

Low Solvent, DCM-Free, Semi-Automated, Negative Pressure Sample Clean Up for Persistent Organic Pollutants Analysis using Pre-Packaged Certified Columns

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 a fully automated "Power Prep" sample cleanup instead of manual preparative open column chromatography.

To meet demands for a 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 is that a minimum amount of solvent is used and no dichloromethane is used at all. This is important since many laboratories around the world are phasing out this solvent. This semi-automated method is ideal for those laboratories that want high quality sample processing without much financial investment.

Instrumentation

- The FMS EZPrep123[®] System
- Vacuum pump
- Thermo Trace GC Ultra with Thermo Quantum XLS Triple Quad GC/MS

Consumables

- FMS, Inc. Acidic Silica column (various kits depending on lipid capacity)
- FMS, Inc. Basic Silica column (optional)
- FMS, Inc. Basic Alumina column
- FMS, Inc. Carbon-Celite column
- Fisher Pesticide Grade Hexane
- Fisher Pesticide Grade Toluene
- ¹³C PCDD/F and PCB labeled spiking and recovery standards

Procedure

Stage 1:

- Assemble columns in order acidic silica-basic silica-carbon-alumina.
- Syringe vial at top is used for conditioning and sample loading.
- Depending on what kit is used (Table 2), columns are conditioned with up to 60 mLs of hexane. Hexane is pulled by vacuum pump across all columns into waste.
- Samples are loaded across system in hexane (vacuum, waste)
- System is eluted with 80 mLs (mini kit) to 180 mLs (XL kit) of hexane (vacuum, waste)

Stage 2:

- Carbon and alumina columns are eluted with toluene for collection under vacuum
- Both columns are each eluted in reverse direction with 40 mL toluene and Fractions are collected
- Fraction 1 with PCDD/Fs and coplanary-PCBs from carbon and Fraction 2 with mono- and di-ortho PCBs from alumina
- Total run time is less than 45 min
- Number of parallel sample clean up channels is unlimited
- Low solvent volume of collected Fractions reduces time required for sample concentration

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



Table 1 with typical ¹³C-labeled recoveries for PCBs

	Soil	Feed	Egg yolk	Olive oil	Fish oil	Hexane
	5 g	10 g	18 g	2 g	2 g	
PCB 28	93	104	71	103	100	95
PCB 52	90	108	69	100	97	95
PCB 77	90	103	122	98	102	108
PCB 81	92	99	60	102	98	92
PCB 101	93	110	74	106	102	98
PCB 105	108	101	61	110	104	106
PCB 114	111	102	64	105	97	104
PCB 118	86	103	60	91	89	102
PCB 123	106	97	69	92	96	93
PCB 126	107	102	89	102	98	115
PCB 138	104	96	76	92	110	111
PCB 153	101	102	68	102	114	102
PCB 156	102	99	60	113	104	105
PCB 157	93	97	60	103	99	108
PCB 167	119	106	60	105	105	107
PCB 169	98	98	81	96	96	117
PCB 170	103	107		103	105	117
PCB 180	98	106	84	102	102	107
PCB 189	108	97	62	95	88	107

Table 2 Types of kits, lipid capacity and solvents and volumes used in sample clean for various EZPrep kits.

Column kits	Fat Removal Capacity	STAGE 1			STAGE 2	
		Hexane conditioning (mL)	Hexane sample volume (mL)	Hexane Elute Silica (mL)	PCBs Toluene Reverse Almina (mL)	Dioxins Toluene Reverse Carbon (mL)
		Mini kit	0.15 g	20	10	80
Classical kit	0.5 g	20	10	90	40	40
Classical Plus	1.0 g	20	10	100	40	40
High Capacity	2.5 g	40	30	160	40	40
Extra high Capacity	5.0 g	60	30	180	40	40



Table 3 with typical ¹³C-labeled recoveries for PCDD/Fs

	Soil	Feed	Egg yolk	Olive oil	Fish oil
	5 g	10 g	18 g	2 g	2 g
2378-TCDF	95	92	83	96	89
2378-TCDD	104	101	70	101	96
12378-PeCDF	86	92	85	97	78
23478-PeCDF	102	94	69	102	98
12378-PeCDD	85	93	75	60	71
123478-HxCDF	88	105	79	92	92
123678-HxCDF	103	109	80	102	94
234678-HxCDF	73	66	80	60	95
123789-HxCDF	107	92	92	95	89
123478-HxCDD	107	95	79	95	92
123789-HxCDD	82	84	87	81	86
1234678-HpCDF	76	82	82	83	87
1234789-HpCDF	91	84	93	84	81
1234678-HpCDD	76	80	87	82	79
OCDD	60	67	60	60	91

Conclusions

Excellent recoveries are seen with the new semi automated method using the FMS EZPrep123 System, as can be seen in Tables 1 and 3. Because the system is mostly composed of disposable parts, the risk of cross-contamination is very low. Note that no dichloromethane is used. The system can be set up at low cost and is an alternative to the more expensive fully automated clean up equipment. Its processing times are much shorter than manual procedures. The certified columns and simple, versatile system guarantee same morning or afternoon POPs analysis.

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