

Automated Solid Phase Extraction (SPE) of Organochlorine Pesticides in Water Using EPA Method 508



Introduction

Organochlorine pesticides are man-made organic chemicals with a history of wide spread use in both the United States as well as globally. Tending to be very persistent in the environment, they have found their way into sediments and drinking water supplies posing serious health risks. Organochlorines have a wide range of both acute and chronic health effects, including cancer, neurological damage, and birth defects. Many organochlorines are also suspected endocrine disruptors.

Liquid/liquid extraction via a sep-funnel is the traditional method used for the extraction of organochlorine pesticides. Not only is it time consuming, it also requires a large volume of chlorinated solvent. The result is high cost and low reproducibility. The automated, solid phase extraction method (EPA 508) described below allows for rapid, reproducible extractions using a minimal volume of solvent that produce consistent results.

Instrumentation

- FMS, Inc. TurboTrace[®] SPE (Solid Phase Extraction) System
- FMS, Inc. SuperVap[®] Concentration
- FMS, direct-to-vial concentrator tubes
- Agilent 7890A GC with uECD

Consumables

- FMS, Inc. 1 g C-18 cartridge
- Sodium sulfate
- Ultra pure DI water
- Fisher 6 N Hydrochloric Acid
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Dichloromethane
- Fisher Pesticide Grade Ethyl Acetate
- 508 spiking Standards

Sample Prep

- 6 samples (1L water each) are prepared and acidified with 1 mL HCl till pH ~ 2
- Spike with Standards
- Put sample bottles in place and fill rinse bottles with 5 mL EtOAc or 5 mL methylene chloride (step 7 below)
- Cartridges are installed in each of the six positions

TurboTrace SPE

1. The C18 Cartridges are conditioned with 5 mL of 1:1 methylene chloride/ethyl acetate
2. The C18 Cartridges are conditioned with 5 mL methanol
3. The C18 Cartridges are conditioned with 5 mL DI water
4. The samples are loaded onto the C18 Cartridges via vacuum
5. The C18 cartridges are dried with nitrogen 10 min
6. Sample bottles rinsed with 5 mL EtOAc and loaded across cartridges
7. Same with 5 mL methylene chloride
8. Rinse cartridges with 2 x 3 mL of 1:1 methylene chloride/ethyl acetate and collect
9. Dry extracts across sodium sulfate, rinse with 2 x 3 mL of 1:1 methylene chloride/ethyl acetate and collect

FMS SuperVap[®]

- Pre-heat temp: 40 °C
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 40 °C under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis



Figure 1: TurboTrace SPE system.



Results

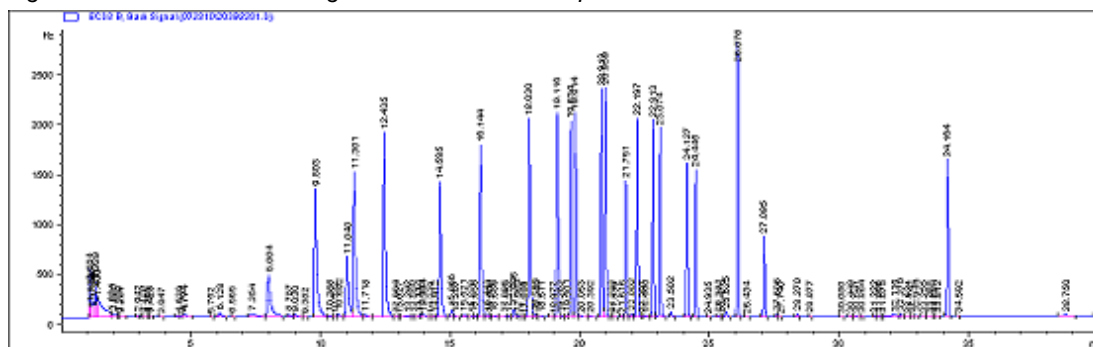
Table 1: Mean recovery and Standard deviation for five replicates

Compound	Spike Conc.	Avg Rec.	STD Dev.
TCMX	.1 ug/L	70.0%	5.1%
Alpha-BHC	.1 ug/L	81.6%	2.0%
Beta-BHC	.1 ug/L	93.9%	4.7%
Gamma-BHC	.1 ug/L	83.1%	4.7%
Delta-BHC	.1 ug/L	98.9%	5.9%
Heptachlor Aldrin	.1 ug/L	80.0%	4.5%
Heptachlor Epoxide	.1 ug/L	89.8%	5.2%
Gamma-Chlordane	.1 ug/L	81.0%	4.6%
Endosulfan I	.1 ug/L	87.8%	4.7%
Alpha-Chlordane	.1 ug/L	82.9%	4.5%
Dieldrin	.1 ug/L	85.9%	4.7%
4,4"-DDE	.1 ug/L	84.0%	4.7%
Endrin	.1 ug/L	70.6%	5.3%
Endosulfan II	.1 ug/L	90.5%	4.8%
4,4'-DDD	.1 ug/L	81.7%	5.1%
Endrin Aldehyde	.1 ug/L	119.1%	5.9%
Endosulfan Sulfate	.1 ug/L	95.0%	5.1%
4,4'-DDT	.1 ug/L	96.2%	6.4%
Endrin Ketone	.1 ug/L	110.9%	5.8%
Methoxychlor	.1 ug/L	92.5%	6.1%
Deca-PCB	.1 ug/L	77.3%	4.1%

Conclusions

The results of five water samples demonstrate the ability of the FMS TurboTrace SPE system to deliver accurate results with excellent reproducibility. The automated SPE Direct to Vial Concentration method described is superior to traditional, time-consuming, inconsistent and expensive liquid/liquid extractions. The addition of the FMS SuperVap system equipped with direct-to-vial tubes enables the transfer of samples directly from sample bottles to GC vials in a single extraction process without handling the extract allows the extract to go directly to the GC for analysis.

Figure 2. Shows the chromatogram of a 1L water sample run on uECD detector



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