

Analysis of Soil for Polychlorinated Dibenzo-p-dioxins, Furans and Biphenyls using Pressurized Liquid Extraction and Automated Clean Up

Introduction

As with sediment, polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs) and biphenyls (PCBs) have been found in many types of soil. Soils are routinely analyzed for these compounds as many countries have adopted regulations for soil remediation, which is often required when concentrations (expressed as Toxic Equivalents) exceed 1 ppb.

US EPA methods 1613 and 1668 are frequently used for PCDD/Fs and PCBs analyses. Traditionally processing of soil for chlorinated compounds has involved multi-day Soxhlet extraction and manual sample clean up using column chromatography. As an alternative to obtain faster and more reliable data, these various steps have been automated. This application note describes the automated Pressurized Liquid Extraction (PLE) and automated open column chromatography clean up (PowerPrep) of soil.

Instrumentation

- FMS, Inc. PLE®
- FMS, Inc. PowerPrep®
- FMS, Inc. SuperVap® 6 Concentrator
- FMS, Inc. SuperVap® Vial Concentrator
- FMS, Inc. 250 mL concentrator tubes (1 mL termination)
- Thermo Trace GC Ultra with high res magnetic sector DFS Thermo mass spec

Consumables

- FMS, Inc. Jumbo Acidified Silica column
- FMS, Inc. Classical Acid-Base-Neutral column
- FMS, Inc. Basic Alumina column
- FMS, Inc. Carbon-Celite column
- Millipore OmniSolv® Benzene
- Fisher Optima® Dichloromethane
- Fisher Optima® Ethylacetate
- Fisher Optima® Hexane

- Fisher Optima® Toluene
- 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

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

PowerPrep Clean Up

- Standard 25-step program
- Install jumbo silica, classical ABN, alumina and carbon/celite columns
- Mixes used are hexane, 2%/98% dichloromethane/hexane, 50%/50% dichloromethane/hexane, 50%/50% ethylacetate/benzene, and toluene



- Run conditioning steps 1-13 with columns in place
- Load sample (in hexane)
- Elute silica with 150 mLs hexane (waste)
- Elute alumina with 60 mLs 2%/98% DCM/hexane (collect as F1)
- Elute alumina with 120 mLs 50%/50% DCM/hexane (collect as F1)
- Elute carbon with 4 mL 50%/50% ethyl-acetate/benzene (collect as F1)
- Elute carbon with 75 mLs toluene (collect as F2)

SuperVap step (above)

Vial Evaporator

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

Table with native soil values and ¹³C-labeled recoveries.

	native pg/g	recoveries %
2378-T4CDF	178.78	83%
2378-T4CDD	150.69	85%
12378-P5CDF	363.36	68%
23478-P5CDF	153.69	72%
12378-P5CDD	39.75	74%
123478-H6CDF	125.33	66%
123678-H6CDF	117.36	67%
234678-H6CDF	73.57	80%
123789-H6CDF	254.48	92%
123478-H6CDD	290.68	80%
123678-H6CDD	97.64	76%
123789-H6CDD	127.60	
1234678-H7CDF	89.06	72%
1234789-H7CDF	197.00	84%
1234678-H7CDD	205.63	87%
OCDF	151.13	
OCDD	270.88	85%



Table with native soil values and ¹³C-labeled recoveries.

		native pg/g	recoveries %
33'44'-T4CB	77	14.08	70%
344'5-T4CB	81	< 2.00	72%
233'44'-P5CB	105	23.10	71%
2344'5-P5CB	114	< 2.00	69%
23'44'5-P5CB	118	48.10	69%
2'344'5-P5CB	123	4.73	69%
33'44'5-P5CB	126	< 2.00	76%
233'44'5-H6CB	156	5.52	63%
233'44'5'-H6CB	157	< 2.00	58%
23'44'55'-H6CB	167	3.07	61%
33'44'55'-H6CB	169	< 2.00	67%
233'44'55'-H7CB	170	8.82	57%
22'344'55'-H7CB	180	22.28	56%
233'44'55'-H7CB	189	< 2.00	62%

Conclusions

PCB concentration found in the soil were low but higher values were seen for PCDD/Fs. The combined PLE and PowerPrep sample processing gave excellent recoveries of the labeled ¹³C standards. With extraction times of ~ 60 min and sample clean up taking only a few hours, same-day analysis of soil samples is now possible.



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