

Dioxin in Drinking Water by One-Step Solid Phase Extraction



Introduction

Dioxins are a group of polyhalogenated compounds which occur as by-products in the manufacture of organochlorides, in the incineration of chlorine-containing substances such as PVC (polyvinyl chloride), in the bleaching of paper, and from natural sources such as volcanoes and forest fires. Aside from being known carcinogens, dioxins and dioxin like compounds have been linked to developmental abnormalities, nervous system, pathology, diabetes, and thyroid disorders as well as damage to the immune system. For these reasons, it is becoming increasingly important to determine dioxin free drinking water and food sources. This procedure demonstrates the use of the FMS SPE system in water samples.

Instrumentation and Consumables

- FMS, Inc. PowerPrep™ SPE system (Solid Phase Extraction)
- FMS, Inc. SuperVap™ Concentrator system
- Waters Oasis® 1 gram HLB Cartridge
- Thermo Fisher Scientific DFS HRGC/MS

Method Summary

PowerPrep SPE

1. Condition Cartridge with MeOH
2. Condition Cartridge with HPLC grade H₂O
3. Load 1 liter of water sample
4. Rinse container and load rinse volume
5. Dry Cartridge
6. Elute sample from Cartridge (DCM)

SuperVap Concentrator

1. Pre-heat temp: 70 °C
2. Pre-heat time: 15 minutes
3. Heat in Sensor mode: 70 °C
4. Nitrogen Pressure: 15 PSI

Procedure

Four, 1 liter water samples were spiked with 100 µL of Cambridge Isotope EDF-4144B Dioxin/Difuran spiking solution. Samples were then loaded onto the FMS SPE system using a vacuum pump and passed across a Waters Oasis HLB cartridge. After loading, samples were then dried using a stream of nitrogen until no residual water was present, and the cartridges were subsequently eluted using methylene chloride. Extracts were eluted directly into the FMS SuperVap Concentrator, spiked with 2 µL dodecane (keeper) and blown to dryness in direct-to-vial tubes. Samples were then re-constituted to 20 µL nonane and injected onto a high-resolution GC/MS system for quantitative analysis.



Figure 1: PowerPrep SPE and SuperVap Concentrator systems.



Results

Compound	Amount Spiked ng/L	Mean Recovered ng/L	Percent
2,3,7,8-TCDD (13C12,99%)	0.250	0.1702	68.09333
1,2,3,7,8-PeCDD (13C12,99%)	0.250	0.2474	98.95333
1,2,3,4,7,8-HxCDD (13C12,99%)	0.600	0.6294	104.9067
1,2,3,6,7,8-HxCDD (13C12,99%)	0.600	0.7649	127.49
1,2,3,7,8,9-HxCDD (13C12,99%)	0.600	0.6103	101.7233
1,2,3,4,6,7,8- HpCDD (13C12,99%)	0.600	0.4244	70.72667
1,2,3,4,6,7,9- HpCDD (13C12,99%)	0.600	0.4244	70.73
OCDD (13C12,99%)	1.250	0.8123	64.98667
2,3,7,8-TCDF (13C12,99%)	0.250	0.1897	75.89333
1,2,3,7,8-PeCDF (13C12,99%)	0.250	0.2479	99.16333
2,3,4,7,8-PeCDF (13C12,99%)	0.250	0.2136	85.44333
1,2,3,4,7,8-HxCDF (13C12,99%)	0.625	0.6456	103.29
1,2,3,6,7,8-HxCDF (13C12,99%)	0.625	0.6226	99.61333
1,2,3,7,8,9-HxCDF (13C12,99%)	0.625	0.6243	99.88
2,3,4,6,7,8-HxCDF (13C12,99%)	0.625	0.6641	106.2533
1,2,3,4,6,7,8- HpCDF (13C12,99%)	0.625	0.5961	95.38333
1,2,3,4,6,7,9- HpCDF (13C12,99%)	0.625	0.5961	95.37333
OCDF (13C12,99%)	1.250	0.7940	63.52333
3,3',4,4'-TCB (13C12, 99%)	0.250	0.2318	92.72667
3,4,4',5'-TCB (13C12, 99%)	0.250	0.2250	89.99
3,3',4,4',5'-PeCB (13C12, 99%)	0.360	0.5509	153.02
3,3',4,4',5,5'-HxCB (13C12, 99%)	0.480	0.8006	166.7933

Conclusions

The FMS PowerPrep SPE system produces reliable, reproducible results for low level dioxin and dioxin like compounds in drinking water. The use of direct-to-vial concentration tubes eliminates the need for sample transfer of very low volume extracts and the risk of extract loss and contamination.

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