

Analysis of Persistent Organic Pollutants in Drinking Water with Semi-Automated Solid Phase Extraction

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Introduction

Continued interest in Persistent Organic Pollutants (POPs), such as organochlorine pesticides (EPA method 508) and polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs), and biphenyls (PCBs) (EPA methods 1613/1668C), has led to a variety of automated systems for the solid phase extraction of various kinds of water samples, including drinking water.

To meet demands for a lower cost method that requires less financial investment than the automated systems, we developed a simple semi-automated system which is fast and inexpensive while yielding high quality data.

Instrumentation

- FMS EZSpe® System
- FMS SuperVap®
- Vacuum pump
- 7010B Agilent TripleQuad
- 7890A Agilent FID-ECD

Consumables

- FMS, Inc. 1 g C18 cartridge
- Ultra pure DI water
- Fisher 6 N Hydrochloric Acid
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Dichloromethane
- Fisher Sodium Sulfate
- Methods 508, 1613 and 1668 C spiking and recovery standards

Material and Methods

SPE Procedure

Procedure

- 12 samples (1L water each) are prepared and acidified with 1 mL HCl till pH ~ 2
- Spike with relevant standards
- Put sample bottles in place and fill dichloromethane rinse bottles with 25 mL solvent
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 5 mL each of dichloromethane, methanol and water
- Samples are loaded across cartridges under vacuum
- Cartridges are dried with nitrogen for 10 min

Stage 2:

- Sample bottles are automatically rinsed from the rinse bottles with 25 mL dichloromethane
- Dichloromethane from sample bottles is loaded across the C18 cartridge and sodium sulfate cartridge and the eluent is collected for analysis into Direct to GC Vial Collection Vessels

FMS SuperVap®

- Pre-heat temp: 50 °C
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 50 °C under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Add recovery standards and reduce to 10 uL at ambient temperature for analysis (1613 and 1668C)
- DCM in samples for ECD exchanged to hexane

Results

Table 1 with ¹³C labeled recoveries (average, %) for PCDD/Fs and PCBs

2378-TCDF	94	PCB 28	66
2378-TCDD	106	PCB 52	77
12378-PeCDF	92	PCB 77	89
23478-PeCDF	78	PCB 81	81
12378-PeCDD	87	PCB 101	77
123478-HxCDF	96	PCB 105	84
123678-HxCDF	74	PCB 114	79
234678-HxCDF	86	PCB 118	75
123789-HxCDF	93	PCB 123	80
123478-HxCDD	106	PCB 126	85
123678-HxCDD	76	PCB 138	87
1234678-HpCDF	73	PCB 153	85
1234789-HpCDF	78	PCB 156	88
1234678-HpCDD	94	PCB 157	84
OCDD	88	PCB 167	90
		PCB 169	80
		PCB 170	91
		PCB 180	92
		PCB 189	83

Compound name	Average (%)	RSD (%)
TCMX	70.0	5.1
Alpha - BHC	81.6	2.0
Beta - BHC	93.9	4.7
Gamma - BHC (Lindane)	83.1	4.7
Delta - BHC	98.9	5.9
Heptachlor	82.5	5.0
Aldrin	80.0	4.5
Heptachlor Epoxide	89.8	5.2
Endosulfan I	87.8	4.7
4, 4 - DDE	84.0	4.7
Dieldrin	85.9	4.7
Endrin	70.6	5.3
Endosulfan II	90.5	4.8
Endrin Aldehyde	119.1	5.9
4, 4 - DDD	81.7	5.1
Endosulfan sulfate	95.0	5.1
4,4 - DDT	96.2	6.4
Endrin Ketone	110.9	5.8
Methoxyclor	92.5	6.1
PCB-209	77.3	4.1

Table 2 with 508 OCP recoveries (1 ug/L spike)

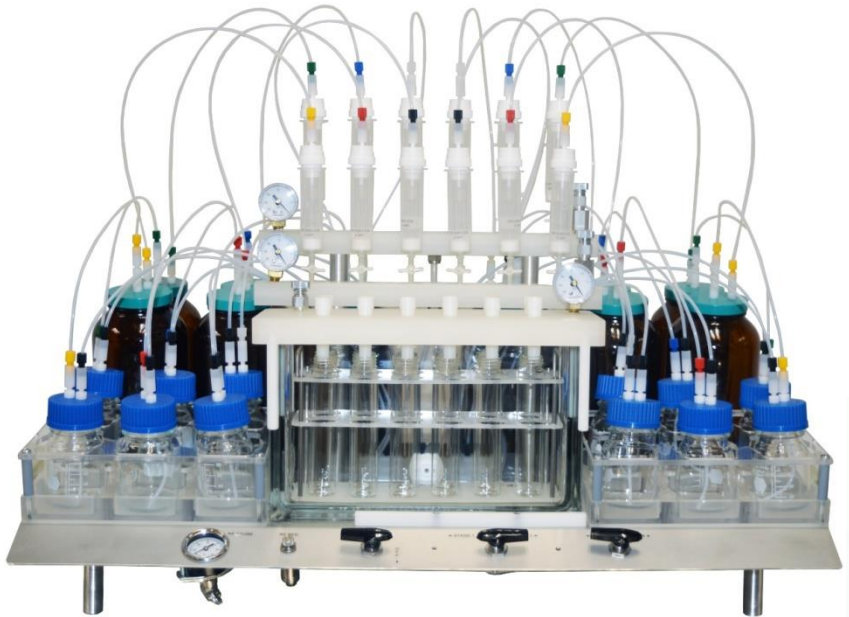
Discussion and Conclusions

The semi-automated FMS EZSpe system produces reliable, reproducible results for organochlorine pesticides, dioxins and pcbs in drinking water. The system is less expensive than fully automated SPE equipment and produces fast and reliable data.



For additional information please contact:

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FMS EZSpe system

Sample
Extraction

Sample
Clean Up

Sample
Concentration