Application Note

Automated Solid Phase Extraction (SPE) of Organochlorine Pesticides in Water

Introduction

Organochlorine pesticides are man-made organic chemicals with a history of wide spread use in both the United States and globally. Since they tend to persist in the environment, they have found there 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 organo-chlorines are also suspected endocrine disruptors.

In response to growing health concerns, the United States has banned several of these compounds such as DDT, dieldrin and chlordane. Others are still in use including lindane, endosulfan and methoxychlor.

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 described below allows for rapid, reproducible extractions using a minimal volume of solvent that produce consistent results.

Instrumentation

- FMS, Inc. PowerPrep™ SPE (Solid Phase Extraction) System
- FMS, Inc. SuperVap™ Concentrator
- FMS, direct-to-vial concentrator tubes
- 1 gram C_{18} cartridges
- Agilent 7890A GC with uECD

PowerPrep SPE

1. The C_{18} Cartridge is conditioned with 10 mL methanol
2. The C_{18} Cartridge is conditioned with 10mL DI H_2O
3. The sample is loaded onto the C_{18} Cartridge via vacuum
4. The sample bottle auto rinsed loaded on to the C_{18} cartridge
5. The C_{18} cartridge is dried with nitrogen
6. Elute with methylene chloride

SuperVap Concentrator

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

Procedure: Sample Prep and Extraction

1. Five, 1 liter water samples spiked with 1 mL EPA 8081 surrogate spiking solution (2 analytes)
2. Samples were spiked with EPA 8081 pesticide spiking solution (20 analytes)
3. Samples allowed to equilibrate for 15 minutes
4. Five samples were loaded onto to corresponding sample ports on FMS PowerPrep SPE System.
5. The program is initiated to run each sample sequentially.
6. The sample is extracted and automatically transferred to the FMS SuperVap Concentrator with direct-to-vial vessels.
7. The Extracts are concentrated using the SuperVap system to 1 mL, exchanged to Hexane (15 mLs) and re-evaporated to 1 mL.
8. The Extract is removed from the SuperVap system and transferred to Agilent GC for analysis.

Figure 1: PowerPrep SPE and SuperVap Concentrator systems.

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Results

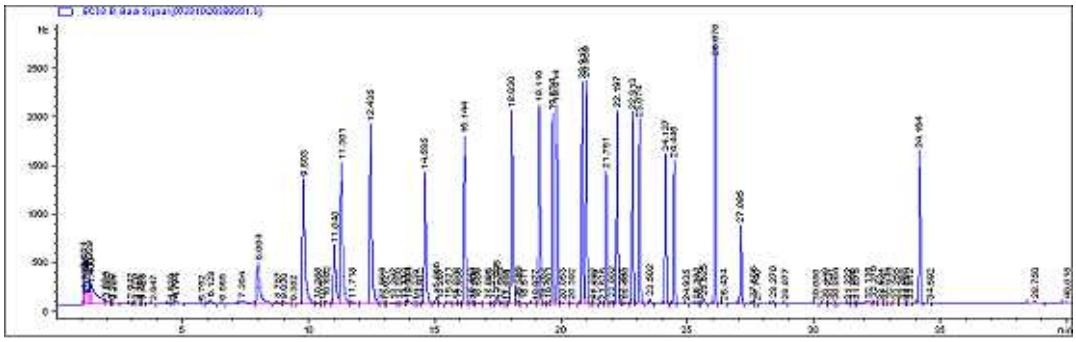
Table 1: Mean recovery and Standard deviation for five replicates

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	.1 ug/L	82.5%	5.0%
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 PowerPrep 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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