

Semi Automated Clean Up for Persistent Organic Pollutants Analysis in Oil Samples - Complete Separation of PCDD/Fs and PCBs Using Florisil in Clean Up

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

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 or Extra-High Capacity Acidic Silica column
- FMS, Inc Florisil column
- FMS, Inc. Basic Alumina column
- Fisher Pesticide Grade Hexane
- Fisher Pesticide Grade Dichloromethane

- CIL Method 1613 ¹³C PCDD/F Stock Solution
- CIL ¹³C PCDD/F Recovery Standard
- CIL ¹³C PCB Internal Isotope Dilution Standard who-12 PCB, PCB-170 and -180, and indicator PCBs
- CIL ¹³C PCB Recovery Standard

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 mLs of hexane. Hexane is pulled by vacuum pump across all columns (vacuum, waste).
- Samples are loaded across system (vacuum, waste)
- Columns are eluted with 180 or 220 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



	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 ¹³C-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 ¹³C-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 a closed system, 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 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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