

POPs Extraction and Clean Up Using Green Low Solvent Total EconoPrep

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 app note we show a newly developed automated sample extraction and clean up system, which allows for rapid extraction of the sample (time approximately one hour) and fast clean up (2 hours). Solvent consumption is limited to 150-200 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-Celite 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

PowerPrep Clean Up

- Install mini ABN (HC-ABN), carbon/celite and alumina columns
- System uses order of columns: silica - carbon - alumina
- Uses only hexane and toluene for 5 steps program
- Add surrogates here to measure recoveries across cleanup step



- Condition with 20 mL hexane
- Load sample in 10 mL hexane
- Elute silica column 60 mL (120 mL HC-ABN) hexane
- Elute carbon in reverse (upward) direction with 30 mL toluene (collect PCDD/F and coplanary PCBs)
- Elute alumina in reverse direction with 30 mL toluene (collect mono-ortho and di-ortho PCBs, not done here)

SuperVap step (above)

Vial Evaporator

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

Compound Name	Feed 2g	Fish Oil 40 mg	Fish Oil 2 g	Sediment 1g	Hexane
2378-TCDF	89	69	98	78	78
2378-TCDD	100	96	111	92	93
12378-PeCDF	91	91	79	80	82
23478-PeCDF	90	92	78	81	80
12378-PeCDD	100	105	99	91	90
123478-HxCDF	95	93	92	79	84
123678-HxCDF	81	96	84	78	85
234678-HxCDF	91	87	90	83	86
123789-HxCDF	93	99	111	88	83
123478-HxCDD	92	86	90	84	88
123678-HxCDD	72	83	76	73	81
1234678-HpCDF	79	87	76	69	69
1234789-HpCDF	71	80	94	82	79
1234678-HpCDD	95	98	84	87	79
OCDD	77	77	74	70	64

Table 1 - PCDD/F ¹³C recoveries across cleanup
- fish oil used as such, no extraction



Compound Name	Fish Oil 40 mg	Fish Oil 2 g	Sediment 1g	Hexane
PCB-77	90	92	94	86
PCB-81	83	91	88	75
PCB-126	89	81	77	77
PCB-169	93	97	100	100

Table 2 - Co-planary PCBs ¹³C recoveries across cleanup - fish oil used as such, no extraction

Conclusions

The integrated PLE/Low Solvent EconoPrep gives excellent recoveries for a number of matrices. It is a green option with low electrical power use. The system uses only a total volume of 150-200 mL of hexane and toluene and no dichloromethane for the cleanup step.



Combined PLE/Low Solvent
Total EconoPrep

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