Application Note

From Samples to Reports: Rapid Dioxins/PCBs Analysis Using Automated Pressurized Liquid Extraction and Semi-Automated Cleanup

Introduction

The continued interest in Persistent Organic Pollutants (POPs), such as polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs), and biphenyls (PCBs) has led to a variety of automated systems for the extraction and cleanup of complex sample matrices. This resulted in development of automated Pressurized Liquid Extraction (PLE) (as an alternative to Soxhlet) and automated cleanup (as an alternative to manual preparative open column chromatography).

The PLE technique has been very successful with extraction of a wide variety of, among others, environmental and food samples lasting only about 1 h, with reliable and reproducible accuracy and recoveries.

To meet demands for a low-cost cleanup method that requires little financial investment, we combined the features of the automated systems - 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

- FMS, Inc. EZPrep Dioxin & PCBs sample preparation system
- FMS, Inc PLE® extraction system
- FMS, Inc. SuperVap® 12 position 50 mL Concentrator
- FMS, Inc. SuperVap® Vial Concentrator
- Agilent 7010B TripleQuad GC/MS/MS System with J&W DB-5 GC Column, 60 m, 0.25 mm, 0.25 µm

Consumables

- FMS, Inc PLE endcaps
- FMS, Inc. High-Capacity Acidic Silica column
- FMS, Inc. Carbon column
- FMS, Inc. 6 g Basic Alumina column
- Dichloromethane Pesticide Grade
- Hexane Pesticide Grade
- Toluene Pesticide Grade
- Relevant ^{13}C PCDD/Fs and PCBs isotope dilution and recovery standards

PLE

- 5-10 g sample mixed with Hydromatrix® 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
- Nitrogen flush to transfer analytes and extract to 250 mL collection tubes

SuperVap Concentration

- Pre-heat temperature: 50 °C
- Pre-heat time: 5 min
- Heat in Sensor mode: 50 °C
- Nitrogen Pressure: 8 psi
- Solvent exchange to hexane
- Reduce sample volume to 1 mLs

Sample Clean Up Procedure

Stage 1:

- Assemble columns in order high-capacity acidic silica-carbon-alumina.
- Syringe vial at top is used for conditioning and sample loading.
- Columns are conditioned with 40 mL 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 160 mL of hexane (vacuum, waste)

FMS
Fluid Management Systems

Stage 2:
■ Carbon and alumina columns are eluted in reverse direction with 40 mL toluene for collection under vacuum
■ Two fractions are collected: Fraction 1 with PCDD/Fs and co-planary- PCBs and Fraction 2 with mono- and di-ortho PCBs
■ Total run time is about 45 min
■ Number of parallel sample cleanup channels is unlimited
■ Low solvent volume of collected fractions reduces time required for sample concentration

SuperVap step (above)
No solvent exchange

Vial Evaporator
■ Reduce sample to 10 uL final volume under 1.5 psi nitrogen at 25 ^{o}C

Analysis 7010B Agilent TripleQuad GC/MS/MS

PCDD/Fs
Carrier gas helium 1.2 mL/min
T_{inj}= 300 ^{o}C split/splitless
Inject 1 mL sample
T-program: 130 ^{o}C 1 min hold
40 ^{o}C/min to 200 ^{o}C no hold
3 ^{o}C/min to 235 ^{o}C no hold
5 ^{o}C/min to 300 oC 13 min hold
Transfer line: 280 ^{o}C
Ion source: 300 ^{o}C with Quad 1 and 2 at 150 ^{o}C

PCBs
Carrier gas helium 1.2 mL/min
T_{inj}= 280 ^{o}C split/splitless
Inject 1 mL sample
T-program: 130 ^{o}C 1 min hold
40 ^{o}C/min to 200 ^{o}C no hold
3 ^{o}C/min to 235 ^{o}C no hold
5 ^{o}C/min to 300 oC 9 min hold
Transfer line: 280 ^{o}C
Ion source: 300 ^{o}C with Quad 1 and 2 at 150 ^{o}C

natives in pg	spike	IDC-1	IDC-2	IDC-3	IDC-4	Average	RSD (%)	Acceptable window
2,3,7,8 TCDF	400.0	91.8%	97.2%	96.4%	93.4%	94.7%	2.7%	70%-130%
2,3,7,8 TCDD	400.0	93.0%	97.4%	95.4%	94.7%	95.1%	1.9%	70%-130%
1,2,3,7,8 PCDF	2000.0	94.9%	99.9%	95.2%	96.7%	96.7%	2.3%	70%-130%
2,3,4,7,8 PCDF	2000.0	91.9%	96.9%	94.7%	94.4%	94.5%	2.1%	70%-130%
1,2,3,7,8 PCDD	2000.0	96.3%	100.0%	101.5%	98.2%	99.0%	2.3%	70%-130%
1,2,3,4,7,8 HxCDF	2000.0	99.2%	101.9%	99.8%	103.7%	101.2%	2.0%	70%-130%
1,2,3,6,7,8 HxCDF	2000.0	98.8%	101.7%	102.3%	97.0%	100.0%	2.5%	70%-130%
2,3,4,6,7,8 HxCDF	2000.0	99.6%	101.7%	100.7%	102.0%	101.0%	1.1%	70%-130%
1,2,3,4,7,8 HxCDD	2000.0	106.2%	100.4%	99.8%	98.0%	101.1%	3.5%	70%-130%
1,2,3,6,7,8 HxCDD	2000.0	94.5%	107.7%	107.0%	106.1%	103.8%	6.0%	70%-130%
1,2,3,7,8,9 HxCDD	2000.0	105.9%	116.6%	111.6%	107.7%	110.4%	4.3%	70%-130%
1,2,3,7,8,9 HxCDF	2000.0	97.2%	101.8%	99.5%	100.4%	99.7%	2.0%	70%-130%
1,2,3,4,6,7,8 HpCDF	2000.0	94.2%	99.3%	95.6%	93.5%	95.7%	2.7%	70%-130%
1,2,3,4,6,7,8 HpCDD	2000.0	96.8%	99.0%	98.9%	97.9%	98.2%	1.1%	70%-130%
1,2,3,4,7,8,9 HpCDF	2000.0	94.0%	97.3%	97.1%	95.1%	95.9%	1.6%	70%-130%
OCDD	4000.0	97.3%	101.4%	103.3%	101.1%	100.8%	2.5%	70%-130%
OCDF	4000.0	100.7%	104.4%	103.9%	102.0%	102.7%	1.7%	70%-130%

Table 1 - Native PCDD/Fs for Initial Demonstration of Capability - Native spike 400-4000 pg - native amounts reported as percent recovery of spike (extraction, cleanup, and concentration - note that these are not ^{13}C recoveries)

natives in pg	spike	IDC-1	IDC-2	IDC-3	IDC-4	Average	RSD (%)	Acceptable window
PCB-81	4000.0	92.3%	93.3%	94.0%	94.1%	93.4%	0.9%	70%-130%
PCB-77	4000.0	97.8%	95.9%	96.3%	98.3%	97.1%	1.2%	70%-130%
PCB-123	4000.0	90.6%	93.4%	95.0%	94.9%	93.5%	2.2%	70%-130%
PCB-118	4000.0	96.3%	107.0%	102.4%	98.8%	101.1%	4.6%	70%-130%
PCB-114	4000.0	88.7%	91.5%	93.1%	94.2%	91.9%	2.6%	70%-130%
PCB-105	4000.0	94.9%	101.0%	99.7%	99.4%	98.8%	2.7%	70%-130%
PCB-126	4000.0	99.7%	101.4%	101.7%	101.4%	101.0%	0.9%	70%-130%
PCB-167	4000.0	90.6%	94.3%	95.7%	96.1%	94.2%	2.7%	70%-130%
PCB-156	4000.0	89.1%	88.2%	78.6%	89.4%	86.3%	6.0%	70%-130%
PCB-157	4000.0	91.5%	90.6%	94.7%	91.9%	92.2%	1.9%	70%-130%
PCB-169	4000.0	91.4%	93.9%	94.1%	94.9%	93.6%	1.6%	70%-130%
PCB-189	4000.0	88.7%	91.0%	92.7%	93.7%	91.5%	2.4%	70%-130%

Table 2 - Native PCBs for Initial Demonstration of Capability - Native spike 4000 pg - native amounts reported as percent recovery of spike (extraction, cleanup, and concentration - note that these are not ^{13}C recoveries)

MDL study	native										
	spike	ppt	ppt	ppt	ppt	ppt	ppt	ppt	ppt	ppt	ppt
	ppt	MDL-1	MDL-2	MDL-3	MDL-4	MDL-5	MDL-6	MDL-7	MB	STDEV	MDL
2,3,7,8 TCDF	0.40	0.35	0.30	0.30	0.28	0.32	0.39	0.32	0.09	0.04	0.12
2,3,7,8 TCDD	0.40	0.39	0.25	0.38	0.30	0.35	0.40	0.31	0.08	0.05	0.17
1,2,3,7,8 PCDF	2.00	1.84	1.36	1.73	1.42	1.57	2.01	1.63	0.41	0.23	0.72
2,3,4,7,8 PCDF	2.00	1.85	1.27	1.71	1.36	1.57	1.94	1.66	0.41	0.24	0.77
1,2,3,7,8 PCDD	2.00	1.82	1.44	1.76	1.29	1.77	1.99	1.57	0.37	0.24	0.76
1,2,3,4,7,8 Hx CDF	2.00	2.00	1.40	1.91	1.46	1.76	2.02	1.96	0.36	0.26	0.82
1,2,3,6,7,8 Hx CDF	2.00	1.93	1.31	1.67	1.35	1.73	1.96	1.62	0.39	0.25	0.80
2,3,4,6,7,8 Hx CDF	2.00	1.89	1.26	1.74	1.38	1.53	2.15	1.78	0.38	0.31	0.96
1,2,3,4,7,8 Hx CDD	2.00	1.85	1.24	1.70	1.27	1.86	1.89	1.62	0.37	0.28	0.87
1,2,3,6,7,8 Hx CDD	2.00	1.84	1.17	1.89	1.40	1.78	2.10	1.77	0.36	0.31	0.99
1,2,3,7,8,9 Hx CDD	2.00	1.86	1.30	1.88	1.48	1.76	2.27	1.88	0.40	0.31	0.98
1,2,3,7,8,9 Hx CDF	2.00	1.91	1.35	1.84	1.30	1.77	1.95	1.82	0.37	0.27	0.84
1,2,3,4,6,7,8 HpCDF	2.00	1.84	1.61	1.95	1.41	1.76	2.10	1.71	0.60	0.22	0.71
1,2,3,4,6,7,8 HpCDD	2.00	1.83	1.46	1.85	1.52	2.04	1.89	1.86	0.51	0.21	0.66
1,2,3,4,7,8,9 HpCDF	2.00	1.96	1.28	1.64	1.43	1.77	2.24	1.62	0.57	0.32	1.02
OCDD	4.00	4.01	2.81	3.98	2.92	4.63	4.59	3.88	1.03	0.72	2.27
OCDF	4.00	4.10	2.50	3.58	2.64	3.51	4.26	3.74	0.93	0.68	2.12

Table 3 - Native PCDD/Fs Method Detection Limit in pg/g - extraction, cleanup, and concentration -

MDL study	native										
	spike	ppt	ppt	ppt	ppt	ppt	ppt	ppt	ppt	ppt	ppt
	ppt	MDL-1	MDL-2	MDL-3	MDL-4	MDL-5	MDL-6	MDL-7	MB	STDEV	MDL
PCB_81	10.00	8.28	9.44	8.71	8.91	8.40	8.64	8.78	0.75	0.38	1.19
PCB_77	10.00	8.06	8.04	7.98	8.72	10.66	10.66	10.99	1.35	1.40	4.40
PCB_123	10.00	7.59	8.78	9.16	8.33	9.16	7.30	8.33	0.60	0.73	2.29
PCB_118	10.00	8.88	9.16	9.14	9.40	9.33	11.24	11.92	1.25	1.20	3.76
PCB_114	10.00	7.64	8.85	9.02	8.03	8.74	8.81	8.75	0.10	0.51	1.60
PCB_105	10.00	8.66	8.26	8.79	7.81	8.16	8.06	9.44	2.50	0.55	1.73
PCB_126	10.00	7.83	8.86	8.53	8.29	8.11	8.15	8.14	0.70	0.33	1.05
PCB_167	10.00	8.46	10.10	8.20	8.60	9.54	8.33	8.42	0.95	0.72	2.27
PCB_156	10.00	9.56	7.91	7.32	7.17	6.67	8.41	8.61	0.70	0.99	3.11
PCB_157	10.00	10.03	8.18	10.27	8.35	11.58	9.40	9.61	0.60	1.17	3.67
PCB_169	10.00	8.39	8.71	8.41	8.18	7.31	8.20	7.71	0.85	0.47	1.48
PCB_189	10.00	8.33	9.48	9.39	8.36	9.71	8.64	8.71	0.50	0.57	1.79

Table 4 - Native PCBs Method Detection Limit in pg/g - extraction, cleanup, and concentration -

	Channel-1	Channel-2	Channel-3	Channel-4	Channel-5	Channel-6	Average	STDEV	RSD (%)
2,3,7,8 TCDF	95	82	83	81	88	92	87	5.7	6.5
2,3,7,8 TCDD	93	88	81	79	87	87	86	4.9	5.8
1,2,3,7,8 PCDF	89	85	77	77	77	87	82	5.5	6.7
2,3,4,7,8 PCDF	97	92	86	81	79	91	88	7.1	8.2
1,2,3,7,8 PCDD	97	91	87	87	91	93	91	3.8	4.2
1,2,3,4,7,8 HxCDF	81	77	73	72	78	75	76	3.4	4.5
1,2,3,6,7,8 HxCDF	74	78	71	70	88	77	76	6.6	8.6
2,3,4,6,7,8 HxCDF	80	77	78	74	90	91	82	7.1	8.7
1,2,3,4,7,8 HxCDD	84	78	73	73	85	92	81	7.5	9.3
1,2,3,6,7,8 HxCDD	80	77	77	73	93	79	80	6.9	8.6
1,2,3,7,8,9 HxCDF	84	76	73	72	78	87	79	6.0	7.7
1,2,3,4,6,7,8 HpCDF	74	71	74	72	74	72	73	1.3	1.8
1,2,3,4,6,7,8 HpCDD	84	80	79	72	79	87	80	5.1	6.3
1,2,3,4,7,8,9 HpCDF	84	74	75	74	87	90	81	7.1	8.8
OCDD	72	77	70	71	87	77	76	6.3	8.4

Table 5 - ^{13}C PCDD/F percent recoveries across extraction, cleanup, and concentration - no matrix

	Channel-1	Channel-2	Channel-3	Channel-4	Channel-5	Channel-6	Average	STDEV	RSD (%)
PCB-81	95	109	93	104	109	100	102	6.9	6.7
PCB-77	105	96	82	93	92	103	95	8.3	8.8
PCB-123	88	95	75	76	84	87	84	7.6	9.1
PCB-118	87	90	76	74	75	87	82	7.2	8.9
PCB-114	88	93	79	82	88	97	88	6.7	7.6
PCB-105	94	90	80	98	86	102	92	8.0	8.8
PCB-126	80	90	78	96	80	92	86	7.6	8.8
PCB-167	84	84	71	72	86	77	79	6.6	8.3
PCB-156	80	83	72	74	86	74	78	5.7	7.3
PCB-157	80	83	76	84	92	73	81	6.7	8.2
PCB-169	88	85	84	95	100	104	93	8.3	8.9
PCB-189	80	83	72	85	90	82	82	6.0	7.3

Table 6 - ^{13}C PCBs percent recoveries across extraction, cleanup, and concentration - no matrix

	Cod oil		Pumpkin oil		Corn oil	
Natives in pg	Channel-1	Channel-2	Channel-3	Channel-4	Channel-5	Channel-6
T 2,3,7,8 TCDF	0.0	0.0	0.3	0.3	0.2	0.1
T 2,3,7,8 TCDD	0.3	0.0	0.0	0.0	0.0	0.2
T 1,2,3,7,8 PCDF	0.1	0.1	0.2	0.2	0.4	0.0
T 2,3,4,7,8 PCDF	0.2	0.2	0.1	0.5	0.4	0.5
T 1,2,3,7,8 PCDD	0.0	0.0	0.4	0.0	0.3	0.5
T 1,2,3,6,7,8 HxCDF	0.0	0.5	0.0	0.2	0.6	0.2
T 1,2,3,4,7,8 HxCDF	0.1	0.2	0.0	0.0	0.0	0.0
T 2,3,4,6,7,8 HxCDF	0.7	0.3	0.6	0.5	0.3	0.4
T 1,2,3,4,7,8 HxCDD	0.5	0.0	0.7	0.6	0.5	0.3
T 1,2,3,6,7,8 HxCDD	0.3	0.2	0.8	0.0	0.5	0.0
T 1,2,3,7,8,9 HxCDD	0.0	0.2	0.3	0.0	0.0	0.0
T 1,2,3,7,8,9 HxCDF	0.3	0.0	0.2	0.4	0.3	0.6
T 1,2,3,4,7,8,9 HpCDF	0.3	0.0	1.0	0.3	0.3	0.3
T 1,2,3,4,6,7,8 HpCDF	0.4	0.0	0.2	0.3	0.3	0.3
T 1,2,3,4,6,7,8 HpCDD	0.0	0.0	0.4	0.2	1.0	0.0
T OCDF	0.0	0.4	0.2	0.0	0.0	0.0
T OCDD	0.0	0.0	0.0	0.1	0.0	0.0

Table 7 - Native PCDD/Fs in oils - cleanup and concentration - 2.5 g oil - data in pg

	Cod oil		Pumpkin oil		Corn oil	
13C recoveries (%)	Channel-1	Channel-2	Channel-3	Channel-4	Channel-5	Channel-6
2,3,7,8 TCDF	85	88	81	97	94	89
2,3,7,8 TCDD	85	87	84	97	92	90
1,2,3,7,8 PCDF	76	80	71	81	80	76
2,3,4,7,8 PCDF	82	74	74	85	83	85
1,2,3,7,8 PCDD	86	83	74	86	82	84
1,2,3,4,7,8 HxCDF	71	95	102	90	90	74
1.2.3.6.7.8 HxCDF	80	84	93	101	94	77
2,3,4,6,7,8 HxCDF	84	92	88	101	95	73
1,2,3,4,7,8 HxCDD	75	81	91	97	102	84
1,2,3,6,7,8 HxCDD	77	88	102	105	96	83
1,2,3,7,8,9 HxCDF	87	85	82	71	97	70
1,2,3,4,6,7,8 HpCDF	71	83	76	82	84	76
1,2,3,4,6,7,8 HpCDD	75	90	84	90	94	71
1,2,3,4,7,8,9 HpCDF	75	86	84	89	92	72
OCDD	72	79	73	76	83	74

Table 8 - ^{13}C PCDD/Fs in oils - cleanup and concentration - 2.5 g oil - recoveries in %

	Cod oil		Pumpkin oil		Corn oil	
Natives in pg	Channel-1	Channel-2	Channel-3	Channel-4	Channel-5	Channel-6
PCB-81	0.8	0.9	0.7	2.9	0.8	0.5
PCB-77	1.4	1.8	5.1	4.3	5.6	3.4
PCB-123	923.0	923.4	92.4	99.8	12.3	10.7
PCB-118	6207.4	6123.5	171.3	194.5	18.7	21.3
PCB-114	148.0	125.6	0.0	0.0	0.0	14.5
PCB-105	2167.8	2108.9	32.5	39.8	4.8	6.7
PCB-126	14.0	13.4	16.7	5.6	6.7	8.4
PCB-167	4256.7	3859.5	31.4	41.3	5.2	0.0
PCB-156	1367.0	1300.4	21.4	21.4	3.2	12.1
PCB-157	345.6	356.8	35.6	51.6	14.8	5.6
PCB-169	7.0	4.5	0.0	0.8	2.1	1.2
PCB-189	0.0	0.0	12.8	9.8	0.0	0.0

Table 9 - Native PCBs in oils - cleanup and concentration - 2.5 g oil - data in pg

	Cod oil		Pumpkin oil		Corn oil	
13C recoveries (%)	Channel-1	Channel-2	Channel-3	Channel-4	Channel-5	Channel-6
PCB-81	88	76	78	84	85	78
PCB-77	85	81	87	82	95	90
PCB-123	93	86	87	81	81	84
PCB-118	80	95	92	93	88	80
PCB-114	79	76	86	79	77	86
PCB-105	85	81	88	77	83	85
PCB-126	95	86	82	85	85	85
PCB-167	89	83	80	78	80	78
PCB-156	82	77	79	75	80	75
PCB-157	89	79	79	75	79	79
PCB-169	95	80	91	82	81	79
PCB-189	93	83	87	80	85	82

Table 10 - ^{13}C PCBs in oils - cleanup and concentration - 2.5 g oil - recoveries in %

Natives (pg)	Feed-1	Feed-2	Soil-1	Soil-2	MB
2,3,7,8 TCDF	0.0	0.0	1.2	4.1	0.0
2,3,7,8 TCDD	0.0	0.0	3.4	1.1	0.1
1,2,3,7,8 PCDF	0.1	0.0	6.3	3.6	0.0
2,3,4,7,8 PCDF	0.1	0.1	2.8	5.0	0.1
1,2,3,7,8 PCDD	0.0	0.0	3.8	5.0	0.1
1,2,3,4,7,8 HxCDF	0.0	0.1	22.6	8.2	0.1
1,2,3,6,7,8 HxCDF	0.0	0.1	38.4	20.4	0.1
2,3,4,6,7,8 HxCDF	0.0	0.0	0.0	20.8	0.0
1,2,3,4,7,8 HxCDD	0.0	0.0	21.4	0.0	0.1
1,2,3,6,7,8 HxCDD	0.1	0.0	22.5	18.1	0.0
1,2,3,7,8,9 HxCDD	0.1	0.0	16.1	19.1	0.1
1,2,3,7,8,9 HxCDF	0.0	0.2	0.0	0.0	0.1
1,2,3,4,6,7,8 HpCDF	0.1	0.2	68.0	69.2	0.0
1,2,3,4,6,7,8 HpCDD	0.0	0.3	165.3	140.9	0.0
1,2,3,4,7,8,9 HpCDF	0.1	0.1	402.9	333.5	0.0
OCDD	1.7	2.5	5606.4	5945.6	0.1
OCDF	0.1	0.1	209.9	209.4	0.1

Table 11 - native PCDD/Fs - extraction, cleanup, and concentration - 5g feed and 10g soil - MB = method blank

13C recoveries (%)	Feed-1	Feed-2	Soil-1	Soil-2	MB
2,3,7,8 TCDF	96	83	114	100	93
2,3,7,8 TCDD	103	91	92	89	91
1,2,3,7,8 PCDF	69	80	93	81	88
2,3,4,7,8 PCDF	72	74	91	83	98
1,2,3,7,8 PCDD	76	72	89	74	88
1,2,3,4,7,8 HxCDF	88	84	74	68	90
1,2,3,6,7,8 HxCDF	88	72	82	73	121
2,3,4,6,7,8 HxCDF	83	72	89	78	96
1,2,3,4,7,8 HxCDD	73	71	96	74	125
1,2,3,6,7,8 HxCDD	76	72	93	91	102
1,2,3,7,8,9 HxCDF	96	93	102	103	106
1,2,3,4,6,7,8 HpCDF	79	79	87	70	90
1,2,3,4,6,7,8 HpCDD	79	95	87	93	93
1,2,3,4,7,8,9 HpCDF	75	81	100	108	110
OCDD	76	73	86	84	77

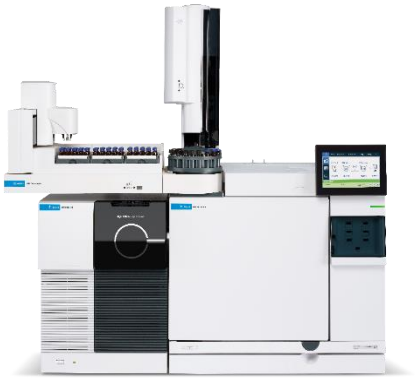
Table 12 - ^{13}C PCDD/Fs - extraction, cleanup, and concentration - 5g feed and 10g soil - MB = method blank

Natives (pg)	Feed-1	Feed-2	Soil-1	Soil-2	MB
PCB-81	0.0	0.0	7.0	0.0	0.0
PCB-77	31.0	21.4	116.8	49.7	5.1
PCB-123	0.0	0.0	0.0	0.0	4.5
PCB-118	113.3	93.0	705.9	515.8	3.0
PCB-114	0.0	0.0	0.0	0.0	3.0
PCB-105	46.0	35.8	309.3	230.6	1.4
PCB-126	0.0	0.0	19.0	23.6	0.0
PCB-167	0.0	9.5	253.3	244.1	0.5
PCB-156	8.7	0.0	0.0	0.0	0.5
PCB-157	8.9	0.0	0.0	0.0	0.5
PCB-169	0.0	0.0	4.9	5.0	0.2
PCB-189	3.1	7.2	2.6	0.0	0.3

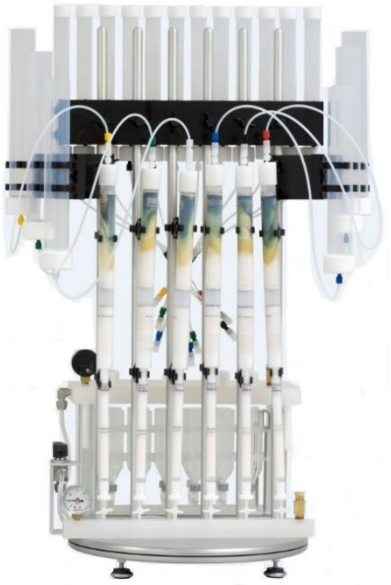
Table 13 - native PCBs - extraction, cleanup, and concentration - 5g feed and 10g soil - MB = method blank

13C recoveries (%)	Feed-1	Feed-2	Soil-1	Soil-2	MB
PCB-81	71	85	103	92	70
PCB-77	75	87	104	94	99
PCB-123	99	113	91	100	74
PCB-118	73	84	83	98	75
PCB-114	76	90	98	103	71
PCB-105	88	80	99	91	75
PCB-126	88	93	90	96	81
PCB-167	75	94	90	86	78
PCB-156	80	77	72	70	72
PCB-157	80	75	93	94	71
PCB-169	78	71	80	94	84
PCB-189	88	81	109	100	80

Table 14 - ^{13}C PCBs - extraction, cleanup, and concentration - 5g feed and 10g soil - MB = method blank



Agilent 7010B
TripleQuad



EZPrep Dioxin & PCBs Sample Preparation System

Conclusions

Excellent Demonstration of Capability, Method Detection Limit results, and ^{13}C PCDD/F and PCB recoveries are seen with FMS automated extraction, FMS semi-automated cleanup and Agilent 7010B TripleQuad GC/MS/MS analysis. Results are shown in Tables 1-6. The Pressurized Liquid Extraction (PLE) is much faster than traditional Soxhlet methods and uses less electrical power. The semi-automated FMS EZPrep123 Cleanup System is mostly composed of disposable parts and 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 inexpensive alternative to the fully automated clean up equipment, and processing times are much shorter than other manual procedures using certified columns. The easy to operate 7010B analytical system has very good sensitivity and is a less expensive alternative to the magnetic sector high resolution instruments. The combination of FMS and Agilent technologies make same-day POPs results possible.

For more information contact FMS:
FMS, Inc.
900 Technology Park Drive
Billerica, MA 01821, USA
Phone : 1-617 393-2396
Fax : 1-617 393-0194
Email: onlineinfo@fms-inc.com
Web site: www.fms-inc.com