

# Analysis of PCDD/Fs and PCBs in Drinking Water Using Semi-Automated Solid Phase Extraction (EZSpe™)

## 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.

## 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®

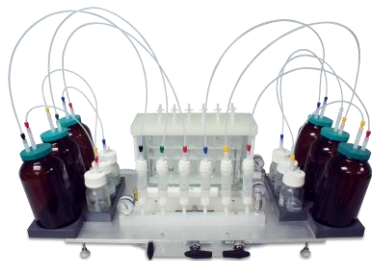
- Pre-heat temp: 50 °C
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 50 °C under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Add recovery standards and reduce to 10 uL at ambient temperature for analysis

Table 1 with <sup>13</sup>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 cheaper than fully automated SPE equipment and produces fast and reliable data.



FMS EZSpe system

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