

# Semi Automated Clean Up for Persistent Organic Pollutants in Fish - PCDD/Fs and PCBs Analysis for Sample Extracts in Toluene

## Introduction

The continued interest in Persistent Organic Pollutants (POPs), such as polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs), biphenyls (PCBs) and PBDEs, has led to a variety of automated systems for the cleanup of complex sample matrices. This has resulted in development of our fully automated "Power Prep" sample cleanup instead of manual preparative open column chromatography.

To meet demands for a lower cost method that requires little financial investment, we combined the features of the "PowerPrep" system - accurate, fast, reliable with short turnaround times and low background using FMS pre-packaged columns - with a relatively simple semi automated approach. An important feature of the semi-automated technique described here is that the samples can be in toluene (the method described here is for fish extracts in 2-10 mL toluene).

This semi-automated method is ideal for laboratories that want high quality sample processing without much financial investment.

## Instrumentation

- FMS EZPrep123™ System
- Vacuum pump
- Thermo Trace 1310 GC with Thermo DFS Magnetic Sector high resolution MS

## Consumables

- FMS, Inc. High Capacity Acidic Silica column
- FMS, Inc. Basic Alumina column
- FMS, Inc. Carbon-Celite column
- Fisher Pesticide Grade Hexane
- Fisher Pesticide Grade Dichloromethane
- Fisher Pesticide Grade Toluene
- 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

### Stage 1:

- Assemble columns in order acidic silica - alumina (no carbon used here).
- Syringe vial at top is used for conditioning and sample loading.
- Columns are conditioned with 40 mLs of hexane. Hexane is pulled by vacuum pump across all columns (vacuum, waste).

### Stage 2:

- Samples are loaded across system in 2-10 mL toluene (Fraction 1, PCBs).
- Columns are eluted with 60 mL hexane (Fraction 1, PCBs)
- Silica columns are removed and alumina is eluted with 30 mL of 10% dichloromethane/hexane (Fraction 1, PCBs)

### Stage 1:

- Carbon columns are connected to the bottom of alumina columns and then both columns are eluted with 50 mL dichloromethane (vacuum, waste).

### Stage 2:

- Alumina columns are disconnected from carbon and discarded. Carbon columns are turned upside down and eluted in reverse with 60 mL toluene (Fraction 2, PCDD/Fs);
- Total run time is less than 45 min
- Number of parallel sample clean up channels is unlimited

## Additional Features

- Low re-use of tubing, syringes, parts and glass ware
- No electronics and mechanical parts to fail
- No service contract or maintenance to worry about
- Fast, 45 minutes run time
- No repetitive motions and minimal cleaning of reusable parts



	Average	STD	RSD	EPA Window
2378-TCDF 13C12 STD	93.4	8.8	9.5	24-169
2378-TCDD 13C12 STD	115.1	12.4	10.8	25-164
12378-PeCDF 13C12 STD	89.8	6.1	6.8	24-185
23478-PeCDF 13C12 STD	84.3	7.7	9.2	21-178
12378-PeCDD 13C12 STD	86.0	13.8	16.0	25-181
123478-HxCDF 13C12 STD	78.2	8.1	10.4	26-152
123678-HxCDF 13C12 STD	76.8	13.2	17.2	26-123
234678-HxCDF 13C12 STD	79.0	6.1	7.7	28-136
123789-HxCDF 13C12 STD	93.0	7.8	8.4	29-147
123478-HxCDD 13C12 STD	84.6	11.2	13.2	32-141
123678-HxCDD 13C12 STD	78.1	8.9	11.4	28-130
1234678-HpCDF 13C12 STD	74.0	9.9	13.3	28-143
1234789-HpCDF 13C12 STD	84.3	7.1	8.5	26-138
1234678-HpCDD 13C12 STD	80.9	6.6	8.1	23-140
OCDD 13C12 STD	74.9	4.5	6.0	17-157

Table 1 with <sup>13</sup>C-labeled recoveries in percent for PCDD/Fs in 10 g fish (n=12).



	Average	STD	RSD	EPA Window
PCB_28	72.3	12.7	17.5	
PCB_52	74.5	14.7	19.7	
PCB_101	76.8	7.0	9.1	
PCB_81	78.1	3.6	4.6	10-145
PCB_77	66.4	10.8	16.3	10-145
PCB_123	65.8	7.5	11.3	10-145
PCB_118	70.4	6.0	8.5	10-145
PCB_114	52.0	6.5	12.4	10-145
PCB_105	78.8	4.3	5.4	10-145
PCB_126	64.9	9.4	14.5	10-145
PCB_153	86.0	6.3	7.3	
PCB_138	89.0	6.2	7.0	
PCB_167	83.8	6.7	8.0	10-145
PCB_156	77.3	7.4	9.6	10-145
PCB_157	67.6	5.7	8.4	10-145
PCB_169	65.6	10.4	15.8	10-145
PCB_180	74.0	8.7	11.7	
PCB_170	61.5	7.2	11.7	
PCB_189	35.1	5.0	14.2	10-145
PCB_209	82.1	10.3	12.6	10-145

Table 2 with <sup>13</sup>C-labeled recoveries in percent for PCBs in 10 g fish (n=12).

### Conclusions

Excellent recoveries are seen with the new semi automated method using the FMS EZPrep123 System, as can be seen in Tables 1 and 2. Because the system is a closed system, mostly composed of disposable parts, the risk of cross-contamination is very low. Note that fish extracts in 2-10 mL toluene can be processed as such without any need for solvent exchange. The system can be set up as a low-cost alternative to the fully automated clean up equipment. Processing times are much shorter than other manual procedures. The certified prepackaged columns and simple, versatile system guarantee same morning or afternoon POPs analysis.

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