

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

Ruud Addink and Tom Hall  
Fluid Management Systems  
Billerica, MA



# 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 °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).



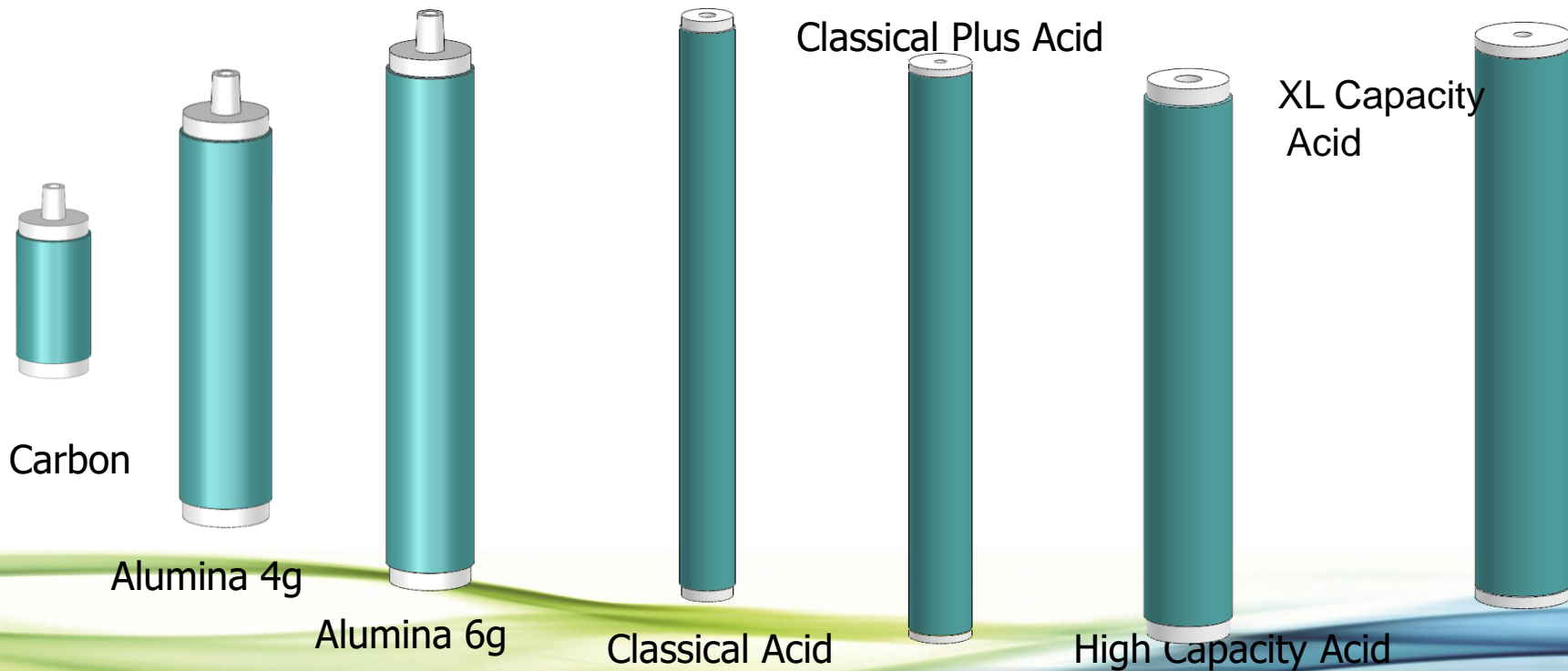
# EP-110 Clean Up



# System Characteristics

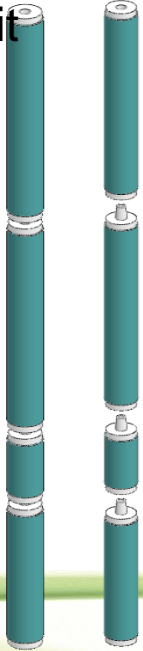
- Control module that pilots valve drive modules connected to a pump and pressure modules responsible for solvent flow in the valve module.
- Built in computer that does not need a stand-alone pc.
- Easy programming and software editing provides custom made sequences of events that drive the required 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 (1)

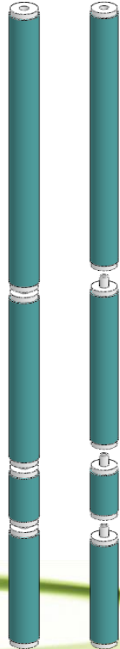


# Columns (2)

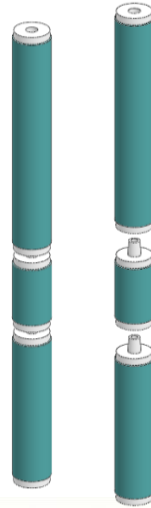
Classical  
Kit



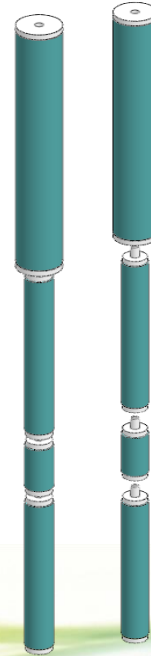
Classical Plus  
Kit



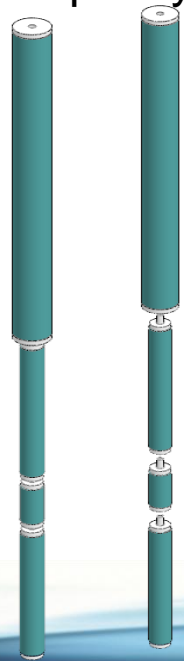
Mini Kit



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

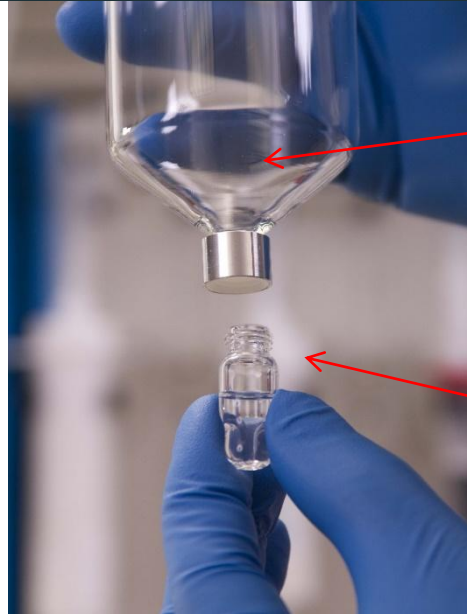


- System pre-heated to 55-60 °C.
- Samples evaporated at stable T under 5-6 psi nitrogen.
- 1 mL extract vial transferred to GC vial (can have direct-to-vial feature).
- Recovery standards added (nonane/dodecane).
- Extract taken to 10 uL volume with a gentle stream of nitrogen at ambient temperature.

# SuperVap 24 Vial Concentrator/Evaporator



# Direct to GC Vial



**Glass Evaporation tube**

**GC vial**





# DFS HRGC/HRMS

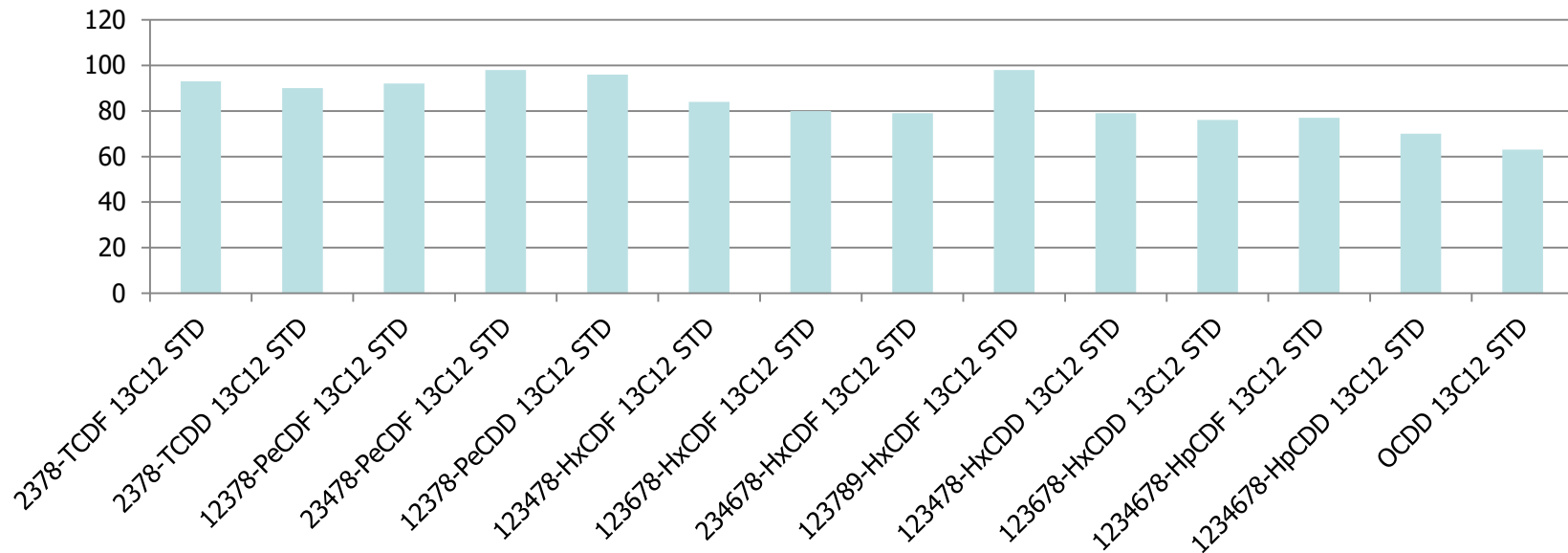




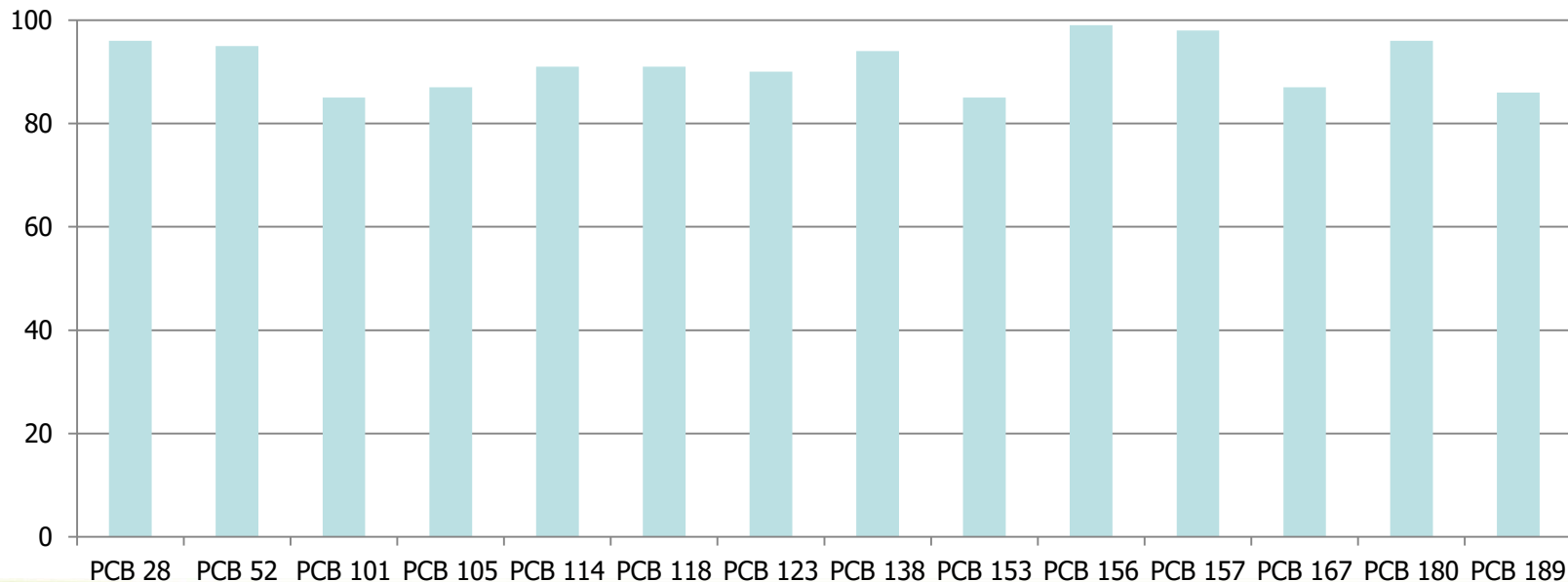
# Mini Kit Data

- 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

# 13C PCDD/F serum



# 13C PCBs serum



# 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
<b>2378-TCDF 13C12 STD</b>	78	89	75	69	93	78
<b>2378-TCDD 13C12 STD</b>	92	100	86	96	92	93
<b>12378-PeCDF 13C12 STD</b>	80	91	70	91	97	82
<b>23478-PeCDF 13C12 STD</b>	81	90	70	92	103	80
<b>12378-PeCDD 13C12 STD</b>	91	100	75	105	104	90
<b>123478-HxCDF 13C12 STD</b>	79	95	74	93	92	84
<b>123678-HxCDF 13C12 STD</b>	78	81	76	96	90	85
<b>234678-HxCDF 13C12 STD</b>	83	91	78	87	96	86
<b>123789-HxCDF 13C12 STD</b>	88	93	80	99	90	83
<b>123478-HxCDD 13C12 STD</b>	84	92	77	86	97	88
<b>123678-HxCDD 13C12 STD</b>	73	72	67	83	93	81
<b>1234678-HpCDF 13C12 STD</b>	69	79	68	87	88	69
<b>1234789-HpCDF 13C12 STD</b>	82	71	76	80	92	79
<b>1234678-HpCDD 13C12 STD</b>	87	95	80	98	92	79
<b>OCDD 13C12 STD</b>	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	
<b>PCB 28</b>	66	67	68	60	86
<b>PCB 52</b>	68	69	70	71	86
<b>PCB 77</b>	94	90	101	87	86
<b>PCB 81</b>	88	83	95	93	75
<b>PCB 101</b>	77	78	80	77	87
<b>PCB 105</b>	98	112	90	88	95
<b>PCB 114</b>	108	109	87	89	97
<b>PCB 118</b>	92	110	88	90	89
<b>PCB 123</b>	112	115	82	86	98
<b>PCB 126</b>	92	89	88	79	77
<b>PCB 138</b>	75	74	75	72	90
<b>PCB 153</b>	71	71	71	65	84
<b>PCB 156</b>	100	95	98	94	98
<b>PCB 157</b>	98	90	87	92	91
<b>PCB 167</b>	92	89	87	87	86
<b>PCB 169</b>	na	93	106	103	100
<b>PCB 170</b>	99	94	90	98	100
<b>PCB 180</b>	96	85	88	89	88
<b>PCB 189</b>	106	78	94	100	101

# 13C PBDEs Recoveries Sediment

	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
<b>Dioxins &amp; PCBs in Serum</b>	45	30	40	60	60	235 min
<b>Dioxins &amp; PCBs in Soil</b>	60	30	60	60	60	270 min
<b>Dioxins &amp; PCBs in Fatty Foods</b>	60	30	60	60	60	270 min
<b>Dioxins &amp; PCBs in Oil</b>	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

Contact us at:  
[raddink@fms-inc.com](mailto:raddink@fms-inc.com) (Ruud Addink)  
[thall@fms-inc.com](mailto:thall@fms-inc.com) (Tom Hall)

