

EPA 1613 Semi Automated Clean Up for Persistent Organic Pollutants in Fish Oil - PCDD/Fs 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 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 the samples can be in toluene (the method described here is for fish oil extracts in 2 mL toluene).

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.

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
- 1613 recovery and spiking standards

Procedure

Stage 1:

- Assemble columns in order acidic silica - alumina - carbon.
- Syringe vial at top is used for conditioning and sample loading.
- Columns are conditioned with 60 mLs of hexane. Hexane is pulled by vacuum pump across all columns (vacuum, waste).
- Samples are loaded across system in 2-mL toluene (vacuum, waste).
- Columns are eluted with 60 mL hexane (vacuum, waste)
- Silica columns are removed and alumina - carbon columns are eluted with 50 mL dichloromethane (vacuum, waste)

Stage 2:

- Carbon columns are turned upside down and eluted in reverse with 60 mL toluene (Fraction 1, 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 °C
- Pre-heat time: 15 min
- Heat in Sensor mode: 55 °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 °C

Analysis

- High Resolution GC/MS



	Average	STDEV	RSD	EPA Window
2378-TCDF 13C12 STD	89.2	8.1	9.1	24-169
2378-TCDD 13C12 STD	113.5	10.4	9.2	24-169
12378-PeCDF 13C12 STD	87.2	10.2	11.8	24-185
23478-PeCDF 13C12 STD	85.2	12.5	14.7	21-178
12378-PeCDD 13C12 STD	89.7	12.0	13.4	25-181
123478-HxCDF 13C12 STD	74.7	4.0	5.3	26-152
123678-HxCDF 13C12 STD	64.8	3.5	5.5	26-123
234678-HxCDF 13C12 STD	66.5	2.4	3.7	28-136
123789-HxCDF 13C12 STD	81.8	6.2	7.6	29-147
123478-HxCDD 13C12 STD	75.3	5.2	6.9	32-141
123678-HxCDD 13C12 STD	68.0	3.0	4.5	28-130
1234678-HpCDF 13C12 STD	62.2	4.0	6.5	28-143
1234789-HpCDF 13C12 STD	75.0	6.5	8.7	26-138
1234678-HpCDD 13C12 STD	68.5	4.7	6.9	23-140
OCDD 13C12 STD	60.7	5.9	9.6	17-157

Table 1 with ¹³C-labeled recoveries in percent for PCDD/Fs in 2 g fish oil (n=6).

Conclusions

Excellent recoveries are seen with the semi automated method using the FMS EZPrep123 System, as can be seen in Tables 1. Because the system is closed, mostly composed of disposable parts, the risk of cross-contamination is very low. Note that fish oil extracts in 2 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 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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