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 polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs), and biphenyls (PCBs), 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 method that requires less financial investment than the automated systems, we developed a simple semi - automated system which is fast and inexpensive and yielding high quality data.

Instrumentation
■ FMS EZSpe®System
■ FMS SuperVap®
■ Vacuum pump
■Thermo Trace 1310 GC coupled with Thermo DFS High Resolution Magnetic Sector Mass Spec

Consumables
■ FMS, Inc. 1 g C-18 cartridge
■ Ultra pure DI water
■ Fisher 6 N Hydrochloric Acid
■ Fisher Pesticide Grade Methanol
■ Fisher Pesticide Grade Dichloromethane
■ Fisher Sodium Sulfate
■ Method 1613 ^{13}C PCDD/Fs Isotope Dilution and Recovery Standards
■ Method 1668 ^{13}C PCBs Isotope Dilution and Recovery Standards

Material and Methods
SPE Procedure

■ 12 samples (1L water each) are prepared and acidified with 1 mL HCl till pH ~ 2
■ Add 5-10 mL methanol and spike with ^{13}C labeled 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 dichloromethane, methanol and water
■ Samples are loaded across cartridges under vacuum
■ Cartridges are dried with nitrogen for 10 min
■ Sample bottles are automatically rinsed from the rinse bottles with 25 mL dichloromethane
Stage 2:
■ 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 ºC
■ Pre-heat time: 15 minutes
■ Heat in Sensor mode at 50 ºC 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

Results
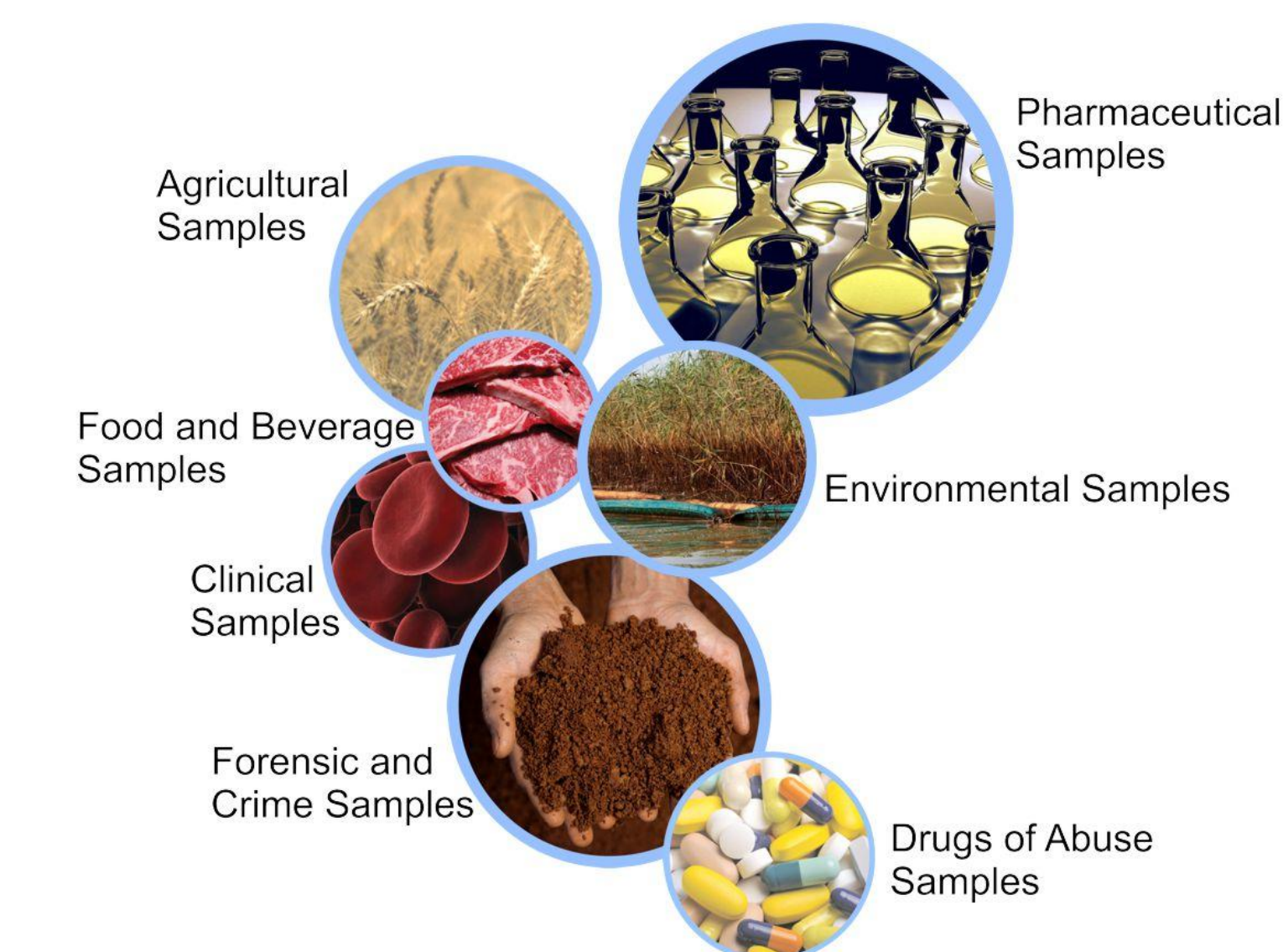
Table 1 with ^{13}C-labeled recoveries for PCDD/Fs and PCBs

	Average (%)		Average (%)
2378-TCDF	91.8	PCB 28	68.8
2378-TCDD	109.8	PCB 52	73.8
12378-PeCDF	89	PCB 77	83.6
23478-PeCDF	81.4	PCB 81	86
12378-PeCDD	89	PCB 101	80
123478-HxCDF	97.2	PCB 105	81
123678-HxCDF	77.6	PCB 114	78
234678-HxCDF	82.6	PCB 118	77.8
123789-HxCDF	90.4	PCB 123	80.2
123478-HxCDD	104.2	PCB 126	84
123678-HxCDD	75.6	PCB 138	85.6
1234678-HpCDF	74	PCB 153	86
1234789-HpCDF	77.8	PCB 156	86.6
1234678-HpCDD	82.6	PCB 157	83.6
OCDD	86.2	PCB 167	86.6
		PCB 169	83.2
		PCB 170	90.8
		PCB 180	88.8
		PCB 189	86.6
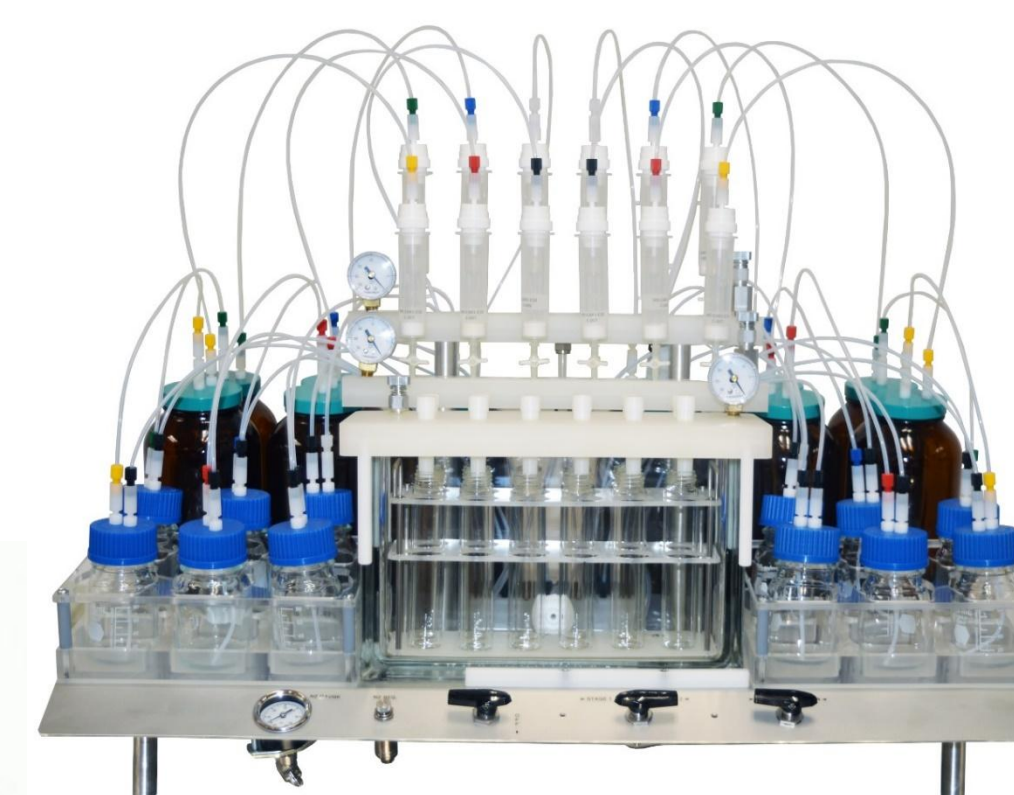
FMS EZSpe system

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

Agricultural Samples
Pharmaceutical Samples
Food and Beverage Samples
Environmental Samples
Clinical Samples
Forensic and Crime Samples
Drugs of Abuse Samples

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Sample Extraction

Sample Clean Up

Sample Concentration