

Analysis of Organochlorine Pesticides in Waste Water Using EPA Method 608.3 with Semi-Automated Solid Phase Extraction (EZSpe®)

Introduction

Organochlorine Pesticides are among the more notorious organic pollutants. Gaining wide spread attention into the latter 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 608.3.

To meet demands for a low-cost method that requires less financial investment than automated systems, FMS developed a simple semi - automated system which is fast, inexpensive and yields high quality data.

Instrumentation

- FMS EZSpe® System
- FMS SuperVap®
- Vacuum pump
- Agilent 7890A GC with uECD

Consumables

- FMS, Inc. 1 g 25 mL C-18 cartridge
- FMS Inc Florisil SPE cartridges
- FMS Inc Hydromatrix® SPE cartridges
- Fisher 6 N Hydrochloric Acid
- Fisher Pesticide Grade Acetone
- Fisher Pesticide Grade Hexane
- Restek 608.3 spiking standards

Procedure

- 6 samples (1L water each) are prepared and acidified with 1 mL HCl till pH ~ 2
- Spike with 608.3 standards
- Put sample bottles in place and fill rinse bottles with 40 mL 10% acetone/hexane
- C-18 cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- C-18 cartridges are conditioned with 10 mL acetone (2 min soak), and 20 mL water
- Samples are loaded across C-18 cartridges under vacuum (~ 12 inch Hg)
- Cartridges are dried under vacuum for 10 min
- Remove C-18 cartridges
- Put Hydromatrix cartridges on top of Florisil cartridges and condition both with 20 mL 10% acetone/hexane

Stage 2:

- Put Hydromatrix/Florisil cartridges assembly on top of C-18 cartridges and put on top of Stage 2 manifold
- Sample bottles are automatically rinsed from the rinse bottles with 40 mL 10% acetone/hexane
 - Elute sample bottles rinses across the cartridges and collect into Direct to GC Vial Collection Vessels for analysis

FMS SuperVap®

- Pre-heat temp: 55 °C
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 55 °C under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

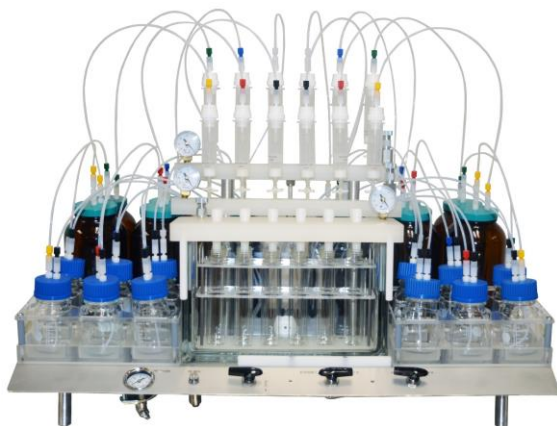


Table 1 with recoveries for 608.3 analytes

Analyte	Mean Rec	RSD
α-BHC	106%	2%
γ-BHC (Lindane)	105%	2%
β-BHC	98%	2%
δ-BHC	110%	2%
Heptachlor	100%	9%
Aldrin	76%	6%
Heptachlor Epoxide	101%	1%
γ-Chlordane	92%	3%
Endosulfan I	94%	2%
α-Chlordane	99%	2%
4,4'-DDE	93%	2%
Dieldrin	102%	2%
Endrin	110%	2%
4,4'-DDD	102%	3%
Endosulfan II	101%	2%
Endrin Aldehyde	84%	5%
4,4'-DDT	103%	4%
Endosulfan Sulfate	103%	3%
Methoxychlor	100%	6%
Endrin Ketone	99%	4%
Toxaphenes	87%	10%
Aroclor 1242	74%	5%

Conclusions

The results of six water samples demonstrate the ability of the FMS EZSpe® system to deliver accurate results with excellent reproducibility. All analytes were within the method's 60-140% acceptance window. The semi-automated EZSpe® is superior to traditional, time-consuming, inconsistent and expensive liquid/liquid extractions.



FMS EZSpe® System

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