Application Note

From Samples to Reports: Rapid Dioxins/PCBs Analysis Using Low-Cost Automated Sample Preparation

Introduction

Persistent organic pollutants (POPs) such as polychlorinated dibenzo-p-dioxins (PCDDs), dibenzofurans (PCDFs) and biphenyls (PCBs) have been strictly regulated in many countries. This has created a steady flow of samples analyzed by small and large environmental, food and government laboratories worldwide. POPs sample processing is labor-intensive and prone to error. Compliance with regulatory procedures and accreditation requirements can result in a lengthy method validation effort. Strict quality assurance and quality control (QA/QC) requirements apply, and sample matrices can be very complex. In many cases native background interferences can be orders of magnitude higher than analytes. Therefore, in most cases elaborate sample cleanup is needed.

Automation of sample cleanup has focused on reduction of background contamination via use of a closed system and pre-packaged disposable columns (often produced in clean rooms). Automated programs via computer and mechanically driven sample processing channels with solvent pumps can reduce time required of cleanup and can be run largely unattended.

Since fully automated cleanup systems for POPs can be quite expensive, a low-cost automated alternative was developed that requires less financial investment, while having most of the same features (closed system, pre-packaged columns). The system is simple in design and uses stackable columns. It uses a multi-pump to do all the sample prep steps.

Instrumentation

■ FMS, Inc. EZPrep Plus Dioxin & PCBs sample preparation system with multi-pump

■ 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. High-Capacity Acidic Silica column

■ FMS, Inc. Carbon column

■ FMS, Inc. 6 g Basic Alumina column

■ Fisher Hexane Pesticide Grade

■ Fisher Toluene Pesticide Grade

■ Relevant ^{13}C PCDD/Fs and PCBs isotope dilution and recovery standards

PLE

■ 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 ºC and 1500 psi

■ Nitrogen flush to transfer analytes and extract to 250 mL collection tubes

SuperVap Concentration

■ Pre-heat temperature: 50 ºC

■ Pre-heat time: 5 min

■ Heat in Sensor mode: 50 ºC

■ 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.

■ Sample cartridge on top is used for sample loading and hexane elution.

■ Columns are conditioned with 40 mL of hexane using multi-pump (waste).

■ Samples are loaded across system and eluted with 160 mL of hexane with multi-pump (waste).

■ Remaining solvent is removed with nitrogen flush.

FMS

Fluid Management Systems

Stage 2:
■ Carbon and alumina columns are each individually eluted in reverse direction with 40 mL toluene for collection with multi-pump.
■ 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 40 min
■ Low solvent volume of collected fractions reduces time required for sample concentration

SuperVap Concentration

- Pre-heat temperature: 50 ^{o}C
- Pre-heat time: 5 min
- Heat in Sensor mode: 50 ^{o}C
- Nitrogen Pressure: 8 psi
- Reduce sample volume to 1 mLs

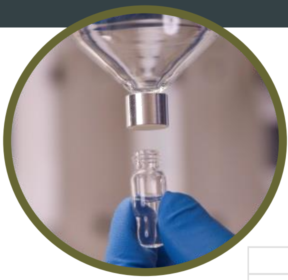
Vial Evaporator
■ Reduce sample to 10 uL final volume under 1.5 psi nitrogen at 25 ^{o}C

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 ^{o}C 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 ^{o}C 9 min hold
Transfer line: 280 ^{o}C
Ion source: 300 ^{o}C with Quad 1 and 2 at 150 ^{o}C

FMS
Fluid Management Systems



natives in pg	spike	IDC-1	IDC-2	IDC-3	IDC-4	IDC-5	IDC-6	Average	RSD (%)	Acceptable window
2,3,7,8 TCDF	400.0	101.3%	86.8%	87.2%	90.6%	92.1%	87.0%	90.8%	6.1%	70%-130%
2,3,7,8 TCDD	400.0	106.1%	94.4%	93.8%	102.2%	101.1%	92.2%	98.3%	5.6%	70%-130%
1,2,3,7,8 PCDF	2000.0	97.8%	91.8%	92.7%	96.5%	97.8%	92.9%	94.9%	2.9%	70%-130%
2,3,4,7,8 PCDF	2000.0	95.9%	92.2%	93.2%	94.6%	97.2%	91.3%	94.1%	2.4%	70%-130%
1,2,3,7,8 PCDD	2000.0	97.7%	93.4%	91.3%	95.0%	95.5%	90.8%	93.9%	2.8%	70%-130%
1,2,3,4,7,8 HxCDF	2000.0	107.4%	92.0%	93.6%	95.5%	97.0%	93.2%	96.4%	5.8%	70%-130%
1,2,3,6,7,8 HxCDF	2000.0	93.4%	87.5%	89.1%	90.9%	92.3%	88.7%	90.3%	2.5%	70%-130%
2,3,4,6,7,8 HxCDF	2000.0	94.3%	92.4%	91.2%	93.0%	95.9%	89.8%	92.7%	2.3%	70%-130%
1,2,3,4,7,8 HxCDD	2000.0	112.3%	91.4%	94.1%	95.9%	94.5%	91.9%	96.7%	8.1%	70%-130%
1,2,3,6,7,8 HxCDD	2000.0	96.8%	91.3%	89.6%	88.8%	88.1%	86.0%	90.1%	4.1%	70%-130%
1,2,3,7,8,9 HxCDD	2000.0	95.6%	101.2%	88.8%	90.0%	92.1%	97.5%	94.2%	5.0%	70%-130%
1,2,3,7,8,9 HxCDF	2000.0	96.6%	90.6%	90.3%	92.5%	91.5%	90.0%	91.9%	2.7%	70%-130%
1,2,3,4,6,7,8 HpCDF	2000.0	99.2%	92.2%	93.9%	92.6%	94.9%	91.7%	94.1%	2.9%	70%-130%
1,2,3,4,6,7,8 HpCDD	2000.0	96.3%	90.3%	91.8%	95.4%	94.8%	91.1%	93.3%	2.7%	70%-130%
1,2,3,4,7,8,9 HpCDF	2000.0	95.7%	93.6%	94.3%	92.5%	92.9%	90.0%	93.2%	2.1%	70%-130%
OCDD	4000.0	100.1%	99.0%	98.9%	102.0%	103.0%	98.1%	100.2%	1.9%	70%-130%
OCDF	4000.0	97.5%	94.0%	96.4%	96.3%	98.6%	95.2%	96.3%	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	IDC-5	IDC-6	Average	RSD (%)	Acceptable window
PCB-81	4000	92.7%	91.9%	109.4%	98.9%	91.6%	104.1%	98.1%	7.5%	2800-5200
PCB-77	4000	101.4%	100.8%	105.8%	108.1%	100.5%	113.8%	105.1%	5.0%	2800-5200
PCB-123	4000	92.5%	89.8%	92.3%	103.7%	90.3%	90.7%	93.2%	5.6%	2800-5200
PCB-118	4000	85.6%	91.9%	89.3%	92.4%	89.6%	92.6%	90.2%	3.0%	2800-5200
PCB-114	4000	112.0%	107.5%	101.5%	100.5%	118.8%	100.4%	106.8%	7.0%	2800-5200
PCB-105	4000	112.1%	100.2%	99.6%	93.1%	100.3%	88.1%	98.9%	8.2%	2800-5200
PCB-126	4000	100.1%	106.0%	93.4%	85.9%	91.0%	105.2%	96.9%	8.4%	2800-5200
PCB-167	4000	97.8%	103.1%	101.0%	88.1%	102.5%	105.0%	99.6%	6.1%	2800-5200
PCB-156	4000	89.2%	91.3%	94.0%	92.1%	95.3%	93.0%	92.5%	2.3%	2800-5200
PCB-157	4000	93.7%	106.6%	91.7%	89.8%	96.6%	96.4%	95.8%	6.1%	2800-5200
PCB-169	4000	85.2%	82.0%	88.4%	80.5%	87.4%	90.6%	85.7%	4.5%	2800-5200
PCB-189	4000	92.8%	95.2%	94.1%	93.8%	95.8%	98.2%	95.0%	2.0%	2800-5200

Table 2 - Native PCBs 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)

MDL study	native spike ppt	ppt MDL-1	ppt MDL-2	ppt MDL-3	ppt MDL-4	ppt MDL-5	ppt MDL-6	ppt MDL-7	ppt MB	ppt STDEV	ppt MDL
2,3,7,8 TCDF	0.2	0.19	0.18	0.22	0.22	0.20	0.21	0.20	0.01	0.02	0.05
2,3,7,8 TCDD	0.2	0.18	0.19	0.21	0.22	0.17	0.22	0.19	0.00	0.02	0.06
1,2,3,7,8 PCDF	1.0	0.93	0.99	1.17	1.06	1.06	1.06	0.94	0.03	0.08	0.26
2,3,4,7,8 PCDF	1.0	1.00	0.96	1.04	1.02	0.99	1.03	0.93	0.02	0.04	0.13
1,2,3,7,8 PCDD	1.0	0.95	0.92	0.95	1.09	0.97	1.01	0.97	0.01	0.06	0.18
1,2,3,4,7,8 HxCDF	1.0	1.06	1.03	1.07	1.07	1.06	1.08	1.01	0.02	0.03	0.08
1,2,3,6,7,8 HxCDF	1.0	1.04	1.05	1.09	1.05	0.95	0.94	1.13	0.02	0.07	0.22
2,3,4,6,7,8 HxCDF	1.0	1.06	1.06	1.09	1.05	1.03	1.05	0.97	0.02	0.04	0.11
1,2,3,4,7,8 HxCDD	1.0	0.89	1.02	1.15	1.15	1.00	1.21	1.17	0.02	0.12	0.37
1,2,3,6,7,8 HxCDD	1.0	1.09	1.04	1.02	1.07	1.07	0.99	1.24	0.01	0.08	0.26
1,2,3,7,8,9 HxCDD	1.0	0.92	0.90	1.01	1.04	0.90	0.88	1.09	0.01	0.08	0.26
1,2,3,7,8,9 HxCDF	1.0	0.99	0.91	0.99	0.99	1.07	1.02	1.03	0.05	0.05	0.16
1,2,3,4,6,7,8 HpCDF	1.0	1.03	0.87	1.09	1.02	1.04	1.04	0.95	0.02	0.07	0.23
1,2,3,4,6,7,8 HpCDD	1.0	1.13	0.90	1.01	1.24	1.01	0.98	1.10	0.02	0.11	0.36
1,2,3,4,7,8,9 HpCDF	1.0	1.05	1.07	1.09	0.99	1.11	1.19	0.91	0.07	0.09	0.28
OCDD	2.0	2.05	2.48	2.26	2.27	2.29	2.32	2.68	0.03	0.20	0.62
OCDF	2.0	2.38	1.97	2.10	2.13	2.02	2.24	2.20	0.05	0.14	0.44

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

MDL study	native spike (ppt)	ppt MDL-1	ppt MDL-2	ppt MDL-3	ppt MDL-4	ppt MDL-5	ppt MDL-6	ppt MDL-7	ppt MB	ppt STDEV	ppt MDL
PCB-81	10.00	10.60	11.25	10.95	11.05	11.10	12.20	11.15	0.00	0.62	1.95
PCB-77	10.00	11.29	12.46	11.51	12.01	12.30	11.46	10.94	0.00	0.80	2.50
PCB-123	10.00	11.44	12.14	11.47	11.05	11.64	12.12	10.64	1.68	0.73	2.30
PCB-118	10.00	11.43	13.51	12.80	12.30	14.89	11.15	11.11	1.25	1.56	4.91
PCB-114	10.00	12.73	11.39	11.18	11.42	11.63	12.01	11.89	1.42	0.78	2.46
PCB-105	10.00	11.80	12.26	11.73	11.80	12.86	12.71	11.83	1.58	0.87	2.75
PCB-126	10.00	13.10	12.34	13.01	13.08	14.18	13.68	12.65	0.00	1.25	3.93
PCB-167	10.00	9.96	11.26	11.14	10.99	12.04	11.73	11.50	0.00	0.76	2.37
PCB-156	10.00	11.15	11.39	10.78	11.14	11.07	11.80	11.24	0.00	0.52	1.64
PCB-157	10.00	11.78	11.97	11.63	11.62	11.90	12.29	11.45	2.56	0.69	2.16
PCB-169	10.00	11.11	11.53	12.17	13.81	12.24	12.45	11.14	0.74	1.14	3.57
PCB-189	10.00	11.19	11.03	10.90	11.15	10.90	11.35	11.26	0.00	0.42	1.33

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	107	89	91	91	86	87	92	7.7	8.4
2,3,7,8 TCDD	102	94	88	85	89	87	91	6.4	7.1
1,2,3,7,8 PCDF	97	85	85	79	92	89	88	6.3	7.1
2,3,4,7,8 PCDF	100	92	90	81	93	90	91	6.2	6.8
1,2,3,7,8 PCDD	102	97	89	81	93	96	93	7.3	7.8
1,2,3,4,7,8 HxCDF	97	90	83	85	92	88	89	5.1	5.7
1,2,3,6,7,8 HxCDF	95	92	82	83	87	89	88	5.2	5.9
2,3,4,6,7,8 HxCDF	102	90	85	87	87	89	90	6.3	6.9
1,2,3,4,7,8 HxCDD	100	91	86	85	88	89	90	5.3	5.9
1,2,3,6,7,8 HxCDD	103	92	87	86	87	83	90	7.1	7.9
1,2,3,7,8,9 HxCDF	100	90	86	83	79	82	87	7.6	8.8
1,2,3,4,6,7,8 HpCDF	85	73	71	74	86	80	78	6.4	8.2
1,2,3,4,6,7,8 HpCDD	90	82	78	75	89	76	82	6.5	8.0
1,2,3,4,7,8,9 HpCDF	89	85	80	79	85	76	82	5.1	6.1
OCDD	76	78	78	72	86	76	78	4.6	6.0

Table 5 - ^{13}C PCDD/Fs 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	101	102	99	98	99	100	100	1.5	1.5
PCB-77	113	103	102	99	100	103	103	5.0	4.8
PCB-123	98	95	95	90	94	96	95	2.7	2.8
PCB-118	100	94	95	93	96	101	97	3.3	3.4
PCB-114	97	94	94	89	94	96	94	2.8	2.9
PCB-105	96	94	93	90	94	95	94	2.1	2.2
PCB-126	96	85	89	84	86	92	89	4.6	5.2
PCB-167	94	91	90	85	90	92	90	3.0	3.3
PCB-156	93	90	90	91	89	87	90	2.0	2.2
PCB-157	93	91	87	85	88	90	89	2.9	3.3
PCB-169	98	90	88	85	88	93	90	4.6	5.1
PCB-189	88	85	83	81	82	85	84	2.5	3.0

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
2,3,7,8 TCDF	3.3	0.1	0.1	0.4	0.2	0.1
2,3,7,8 TCDD	0.1	0.3	0.2	0.1	0.0	0.2
1,2,3,7,8 PCDF	0.5	1.0	0.1	0.3	0.6	1.6
2,3,4,7,8 PCDF	1.4	1.1	0.4	0.4	0.6	1.9
1,2,3,7,8 PCDD	0.7	0.1	0.4	0.3	0.7	0.8
1,2,3,6,7,8 HxCDF	2.1	0.2	2.2	0.6	0.2	1.8
1,2,3,4,7,8 HxCDF	2.0	0.2	2.1	0.6	0.2	1.7
2,3,4,6,7,8 HxCDF	2.0	0.7	1.9	0.7	0.2	1.8
1,2,3,4,7,8 HxCDD	2.3	1.2	1.0	0.6	1.2	2.6
1,2,3,6,7,8 HxCDD	2.2	1.2	0.9	0.4	1.1	2.4
1,2,3,7,8,9 HxCDD	2.2	1.2	0.9	0.3	0.9	2.4
1,2,3,7,8,9 HxCDF	2.1	1.3	0.6	0.3	0.2	2.2
1,2,3,4,7,8,9 HpCDF	1.1	0.5	0.2	0.2	0.7	1.1
1,2,3,4,6,7,8 HpCDF	0.9	0.2	0.5	0.2	0.3	1.6
1,2,3,4,6,7,8 HpCDD	0.8	0.5	0.4	0.2	0.1	1.0
OCDF	0.6	2.4	2.0	0.4	1.0	4.2
OCDD	1.2	0.0	0.5	0.6	0.7	0.1

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	95	87	83	91	79	98
2,3,7,8 TCDD	101	94	89	99	84	106
1,2,3,7,8 PCDF	92	84	81	93	75	92
2,3,4,7,8 PCDF	99	89	88	101	81	106
1,2,3,7,8 PCDD	97	87	87	101	80	105
1,2,3,4,7,8 HxCDF	80	72	74	92	74	89
1.2.3.6.7.8 HxCDF	99	91	90	88	86	95
2,3,4,6,7,8 HxCDF	96	85	84	91	80	89
1,2,3,4,7,8 HxCDD	85	76	74	79	70	85
1,2,3,6,7,8 HxCDD	104	95	93	110	87	95
1,2,3,7,8,9 HxCDF	96	85	84	94	82	86
1,2,3,4,6,7,8 HpCDF	82	75	71	79	74	75
1,2,3,4,6,7,8 HpCDD	83	76	75	88	76	95
1,2,3,4,7,8,9 HpCDF	82	73	73	86	76	94
OCDD	74	71	74	77	72	91

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

Natives in pg	Cod oil		Pumpkin oil		Corn oil	
	Channel-1	Channel-2	Channel-3	Channel-4	Channel-5	Channel-6
PCB-81	0.0	0.0	0.0	0.0	0.0	0.0
PCB-77	0.0	0.0	0.0	0.0	0.0	2.4
PCB-123	787.8	854.0	182.4	195.5	26.1	19.0
PCB-118	5858.0	5451.8	150.5	178.9	17.9	13.9
PCB-114	161.4	102.9	0.0	0.0	0.0	0.0
PCB-105	2027.4	1939.6	66.1	73.6	6.9	4.1
PCB-126	7.2	5.6	8.7	0.0	2.4	5.5
PCB-167	3579.5	3409.8	27.7	33.3	0.0	0.0
PCB-156	1261.0	1199.6	11.9	15.1	15.0	23.7
PCB-157	259.7	244.4	39.5	76.9	24.7	9.0
PCB-169	0.0	0.0	0.0	0.0	0.0	0.8
PCB-189	0.0	0.0	7.9	9.6	0.0	0.0

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

13C recoveries (%)	Cod oil		Pumpkin oil		Corn oil	
	Channel-1	Channel-2	Channel-3	Channel-4	Channel-5	Channel-6
PCB-81	92	91	83	87	82	88
PCB-77	97	98	82	83	87	92
PCB-123	79	86	80	79	73	79
PCB-118	80	90	97	87	89	89
PCB-114	81	90	96	80	90	80
PCB-105	77	84	85	76	78	74
PCB-126	99	100	88	97	96	102
PCB-167	89	97	85	83	84	78
PCB-156	86	93	85	79	79	73
PCB-157	90	95	85	81	79	73
PCB-169	83	86	75	86	77	77
PCB-189	72	75	74	74	73	74

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	3.1	3.0	0.1
2,3,7,8 TCDD	0.0	0.0	3.0	5.8	0.1
1,2,3,7,8 PCDF	0.0	0.0	4.7	6.0	0.1
2,3,4,7,8 PCDF	0.0	0.0	3.0	2.7	0.1
1,2,3,7,8 PCDD	0.0	0.0	3.9	7.3	0.0
1,2,3,4,7,8 HxCDF	0.0	0.0	19.1	11.7	0.0
1,2,3,6,7,8 HxCDF	0.1	0.1	7.5	37.9	0.0
2,3,4,6,7,8 HxCDF	0.1	0.0	0.0	7.2	0.7
1,2,3,4,7,8 HxCDD	0.1	0.0	18.8	0.0	0.6
1,2,3,6,7,8 HxCDD	0.0	0.0	19.7	14.5	0.2
1,2,3,7,8,9 HxCDD	0.2	0.1	5.4	15.2	0.5
1,2,3,7,8,9 HxCDF	0.2	0.0	4.4	0.6	0.0
1,2,3,4,6,7,8 HpCDF	0.2	0.1	69.9	76.1	0.0
1,2,3,4,6,7,8 HpCDD	0.1	0.0	400.7	465.4	0.0
1,2,3,4,7,8,9 HpCDF	0.2	0.0	145.0	164.3	0.2
OCDD	1.4	1.4	6738.4	6522.4	0.5
OCDF	0.0	0.0	239.4	276.4	0.9

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	73	83	89	106	111
2,3,7,8 TCDD	80	91	86	85	99
1,2,3,7,8 PCDF	77	90	97	90	74
2,3,4,7,8 PCDF	80	74	98	86	96
1,2,3,7,8 PCDD	83	72	90	74	70
1,2,3,4,7,8 HxCDF	103	84	80	74	88
1,2,3,6,7,8 HxCDF	91	70	82	71	87
2,3,4,6,7,8 HxCDF	82	72	83	71	100
1,2,3,4,7,8 HxCDD	90	71	103	84	116
1,2,3,6,7,8 HxCDD	91	72	105	88	106
1,2,3,7,8,9 HxCDF	94	93	84	91	120
1,2,3,4,6,7,8 HpCDF	97	79	71	84	79
1,2,3,4,6,7,8 HpCDD	91	95	77	103	85
1,2,3,4,7,8,9 HpCDF	93	81	92	97	78
OCDD	78	73	71	82	72

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	0.0	0.0	0.00
PCB-77	17.8	26.6	29.8	38.6	0.15
PCB-123	0.0	0.0	64.2	83.1	0.10
PCB-118	89.3	85.0	492.8	515.6	0.00
PCB-114	0.0	0.0	0.0	0.0	0.00
PCB-105	32.8	35.0	231.2	249.8	0.50
PCB-126	0.0	0.0	19.0	17.3	1.15
PCB-167	1.2	9.7	237.1	252.2	0.00
PCB-156	0.0	0.0	0.0	0.0	0.00
PCB-157	0.0	0.0	0.0	0.0	2.90
PCB-169	0.0	0.0	5.5	12.3	0.55
PCB-189	8.1	6.3	0.0	0.0	0.00

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	74	85	83	105	73
PCB-77	73	87	93	115	89
PCB-123	75	113	72	77	79
PCB-118	81	84	70	73	88
PCB-114	82	90	74	81	83
PCB-105	75	80	82	71	84
PCB-126	79	93	106	83	86
PCB-167	75	94	75	81	75
PCB-156	74	77	72	72	73
PCB-157	76	75	75	71	76
PCB-169	95	78	76	74	88
PCB-189	71	81	79	75	75

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

EZPrep Plus Dioxins & PCBs Sample
Preparation System

Agilent 7010B
TripleQuad

Conclusions
This work shows the feasibility of automation in POPs cleanup. The system can be set up at low cost and is an alternative to other more expensive fully automated clean up equipment. An important feature is also that no dichloromethane is used. Hexane and toluene guarantee an efficient and quick cleanup. Pre-treatment of samples with, e.g., an acid wash, is not necessary when choosing an acidified silica column with sufficient oxidizing capacity.

Combined with the Agilent 7010B TripleQuad GC/MS/MS, the simple, versatile system guarantees same morning or afternoon POPs analysis.

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