Automated Sample Processing of POPs with No Dichloromethane and Low Volume Solvent Use

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Introduction

- POPs (PCDD/Fs, PCBs) continue to attract interest around the world due to strict regulations in force in many countries (Stockholm Convention).

- 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.

- Acidified silica; alumina; carbon.

- Automated cleanup reduces background and is less time consuming.

Automating Sample Prep

- Automated Pressurized Liquid Extraction (PLE) for sample extraction is fast (60 min), efficient (120 ^{o}C, 1500 psi), green (less power), reliable (long track record).

- Solid Phase Extraction for serum and water is fully automated, fast (less time than manual), low background (closed system), versatile for many cartridges and sample sizes.

- Low Solvent clean up system: fast (40-60 min), no DCM used, low solvent (150-250 mLs).

EP-110 Clean Up

Touch Screen Control

Real Time Display of Program Steps

On line evaporator, starts during collection

Up to 4 modules, 8 samples parallel

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. Nitrogen valve enables push through sample lines (optional).
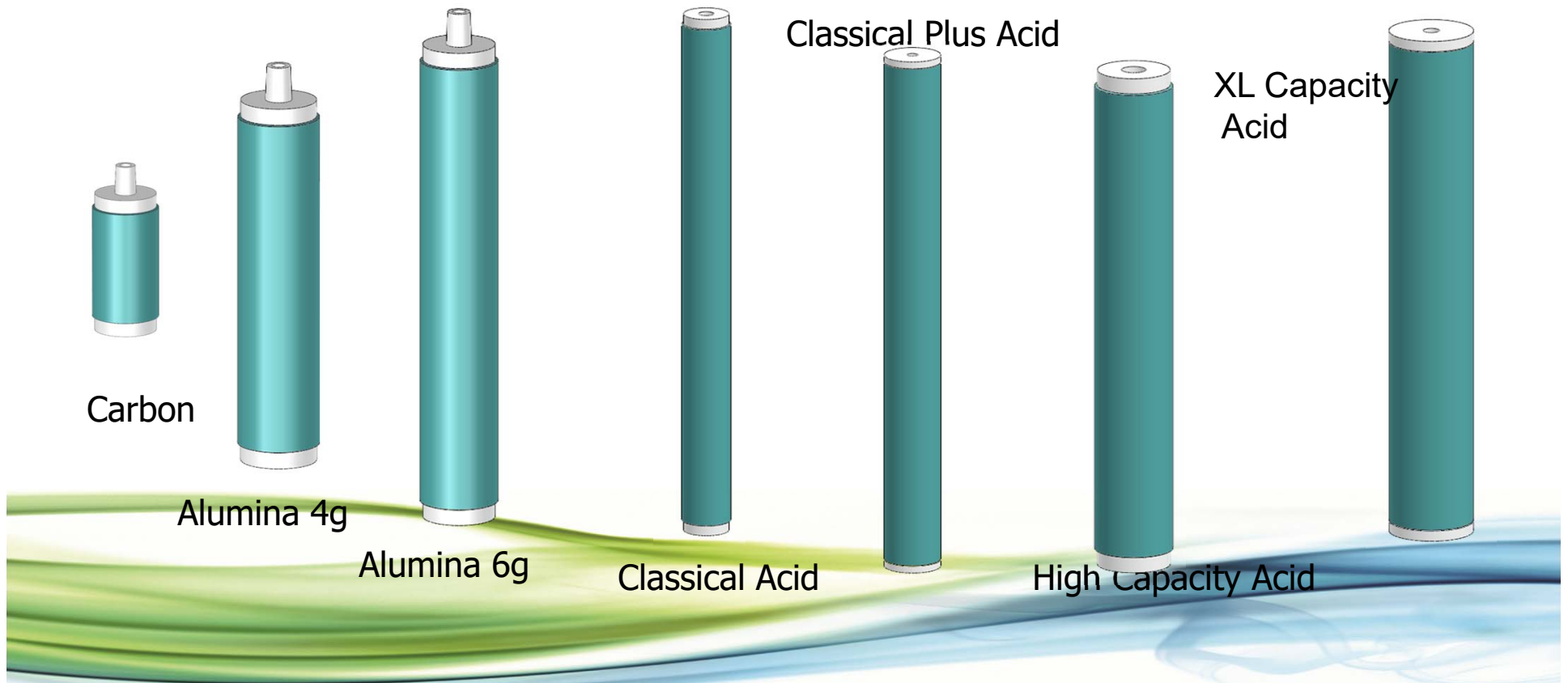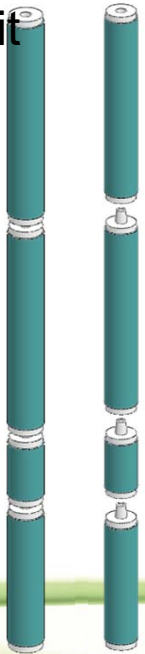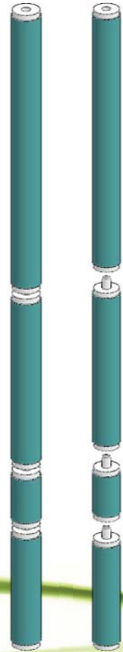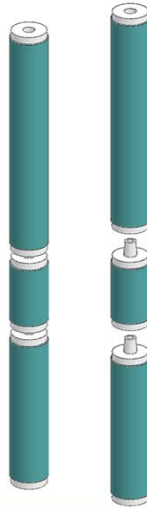
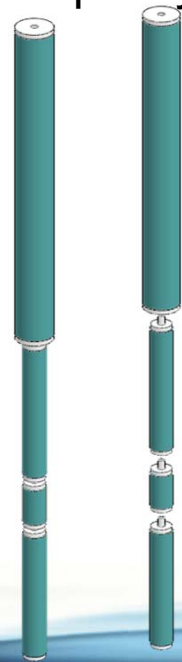# Columns (2)

Classical Kit

Classical Plus Kit

Mini Kit

High Capacity Kit

XL Capacity Kit

Columns

- Silica - PCB-free acidic silica gel column (5 different capacities).

- Carbon – PCB-free carbon/celite column.

- Alumina – PCB-free basic alumina column.

- Packed in disposable Teflon tubes; individually sealed packaging; production in clean room environment.

EP-110 Features

- EP-110 fully automated sample load and elution.

- Load Sample Extracts in hexane directly onto the system with no Manual Pretreatment

- Easy to perform QC sample simultaneously with a Real sample.
    - 2 samples per module

- Different column configuration: silica-carbon-alumina.

- Uses no DCM, only Hexane and Toluene.

- Total Clean Up time 40-60 min.

- Low volumes 150-250 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).

SuperVap 12 50ml Concentration/Evaporation

SuperVap Concentration/Evaporation

- System pre-heated to 55-60 ºC.

- 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.

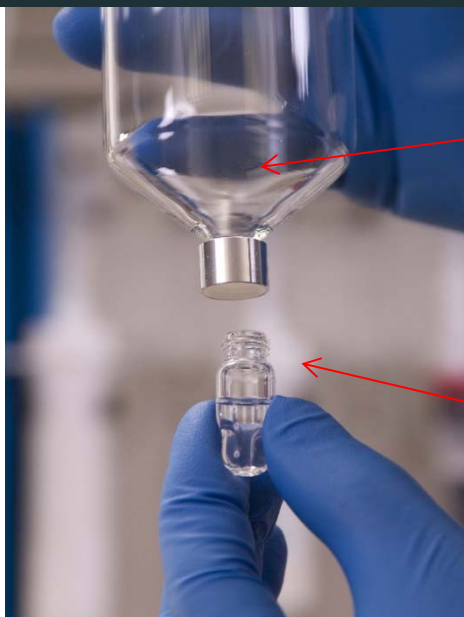# Direct to GC Vial

Glass Evaporation tube

GC vial

DFS HRGC/HRMS
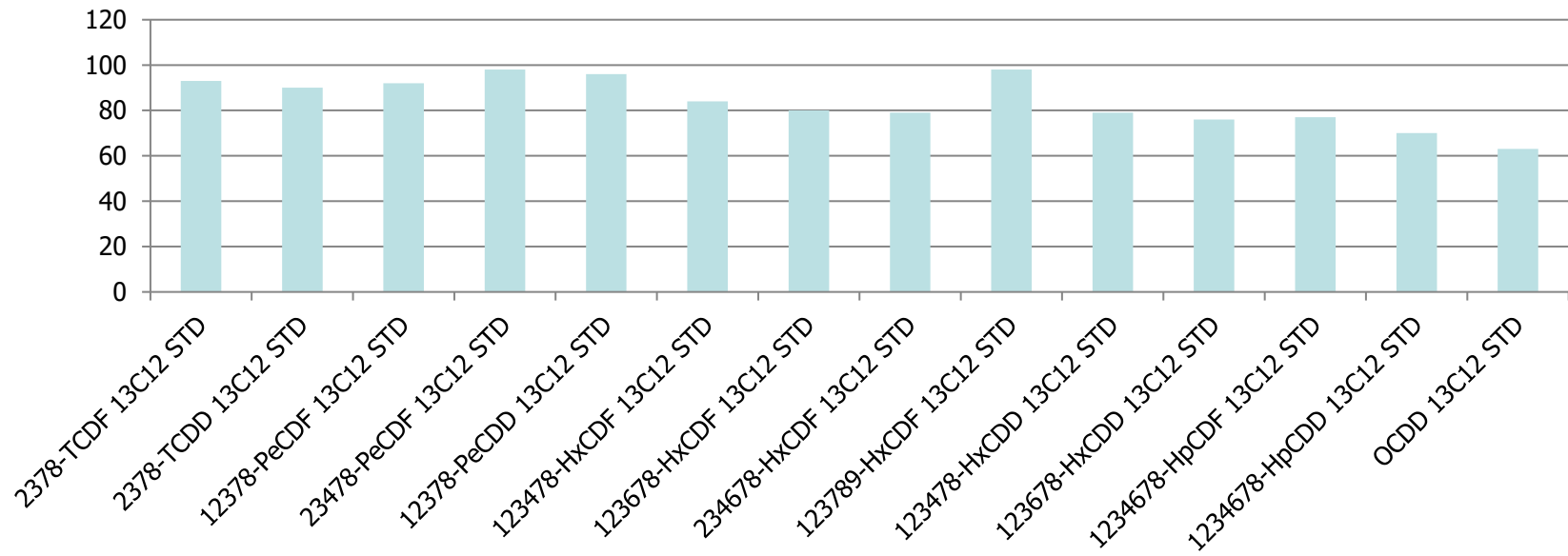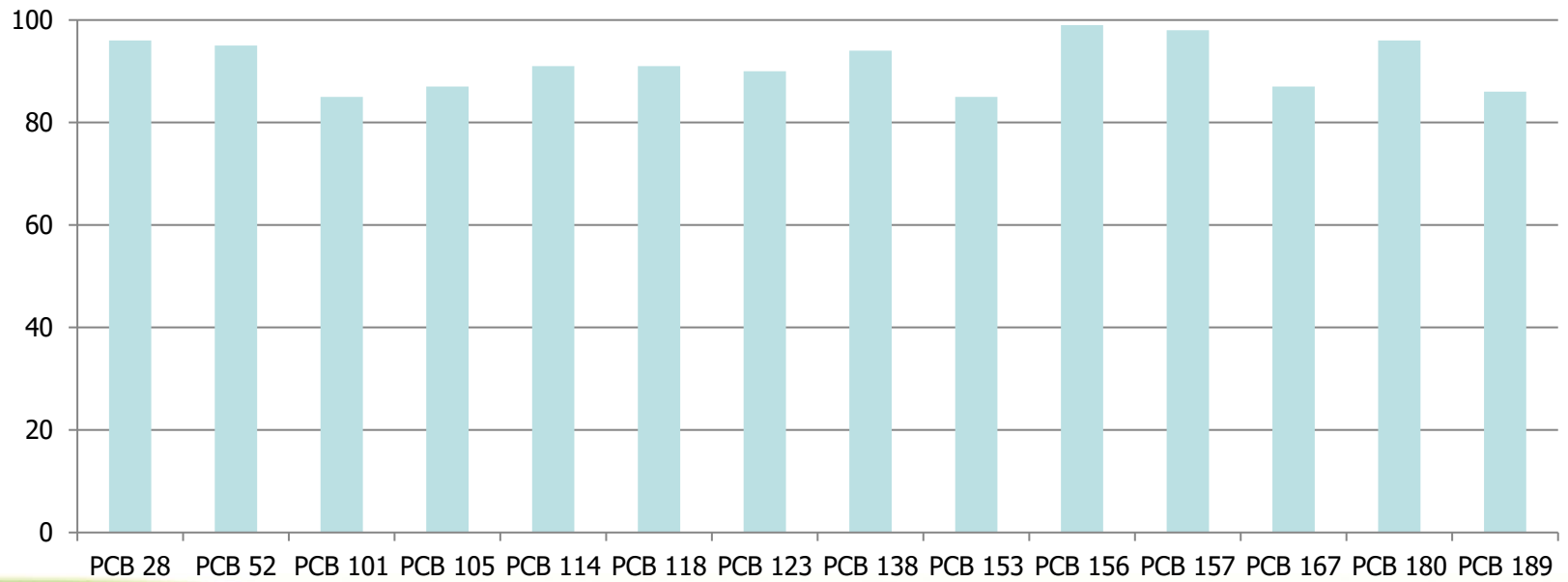
Mini Kit Data

- Used automated SPE for serum extraction

- Clean up with mini silica, carbon, mini alumina

- Total time for clean up 40 min

- Total solvent volume 150 mLs

- No DCM

High Capacity Kit Data

- Used automated PLE for egg, feed, soil extraction

- Clean up with high capacity silica, carbon, alumina

- Total time for clean up 60 min

- Total solvent volume 250 mLs

- No DCM

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

	Sediment
	1g
BDE-28	67
BDE-47	71
BDE-99	81
BDE-100	80
BDE-153	79
BDE-154	77
BDE-183	80
BDE-209	60

Time from sample to results

	Extraction	Concentration	Cleanup	Concentration	GC/MS	Total Time
Dioxins & PCBs in Serum	45	30	40	60	60	235 min
Dioxins & PCBs in Soil	60	30	60	60	60	270 min
Dioxins & PCBs in Fatty Foods	60	30	60	60	60	270 min
Dioxins & PCBs in Oil	0	0	60	60	60	180 min

Conclusions

- **PLE/SPE and EP-110 with silica-carbon-alumina configuration deliver very good recoveries for various matrices.**

- **EP-110 is Green option with low solvent and power use.**

- **Clean up step time between 40 and 60 min.**

- **EP-110 uses no DCM.**

- **Low solvent use 150-250 mLs.**

- **EP-110 delivers the extract directly to the SuperVap**
  - **SuperVap concentrates directly into a GC Vial**

- **Total time from sample to data between 3-4.5 h.**

- **PLE/SPE, SuperVap and EP-110 can be purchased in one system.**

Questions

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