

A Low Solvent Extraction and Zero DCM Sample Clean Up for POPs analysis

Introduction

Persistent organic pollutants (POPs) such as polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs) and biphenyls (PCBs) continue to attract interest around the world due to strict regulations in force in many countries. Rapid extraction, sample clean up, and analysis are therefore needed for many laboratories processing samples for POPs. In addition to processing times, solvent use is an important consideration as the cost of sample clean up involving large amounts of solvents can be very high.

In this note we show a newly developed automated sample extraction and clean up system, which allows for rapid extraction of the sample (1 hour) and fast cleanup (35 minutes). Solvent consumption is limited to 120-200 mLs and no dichloromethane is used during the cleanup step. Our new fully automated system allows for sample turnaround times of only a few hours and same day analysis and results.

Instrumentation

- FMS, Inc. PLE®/Low Solvent (LS) EconoPrep
- FMS, Inc. SuperVap® 6 position 250 mL Concentrator
- FMS, Inc. SuperVap® 12 position 50 mL Concentrator
- FMS, Inc. SuperVap® Vial Concentrator
- FMS, Inc. 50 and 250 mL concentrator tubes (1 mL termination)
- Thermo 1310 Trace GC with high res magnetic sector DFS Thermo mass spec
- Thermo Trace GC Ultra with TSQ Quantum XLS TripleQuad mass spec

Consumables

- FMS, Inc. High Capacity Acid-Base-Neutral Silica column
- FMS, Inc. Mini Acid-Base-Neutral Silica Column
- FMS, Inc. Mini Basic Alumina column

- FMS, Inc. Carbon column
- Fisher Dichloromethane Pesticide Grade
- Fisher Hexane Pesticide Grade
- Fisher Toluene Pesticide Grade
- Cambridge Isotope Labs (CIL) EDF-8999 Method 1613 ¹³C PCDD/F Stock Solution
- CIL EDF-5999 ¹³C PCDD/F Recovery Standard
- CIL EC-4995 ¹³C PCB Internal Isotope Dilution Standard who-12 PCB and 170/180
- CIL EO-5275 ¹³C PCB Recovery Standard

Pressurized Liquid Extraction

- 1 - 2 g of sediment or feed mixed with 10 g inert Hydro-matrix®
- Sample placed in extraction cell
- Capped with disposable Teflon end caps
- Heated with 50% Dichloromethane/50% Hexane for 20 min at 120 °C and 1500 psi
- 20 min cool down
- Nitrogen and solvent flush to transfer analytes and extract to 250 mL collection tubes

SuperVap Concentration

- Pre-heat temperature: 45 °C
- Pre-heat time: 15 min
- Heat in Sensor mode: 45 °C
- Nitrogen Pressure: 6-8 psi
- Solvent exchange to hexane

EconoPrep Clean Up

- Install silica, carbon and alumina columns
- System uses order of columns: silica -carbon - alumina
- Uses only hexane and toluene for 5 step program
- Add surrogates here to measure recoveries across cleanup step



- Condition with hexane
- Load sample in hexane
- Elute silica column with hexane
- Elute carbon in reverse (upward) direction with toluene (collect PCDD/F and co-planary PCBs)
- Elute alumina in reverse direction with toluene (collect mono-ortho and di-ortho PCBs)

SuperVap step (above)

Vial Evaporator

- Reduce sample to 10 uL final volume under 1-1.5 psi nitrogen at 25 °C

Compound Name	Sediment 1g	Fish Oil 40 mg	1.5 g fatty acid	2g fatty acid	Hexane
2378-TCDF 13C12 STD	78	69	96	93	78
2378-TCDD 13C12 STD	92	96	95	92	93
12378-PeCDF 13C12 STD	80	91	110	97	82
23478-PeCDF 13C12 STD	81	92	110	103	80
12378-PeCDD 13C12 STD	91	105	111	104	90
123478-HxCDF 13C12 STD	79	93	100	92	84
123678-HxCDF 13C12 STD	78	96	102	90	85
234678-HxCDF 13C12 STD	83	87	107	96	86
123789-HxCDF 13C12 STD	88	99	93	90	83
123478-HxCDD 13C12 STD	84	86	101	97	88
123678-HxCDD 13C12 STD	73	83	91	93	81
1234678-HpCDF 13C12 STD	69	87	92	88	69
1234789-HpCDF 13C12 STD	82	80	84	92	79
1234678-HpCDD 13C12 STD	87	98	89	92	79
OCDD 13C12 STD	70	77	81	80	64
PCB-77	94	90	101	87	86
PCB-81	88	83	95	93	75
PCB-126	92	89	88	79	77
PCB-169	91	93	106	103	100

Table 1 - PCDD/F and co-planary PCBs ¹³C recoveries across cleanup - fish oil and fatty acid used as such, no extraction



	Sediment 1 g	Fish Oil 40mg	1.5 g fatty acid	2 g fatty acid	Hexane
PCB 105	98	112	90	88	95
PCB 114	108	109	87	89	97
PCB 118	92	110	88	90	89
PCB 123	112	115	82	86	98
PCB 156	100	95	98	94	98
PCB 157	98	90	87	92	91
PCB 167	92	89	87	87	86
PCB 170	99	94	90	98	100
PCB 180	96	85	88	89	88
PCB 189	106	78	94	100	101

Table 2 – mono- and di-ortho PCBs ¹³C recoveries across cleanup - fish oil and fatty acid used as such, no extraction

Conclusions

The integrated PLE/Low Solvent Zero DCM EconoPrep generates clean extracts in less than 2 hours and generates consistent, reproducible recoveries for a number of matrices. It is a green option that cuts electrical costs. The system uses only a total volume of 120-200 mL of hexane and toluene it uses zero dichloromethane for the cleanup step.

For more information contact FMS:
 FMS, Inc.
 580 Pleasant Street
 Watertown, MA 02472
 Phone: (617) 393-2396
 Fax: (617) 393-0194
 Email: onlineinfo@fms-inc.com
 Web site: www.fms-inc.com