Application Note

EPA 1613 and 1668C Semi-Automated Cleanup of Sample Extracts for Persistent Organic Pollutants Analysis in Soil - Complete Separation of PCDD/Fs and PCBs

Introduction

The 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 cleanup of complex sample matrices. This has resulted in development of a fully automated “Power Prep” sample cleanup instead of manual preparative column chromatography.

To meet demands for an inexpensive 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 PCBs are collected in one fraction and PCDD/Fs in another fraction.

This semi-automated method is ideal for environmental laboratories that want high quality sample processing without much financial investment. This application follows EPA method 1613 and 1668C.
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Instrumentation

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

Consumables

- FMS, Inc. Classical Plus Acidic Silica column
- FMS, Inc. Neutral-Basic Silica column(optional)
- FMS, Inc. Basic Alumina column 4 g
- FMS, Inc. Carbon-Celite 034-034 column
- Fisher Pesticide Grade Hexane
- Fisher Pesticide Grade Dichloromethane
- Fisher Pesticide Grade Toluene
- 1613 and 1668C recovery and spiking standards

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 20 mLs of hexane. Hexane is pulled by vacuum pump across silica-alumina (forward, vacuum, waste).
- Samples are loaded across system in hexane (forward, vacuum, waste).
- Columns are eluted with 100 mL hexane (forward, vacuum, waste)

Stage 2:

- Silica columns are removed and alumina is eluted with 30 mL of 10% dichloromethane/hexane (forward, Fraction 1, PCBs; all PCBs are collected in one fraction)

Stage 1:

- Carbon columns are connected to the bottom of alumina columns and then both columns are eluted with 50 mL dichloromethane (forward, 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, collect all PCDD/Fs in this fraction)
- 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

SuperVap Concentration

- Pre-heat temperature: 55 ^{o}C
- Pre-heat time: 15 min
- Heat in Sensor mode: 55 ^{o}C
- Nitrogen Pressure: 7-10 psi
- Collect in Direct-to-Vial GC vials and reduce to 1 mL

FMS
Fluid Management Systems

Vial Evaporator
■ Reduce sample to 10 uL final volume under 1-1.5 psi nitrogen at 25 ^{o}C

Analysis
■ High Resolution GC/MS

		Average	STDEV	RSD	EPA Window
224'-Tr-PCB 13C STD	PCB_28	86.3	8.9	10.3	
22'55'-Te-PCB 13C STD	PCB_52	62.4	8.5	13.6	
22'455'-Pe-PCB 13C STD	PCB_101	69.2	4.8	7.0	
344'5-Te-PCB 13C STD	PCB_81	71.4	11.7	16.4	10-145
33'44'-Te-PCB 13C STD	PCB_77	67.3	9.6	14.3	10-145
2'344'5-Pe-PCB 13C STD	PCB_123	77.2	4.6	5.9	10-145
23'44'5-Pe-PCB 13C STD	PCB_118	72.4	7.1	9.8	10-145
2344'5-Pe-PCB 13C STD	PCB_114	73.9	5.3	7.2	10-145
233'44'-Pe-PCB 13C STD	PCB_105	76.0	6.2	8.2	10-145
33'44'5-Pe-PCB 13C STD	PCB_126	82.2	6.0	7.2	10-145
22'44'55'-Hx-PCB 13C STD	PCB_153	71.9	16.3	22.7	
22'344'5'-Hx-PCB 13C STD	PCB_138	89.0	23.0	25.8	
23'44'55'-Hx-PCB 13C STD	PCB_167	84.0	13.6	16.2	10-145
233'44'5-Hx-PCB 13C STD	PCB_156	109.9	18.6	16.9	10-145
233'44'5'-Hx-PCB 13C STD	PCB_157	81.3	12.7	15.6	10-145
33'44'55'-Hx-PCB 13C STD	PCB_169	83.4	6.0	7.2	10-145
22'344'55'-Hp-PCB 13C STD	PCB_180	82.9	13.9	16.7	
22'33'44'5-Hp-PCB 13C STD	PCB_170	80.8	13.4	16.6	
233'44'55'-Hp-PCB 13C STD	PCB_189	84.8	16.8	19.8	10-145

Table 1 with ^{13}C-labeled recoveries in percent for PCBs in 10 g soil

	Average	STDEV	RSD	EPA Window
2378-TCDF 13C12 STD	94.1	5.3	5.7	24-169
2378-TCDD 13C12 STD	115.7	6.1	5.3	25-164
12378-PeCDF 13C12 STD	94.5	5.4	5.8	24-185
23478-PeCDF 13C12 STD	89.7	6.8	7.6	21-178
12378-PeCDD 13C12 STD	97.0	5.1	5.3	25-181
123478-HxCDF 13C12 STD	76.3	8.0	10.5	26-152
123678-HxCDF 13C12 STD	68.5	6.6	9.6	26-123
234678-HxCDF 13C12 STD	71.4	5.3	7.4	28-136
123789-HxCDF 13C12 STD	55.9	9.5	16.9	29-147
123478-HxCDD 13C12 STD	78.5	6.7	8.5	32-141
123678-HxCDD 13C12 STD	72.1	8.2	11.4	28-130
1234678-HpCDF 13C12 STD	66.6	8.3	12.5	28-143
1234789-HpCDF 13C12 STD	87.6	6.2	7.1	26-138
1234678-HpCDD 13C12 STD	78.8	9.9	12.6	23-140
OCDD 13C12 STD	73.5	7.4	10.1	17-157

Table 2 with ^{13}C-labeled recoveries in percent for PCDD/Fs in 10 g soil

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 closed, mostly composed of disposable parts, the risk of cross-contamination is very low. Note that a complete separation of PCBs and PCDD/Fs is achieved. The system can be set up as an inexpensive alternative to the fully automated clean up equipment. Processing times are much shorter than other manual procedures. The certified pre-packaged columns and simple, versatile system guarantee same morning or afternoon POPs analysis.

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