Application Note

EPA 1613 and 1668C Semi-Automated Cleanup for Persistent Organic Pollutants Analysis in Oil Samples - Complete Separation of PCDD/Fs and PCBs Using Florisil in Cleanup

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. The work described here includes complete separation of PCDD/Fs and PCBs in oil samples using high capacity acidified silica, Florisil and alumina.

This method is ideal for laboratories that want high quality sample processing without much financial investment and follows US EPA methods 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. High (SNHC-ACD-029) or Extra-High Capacity Acidic Silica (SNEH-ACD-050) column
- FMS, Inc Florisil column (SNFL-FLL-007)
- FMS, Inc. Basic Alumina column (SNAL-BAS-006)
- Fisher Pesticide Grade Hexane
- Fisher Pesticide Grade Dichloromethane
- 1613 and 1668C recovery and spiking standards

Procedure

Stage 1:

- Assemble columns in order acidic silica-Florisil-alumina.
- Syringe vial at top is used for conditioning and sample loading.
- Columns are conditioned with 40-60 mL of hexane. Hexane is pulled by vacuum pump across all columns (vacuum, waste).
- Samples are loaded across system (vacuum, waste)
- Columns are eluted with 160 or 180 mL hexane (vacuum, waste)
- Silica columns are removed

Stage 2:

- Florisil and alumina columns (together) are eluted with 50 mL 10% dichloromethane/hexane (Fraction # 1, PCBs).
- Florisil and alumina columns are both reversed but kept attached to each other. Alumina stays at bottom. Elute with 50 mL dichloromethane (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

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

Vial Evaporator

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

Analysis

- High Resolution GC/MS

FMS
Fluid Management Systems

	2.5 g fish oil	2.5 g olive oil	EPA Window
2378-TCDF 13C12 STD	97	94	24-169
2378-TCDD 13C12 STD	91	88	25-164
12378-PeCDF 13C12 STD	73	71	24-185
23478-PeCDF 13C12 STD	76	74	21-178
12378-PeCDD 13C12 STD	73	71	25-181
123478-HxCDF 13C12 STD	103	97	26-152
123678-HxCDF 13C12 STD	102	96	26-123
234678-HxCDF 13C12 STD	111	103	28-136
123789-HxCDF 13C12 STD	99	93	29-147
123478-HxCDD 13C12 STD	100	95	32-141
123678-HxCDD 13C12 STD	95	89	28-130
1234678-HpCDF 13C12 STD	97	91	28-143
1234789-HpCDF 13C12 STD	86	81	26-138
1234678-HpCDD 13C12 STD	86	82	23-140
OCDD 13C12 STD	74	69	17-157

Table 1 with ^{13}C-labeled recoveries in percent for PCDD/Fs in fish oil and olive oil.

	2.5 g fish oil	2.5 g olive oil	EPA Window
PCB_28	110	105	
PCB_52	119	124	
PCB_101	120	128	
PCB_81	109	114	10-145
PCB_77	104	123	10-145
PCB_123	127	125	10-145
PCB_118	129	132	10-145
PCB_114	129	137	10-145
PCB_105	127	135	10-145
PCB_126	119	126	10-145
PCB_153	137	134	
PCB_138	130	134	
PCB_167	131	137	10-145
PCB_156	133	133	10-145
PCB_157	131	136	10-145
PCB_169	96	117	10-145
PCB_180	127	132	
PCB_170	121	134	
PCB_189	119	134	10-145

Table 2 with ^{13}C-labeled recoveries in percent for PCDD/Fs in fish oil and olive oil.

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 complete separation of PCBs and PCDD/Fs is achieved using silica, Florisil and alumina. 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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