Workflows Optimized for High Throughput, Robust Persistent Organic Pollutants (POPs) Analysis in Environmental Matrices

Ruud Addink, Tom Hall
Fluid Management Systems, 900 Technology Park Drive Billerica, MA 01821
www.fms-inc.com

Introduction

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Persistent organic pollutants (POPs) such as polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs) and biphenyls (PCBs) have been a major environmental concern for several decades. Due to their low solubility in water and their resistance to breakdown, they tend to accumulate in river sediment. Analyses of sediment samples using US EPA methods 1613 (PCDD/Fs) and 1668 (PCBs) have been carried out around the world. Study of sediments often involves large amounts of samples, making fast processing (extraction, clean up, analysis) more important. This work describes the automated Pressurized Liquid Extraction (PLE) and semi-automated column chromatography cleanup of river sediment. Quick and easy processing results in samples being ready for same-day analysis.

Material and methods

PLE

- 1 g of sample mixed with 10 g inert Hydro-matrix® and spiked with surrogates
- Sample placed in extraction cell
- Capped with disposable Teflon end caps
- Heated with 50% Dichloromethane/50% Hexane for 20 min at 120 ºC and 1500 psi
- 20 min cool down
- Nitrogen flush to transfer analytes and extract to 250 mL collection tubes

SuperVap Concentration

- Pre-heat temperature: 45 ºC
- Pre-heat time: 15 min
- Heat in Sensor mode: 45 ºC
- Nitrogen Pressure: 8 psi
- Solvent exchange to hexane

Materials and methods

Procedure EZPrep

Stage 1:

- Assemble EZPrep with acid silica-alumina-carbon.
- Syringe vial at top is used for conditioning and sample loading.
- Condition all columns with 40 mL hexane (vacuum, waste).
- Load samples in hexane (vacuum, waste).
- Elute all columns with 100 mL hexane (vacuum, waste).
- Discard acid silica columns

Stage 2:

- Elute alumina-carbon columns with 50 mL dichloromethane to collect mono- and di-ortho PCBs (Fraction # 1).
- Discard alumina columns.
- Reverse carbon columns and elute with 50 mL toluene, collecting co-planary PCBs and PCDD/Fs (Fraction # 2).

SuperVap Concentration

- Collected fractions are reduced to 1 mL final volume at ~ 8 psi nitrogen flow at 50 ºC, followed by reduction to 10 uL.

Analysis

- Samples were analyzed on Agilent 7010B Triple Quad.

Additional Features EZPrep

- Total run time is about 35 min
- Re-use of tubing, syringes, parts and glass ware
- No electronics and mechanical parts to fail
- No service contract or maintenance to worry about
- No repetitive motions and minimal cleaning of reusable parts

Results

	Native measured	Reference Material	Recoveries
	pg/g	pg/g	%
2378-T4CDF	742	818	94%
2378-T4CDD	268	351	92%
12378-P5CDF	533	584	103%
23478-P5CDF	554	623	91%
12378-P5CDD	216	273	101%
123478-H6CDF	178	160	82%
123678-H6CDF	705	779	83%
234678-H6CDF	732	805	84%
123789-H6CDF	410	455	77%
123478-H6CDD	503	519	82%
123678-H6CDD	393	325	80%
123789-H6CDD	289	221	
1234678-H7CDF	859	805	81%
1234789-H7CDF	163	182	90%
1234678-H7CDD	453	455	82%
OCDF	456	455	
OCDD	1616	1420	88%

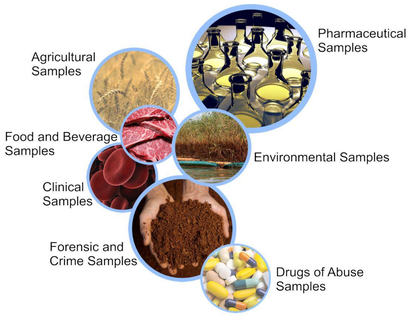
Table 1. 1 g sediment reference material analyzed. Values found vs reference material values for PCDD/Fs.

	PCB #	Native measured	Reference material	Recoveries
		pg/g	pg/g	%
33'44'-T4CB	77	35	37	102%
344'5-T4CB	81	312	279	95%
233'44'-P5CB	105	20	22	102%
2344'5-P5CB	114	185	182	100%
23'44'5-P5CB	118	80	98	99%
2'344'5-P5CB	123	180	195	102%
33'44'5-P5CB	126	301	316	103%
233'44'5-H6CB	156	316	279	110%
233'44'5'-H6CB	157	153	156	107%
23'44'55'-H6CB	167	150	145	88%
33'44'55'-H6CB	169	264	258	119%
233'44'55'-H7CB	189	31	32	118%

Table 2. 1 g sediment reference material analyzed. Values found vs reference material values for PCBs.

Discussion and Conclusions

As can be seen the sediment analysis showed excellent agreement between the values found with our automated extraction and semi-automated clean up and the acceptable reference values provided for this material. Furthermore, the method gave excellent recoveries. Extraction, clean up and analysis by properly trained personnel can be carried out in one day, resulting in low turnaround times for large (and small) sample batches.



For additional information please contact:

Ruud Addink
r.addink@fms-inc.com