

# Analysis of PCDD/Fs and PCBs in Drinking Water Using Semi-Automated Solid Phase Extraction (EZSpe®) with EPA Methods 1613 and 1668C

## 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 lower cost method that requires less financial investment than the automated systems, we developed a simple semi-automated system which is faster and cheaper while yielding high quality data. Relevant EPA methods are 1613 and 1668C.

## 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
- CIL Method 1613 <sup>13</sup>C PCDD/F Stock Solution
- CIL <sup>13</sup>C PCDD/F Recovery Standard

- CIL <sup>13</sup>C PCB Internal Isotope Dilution Standard who-12 PCB, PCB-170 and -180, and indicator PCBs
- CIL <sup>13</sup>C PCB Recovery Standard

## Procedure

- 6 samples (1L water each) are prepared and acidified with 1 mL HCl till pH ~ 2
- Add 5-10 mL methanol and spike with <sup>13</sup>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: 55 °C
- Pre-heat time: 15 min
- Heat in Sensor mode at 55 °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



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

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

### Conclusions

The semi-automated FMS EZSpe system produces reliable, reproducible results for dioxins and pcbs in drinking water. The system is a lower cost alternative to fully automated SPE equipment and produces fast and reliable data.



FMS EZSpe® System

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