

A Quick, High Throughput, Low Solvent Extraction and DCM-Free Sample Clean Up for Same Day POPs Analysis

Rashid Juma, Ruud Addink, Tom Hall
Toxic Report, LLC
Watertown MA

Introduction

- ▶ POPs (PCDD/Fs, PCBs), continue to attract interest around the world due to strict regulations in force in many countries.
- ▶ Rapid sample clean up and analysis needed for many laboratories processing samples.
- ▶ Processing times and solvent use are important considerations.

Manual Clean Up

- ▶ Traditional Soxhlet extraction can take 24-36 h depending on matrix.
- ▶ Labor and time intensive; uses more electrical power than automated options.
- ▶ Manual preparative column chromatography with on-site made columns for cleanup (often prepared in lab).
- ▶ Acidified and basic silica; alumina; carbon.
- ▶ Automated cleanup reduces background and is less time consuming.

Automating Sample Prep

- ▶ Using the FMS Total Prep solution (TRP) for the extraction and extract clean-up of wide variety of matrices is fast, safe and reliable.
- ▶ Products include Pressurized Liquid Extraction (PLE), original PowerPrep, PowerPrep Next Generation, EconoPrep, EP-110 and evaporators for collection tubes and for GC vials.

Pressurized Liquid Extraction



Extraction (1)

- Matrix mixed with Hydromatrix or Ottawa Sand.
- ¹³C labeled surrogate or other standards added.
- Dried samples are transferred to extraction cells.
- Remaining cell volume topped off with Ottawa Sand.

Extraction (2)

- ▶ Extraction cells loaded onto PLE.
- ▶ Cells filled sequentially with 50% DCM in Hexane.
- ▶ Extraction cells pressurized to 1500 PSI.
- ▶ Cells heated to 120 oC and held for 20 min.
- ▶ Cells cooled to approximately ambient temperature.
- ▶ Cells flushed with extraction solvent.
- ▶ Solvent purged from cells.
- ▶ Concentration and exchange to hexane.

EP-110 Clean Up



System Characteristics

- ▶ Control module that pilots valve drive modules connected to a pump and pressure modules responsible for solvent flow in the valve module.
- ▶ Built in computer that does not need a stand-alone pc.
- ▶ Easy programming and software editing provides custom made sequences of events that drive the required solvent at the right place at the right moment.
- ▶ Low pressure (5-30 psi). Flow rates of 5-10mL/min are used.

Columns

- ▶ Silica - PCB-free multilayer ABN silica gel column (mini, high capacity).
- ▶ Carbon – PCB-free carbon/celite column.
- ▶ Alumina – PCB-free basic alumina column.
- ▶ Packed in disposable Teflon tubes; individually sealed in Mylar packaging; production in clean room environment.

Column kits

- ▶ Different column kits:
- ▶ Size of silica determines lipid capacity:
- ▶ Mini – 0.2 g
- ▶ Classical – 0.4 g
- ▶ High Capacity – 5 g
- ▶ XL – 7 g.

EP-110 Features

- ▶ EP-110 fully automated sample load and elution.
- ▶ Different column configuration: silica-alumina-carbon.
- ▶ Uses no DCM, only Hexane and Toluene.
- ▶ Total Clean Up time 30-40 min.
- ▶ Low volumes 110-270 mLs.

Program

- ▶ Condition columns with hexane (step 1).
- ▶ Load sample in hexane onto silica (step 2).
- ▶ Elute silica column with hexane, analytes onto carbon and alumina (step 3).
- ▶ Flush with toluene (step 4).
- ▶ Elute carbon with toluene (step 5). Collect all PCDD/Fs and co-planary PCBs (F1).
- ▶ Elute alumina with toluene (step 6), PCBs fraction collected here (F2).

12 position evaporator 50 mLs

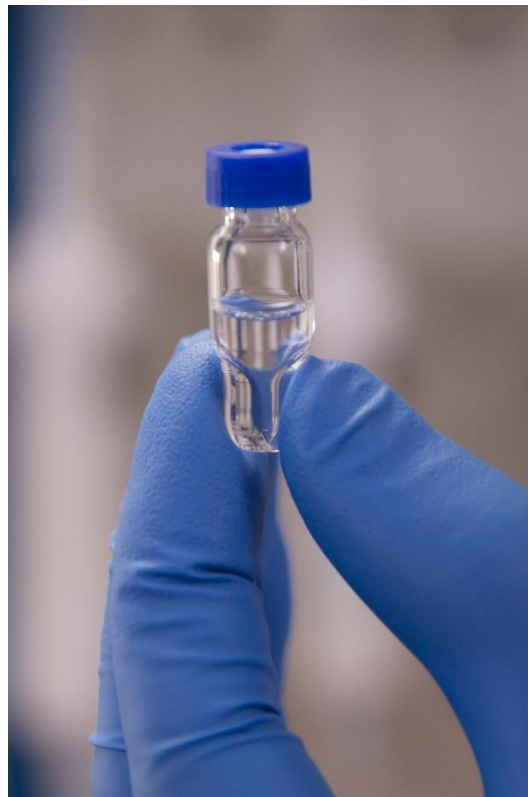


SuperVap Evaporation

- ▶ System pre-heated to 45-60 °C.
- ▶ Samples evaporated at stable T under 5-6 psi nitrogen.
- ▶ 1 mL extract vial transferred to GC vial (can have direct-to-vial feature).
- ▶ Recovery standards added (nonane/dodecane).
- ▶ Extract taken to 10 uL volume with a gentle stream of nitrogen at ambient temperature.

24 position vial evaporator





GC vial

DFS HRGC/HRMS



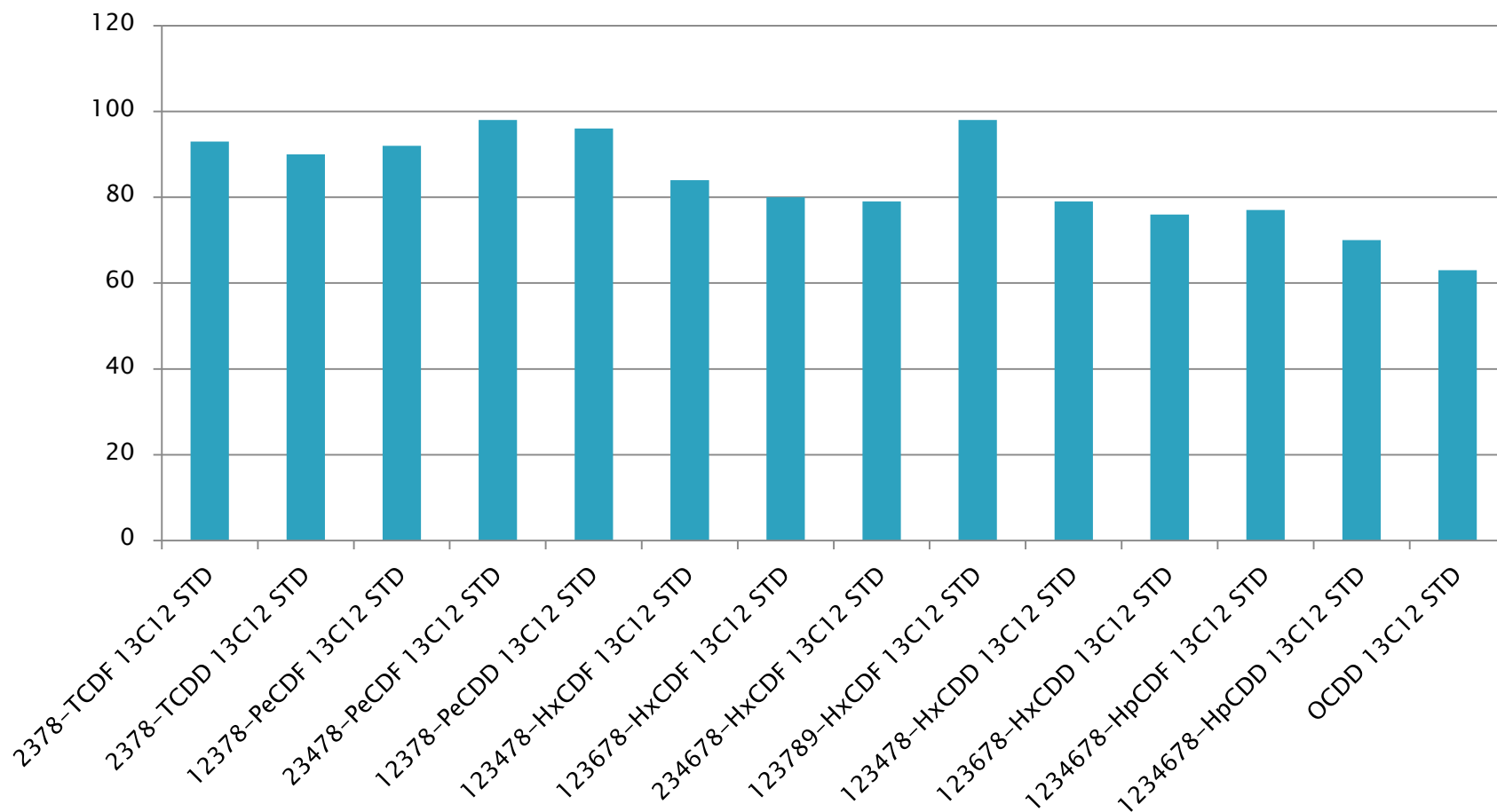
13C Recoveries DD/F Matrices

Compound Name	Sediment 1g	Feed 2g	Egg 8 g	Fish Oil 40 mg	Fatty Acid 2 g	Hexane
2378-TCDF 13C12 STD	78	89	75	69	93	78
2378-TCDD 13C12 STD	92	100	86	96	92	93
12378-PeCDF 13C12 STD	80	91	70	91	97	82
23478-PeCDF 13C12 STD	81	90	70	92	103	80
12378-PeCDD 13C12 STD	91	100	75	105	104	90
123478-HxCDF 13C12 STD	79	95	74	93	92	84
123678-HxCDF 13C12 STD	78	81	76	96	90	85
234678-HxCDF 13C12 STD	83	91	78	87	96	86
123789-HxCDF 13C12 STD	88	93	80	99	90	83
123478-HxCDD 13C12 STD	84	92	77	86	97	88
123678-HxCDD 13C12 STD	73	72	67	83	93	81
1234678-HpCDF 13C12 STD	69	79	68	87	88	69
1234789-HpCDF 13C12 STD	82	71	76	80	92	79
1234678-HpCDD 13C12 STD	87	95	80	98	92	79
OCDD 13C12 STD	70	77	64	77	80	64

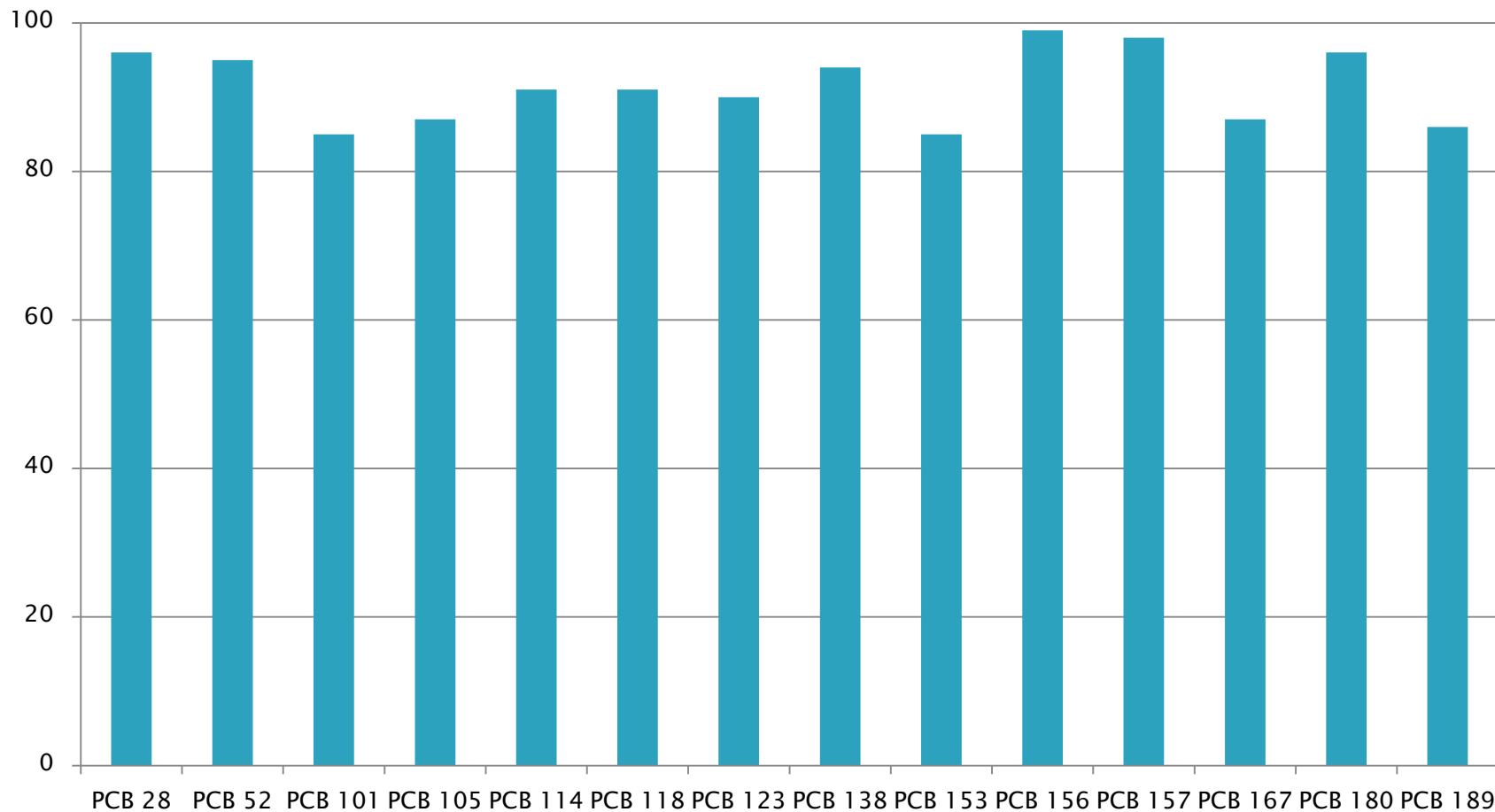
13C PCBs Recoveries Matrices

	Sediment	Fish Oil	Fatty Acid	Fatty Acid	Hexane
	1 g	40mg	1.5 g	2 g	
PCB 28	66	67	68	60	86
PCB 52	68	69	70	71	86
PCB 77	94	90	101	87	86
PCB 81	88	83	95	93	75
PCB 101	77	78	80	77	87
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 126	92	89	88	79	77
PCB 138	75	74	75	72	90
PCB 153	71	71	71	65	84
PCB 156	100	95	98	94	98
PCB 157	98	90	87	92	91
PCB 167	92	89	87	87	86
PCB 169	na	93	106	103	100
PCB 170	99	94	90	98	100
PCB 180	96	85	88	89	88
PCB 189	106	78	94	100	101

13C PCDD/F serum



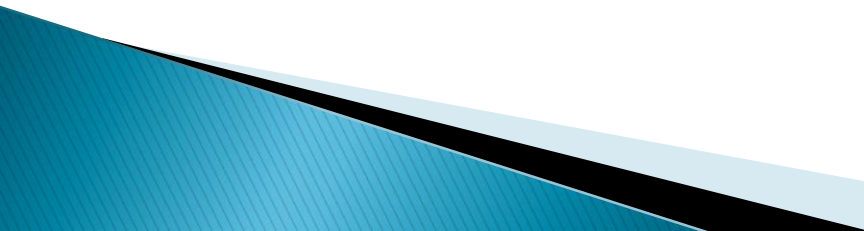
13C PCBs serum



Time from sample to results

	Extraction	Concen- tration	Cleanup	Concen- tration	GC/MS	Total Time
Dioxins & PCBs in Water	45	30	30	60	60	225 min
Dioxins & PCBs in Soil	30	30	30	60	60	210 min
Dioxins & PCBs in Fatty Foods	60	30	40	60	60	250 min
Dioxins & PCBs in Oil	0	0	40	60	60	160 min

Now if you still want to do manual

- ▶ Pre-packaged FMS columns silica-carbon-alumina connected
 - ▶ Reservoir with hexane or toluene at top
 - ▶ Condition with hexane, driven by vacuum pump
 - ▶ Load sample on top silica, elute hexane to carbon and alumina
 - ▶ Disconnect columns and elute carbon and alumina with toluene
 - ▶ Low total solvent 100-200 mLs
 - ▶ Investment very low, need reservoir and vacuum pump
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Data for PCDD/F and PCBs

	hexane	2 g fish oil		hexane	2 g fish oil
PCB_28	67	86	2378-TCDF	101	64
PCB_52	80	84	2378-TCDD	118	60
PCB_101	77	93	12378-PeCDF	92	67
PCB_81	94	76	23478-PeCDF	89	62
PCB_77	94	86	12378-PeCDD	100	71
PCB_123	84	96	123478-HxCDF	82	100
PCB_118	79	96	123678-HxCDF	78	90
PCB_114	77	98	234678-HxCDF	95	104
PCB_105	71	100	123679-HxCDF	83	90
PCB_126	101	89	123478-HxCDD	83	108
PCB_153	95	100	123678-HxCDD	72	114
PCB_138	112	105	1234678-HpCDF	69	72
PCB_167	79	103	1234789-HpCDF	89	109
PCB_156	81	103	1234678-HpCDD	86	108
PCB_157	73	95	OCDD	70	91
PCB_169	101	108			
PCB_180	95	110			
PCB_170	103	115			
PCB_189	99	113			

Conclusions

- ▶ PLE and EP-110 with silica-carbon-alumina configuration deliver very good recoveries for various matrices.
- ▶ EP-110 is Green option with low power use.
- ▶ Clean up step time between 20 and 40 min.
- ▶ EP-110 uses no DCM.
- ▶ Low solvent use 110-270 mLs.
- ▶ Total time from sample till data between 2.5-4h.
- ▶ PLE and EP-110 can be purchased in one system.
- ▶ Manual clean up can be good alternative with low investment and pre-packaged columns.