and spiking solutions to samples
6. Attach sample bottles to SPE system

SPE Procedure

1. Condition Florisil/NaSO₄ SPE cartridge with 20mls 10% Acetone in Hexane
2. Condition C18 SPE cartridge with 10mls Acetone
3. Condition C18 SPE cartridges with 20mls H₂O
4. Load Samples across C18 SPE cartridges via vacuum
5. Dry C18 Cartridge with N₂ for 10 minutes
6. Automatic solvent rinse of sample bottle with 10% acetone in hexane
7. Bottle rinse loaded across both cartridges and collected in SuperVap concentrator
8. Additional 10 mls elution solvent passed C18 cartridges
9. 10 mls elution solvent passed across Florisil cartridge and nitrogen purged to SuperVap

Total elution solvent: ~40mls



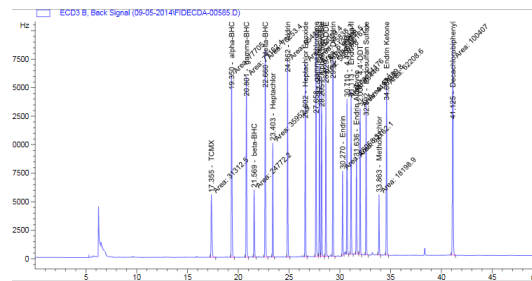
SuperVap

1. Preheat temperature: 55 °C
2. Evap mode: 7-10PSI Nitrogen with sensor

Results:

Analyte	Mean Rec	RSD
α -BHC	105.9%	2.2%
γ -BHC (Lindane)	105.1%	2.0%
β -BHC	98.8%	2.1%
δ -BHC	110.8%	2.1%
Heptachlor	100.3%	9.8%
Aldrin	76.4%	6.9%
Heptachlor Epoxide	101.3%	1.9%
γ -Chlordane	92.1%	3.1%
Endosulfan I	94.9%	2.0%
α -Chlordane	99.7%	2.0%
4,4'-DDE	93.9%	2.6%
Dieldrin	102.0%	2.1%
Endrin	110.5%	2.7%
4,4'-DDD	102.9%	3.7%
Endosulfan II	101.8%	2.7%
Endrin Aldehyde	84.1%	5.8%
4,4'-DDT	103.9%	4.1%
Endosulfan Sulfate	103.5%	3.0%
Methoxychlor	100.1%	6.3%
Endrin Ketone	99.8%	4.5%
Surrogate		
TCMX	72.3%	5.7%
PCB-209	78.7%	11.2%

Results of IPR study performed on Turbo Trace ABN system with in-line Florisil



Chromatogram of an LFB extraction at .5 µg/L

Conclusions

Extraction of the water samples performed on the TurboTrace ABN system enabled a fully automated extraction process due to its dual cartridge capabilities. Samples were loaded across the C18 cartridges, then eluted through the Florisil cartridges in succession. The addition of NaSO₄ to the clean-up cartridge enabled residual water removal from the extract without a separate, manual step.

Analytical results yielded spiked recoveries of Organochlorine pesticides to all be between 70-130%, with low RSDs. Analysis of Method blanks showed no residual background or carryover on the SPE system. Comparisons of dirty extracts using Florisil with extracts conventionally extracted with no clean-up procedure showed effective removal of non-target interferences and minimal chromatographic interferences using the in-line clean-up procedure.

The ability to eliminate an entire evaporation step, paired with a methylene chloride free extraction resulted in both a more efficient extraction process, as well as a more environmentally friendly one as well.



FMS TurboTrace ABN and SuperVap System

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