

# **A High Throughput, Low Cost and Green Approach to Automated Extraction, Clean Up, and Concentration for Same Day POPs Analysis**

Philip Bassignani  
Fluid Management Systems  
Watertown MA USA



- Stockholm Convention on Persistent Organics Pollutants 2001.
- Compounds of interest: polychlorinated biphenyls (PCBs) and polychlorinated dibenzo-p-dioxins (PCDDs), and furans (PCDFs).
- Known toxicity.
- Strict environmental regulations in force in most countries.



# Health Effects

- Endocrine disruptors.
- Immune system.
- Nervous system.
- Reproductive functions.
- Carcinogenic.
- Chloracne.
- Main exposure (> 90%) is through dietary intake: meat, dairy, fish.



# Sample Processing

- Analysis of various matrices for PCDD/Fs and PCBs using extraction and clean up.
- Soxhlet extraction (typically up to 24-36 h).
- Preparative multi column chromatography involving various solvents and steps.
- Can include acid-base-neutral silica, pure acidified silica, alumina, florisil and carbon columns. Use of 22% or 44% H<sub>2</sub>SO<sub>4</sub> acid mixed with silica; 33% NaOH mixed with silica.





# Automation

- Advantages of automated sample prep are:
- Reduced time: Pressurized Liquid Extraction (PLE) takes 60 min start-to-finish (50/50 DCM/hexane, 20 min at 120 oC, 1500 psi). Compare Soxhlet up to 36 h.
- Reduced cost: less labor involved, shorter turnover time per sample, less electricity use for PLE than Soxhlet.
- Reduced volume: less solvent used.

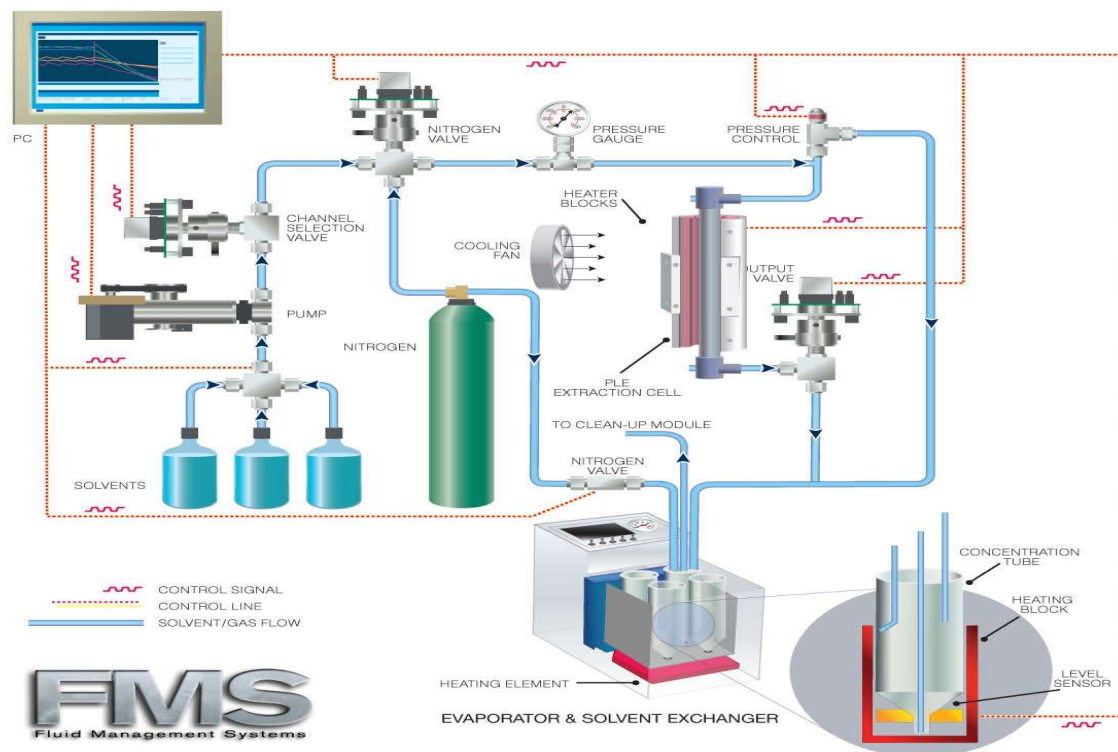


# TotalEconoPrep



# Pressurized Liquid Extraction

**PLE® Fast Extraction & Concentration System**



# Extraction Procedure

- 1 g sample mixed with Hydromatrix™ to dry, transferred to extraction cells
- Spiked with  $^{13}\text{C}$  PCDD/Fs and PCBs standards.
- Void volume filled with Hydromatrix™.
- Sample Cells filled with 50% mixture Hexane/Methylene Chloride.
- Cells pressurized to 1500 PSI and heated to 120 °C.
- Temperature held for 20 minutes.
- Extraction cells cooled, flushed with solvent (50% cell volume), and nitrogen; collected in 250 mL tubes.





# 6 position evaporator



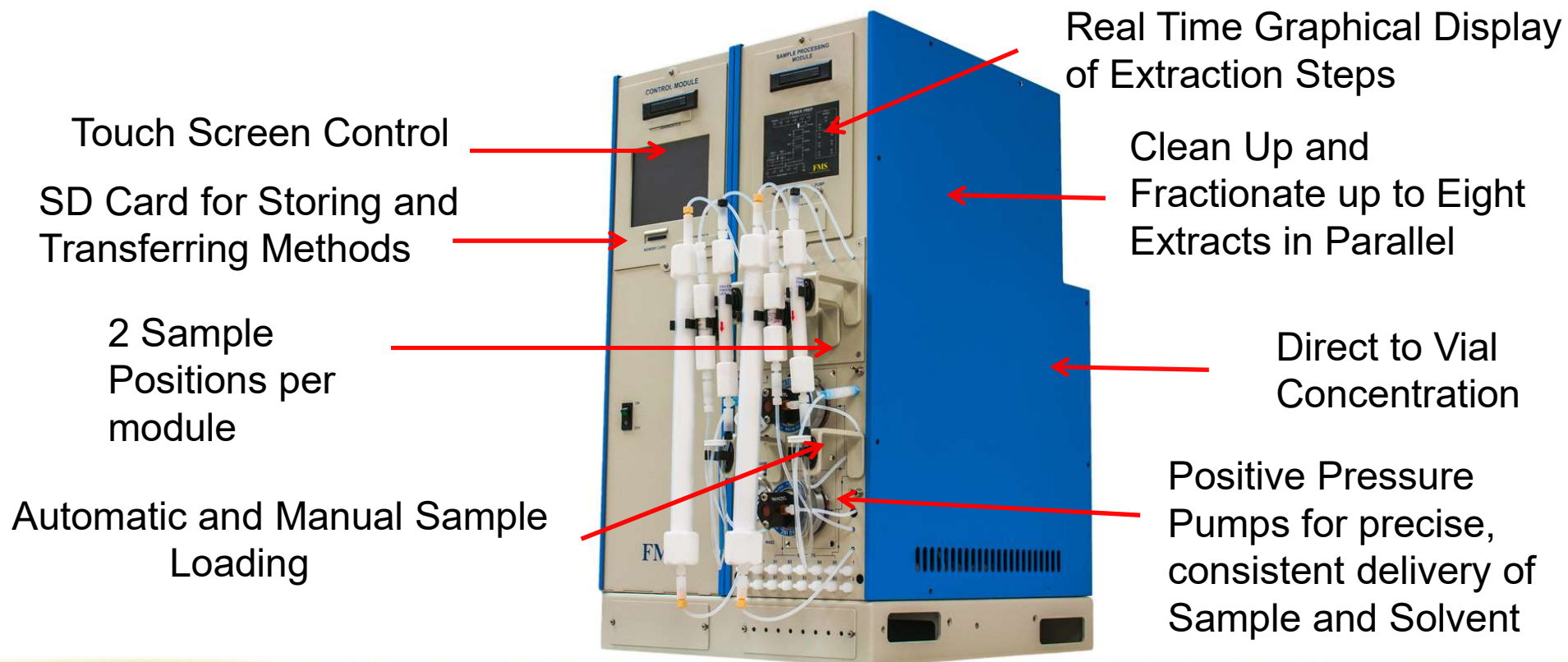


# SuperVap Evaporation

- System pre-heated to 45-60 °C.
- Extracts evaporated at stable temperature under 5-6 psi nitrogen.
- Solvent exchange with hexane to eliminate dichloromethane.
- Dichloromethane would interfere with subsequent sample clean up.
- Extracts reduced to a few mLs.



# EconoPrep®



# Expandable and Modular

- Low Cost POPs analysis
- Runs 2 samples per Module In parallel
- Expandable up to 4 Modules
- Run up to 8 samples in Parallel
- Run up to 8 samples in 30 to 40 minutes





# Expandable

**EconoPrep 4**  
Capable of running  
8 Samples in Parallel



# Columns

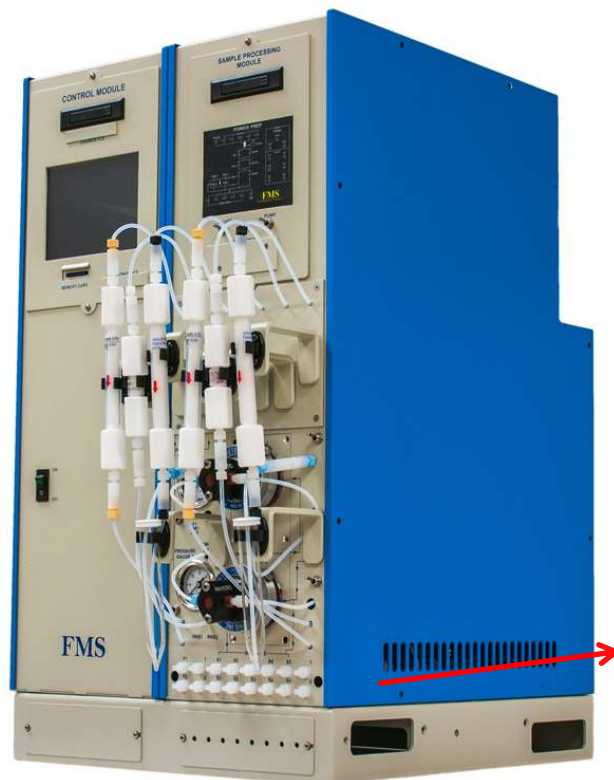
- Silica - PCB-free multilayer ABN silica gel column (sizes half, classical, high capacity, XL).
- Alumina – PCB-free basic alumina column (mini and regular size).
- Carbon – PCB-free carbon/celite column.
- Packed in disposable Teflon tubes; individually sealed in Mylar packaging; production in clean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





# EconoPrep Mini

Total run time  
20 minutes



mini  
Up to 0.2 gm  
lipid

140 mL  
Total  
Solvent  
PCB / Dioxin  
Fractions

# EconoPrep Classic

Total run time  
30 minutes



Classical  
Up to 0.4 gm  
lipid

225 mL  
Total  
Solvent  
PCB / Dioxin  
Fractions

# EconoPrep HC

Total run time  
40 minutes



High  
Capacity  
Up to 5 gms  
lipid

335mL  
Total  
Solvent  
PCB / Dioxin  
Fractions



# Procedure (1)

- Columns are conditioned with hexane.
- Samples are loaded onto silica column in hexane.
- Silica column is eluted with hexane, analytes go onto alumina.
- Alumina is eluted with methylene chloride; mono- and di-ortho PCBs are collected in Fraction 1.
- Co-planary PCBs and PCDD/Fs are bound on carbon column.
- Carbon is eluted in reverse (upward) direction with toluene collecting Fraction 2 (co-PCBs, PCDD/F).

## Procedure (2)

- Hexane, DCM and toluene used as solvents.
- Solvent use between 150-335 mL depending on application.
- Original 25-step PowerPrep program used ~ 800 mLs solvent.
- For food application two fractions: PCB fraction (mono-, di-ortho) and PCDD/F/co-PCB fraction.
- EconoPrep with different plumbing allows for collecting all PCBs in one fraction and all PCDD/F in one fraction; ideal for environmental samples. Depends on country/regulation.





# 24 position vial evaporator





GC vial

# DFS HRGC/HRMS



# Classical PCB data

Compound Name			Olive Oil	Olive Oil
			Module 1 Position 1	Module 1 Position 2
344'5-Te-PCB 13C STD			78	100
33'44'-Te-PCB 13C STD			82	64
2'344'5-Pe-PCB 13C STD			82	107
23'44'5-Pe-PCB 13C STD			87	118
2344'5-Pe-PCB 13C STD			78	106
233'44'-Pe-PCB 13C STD			69	99
33'44'5-Pe-PCB 13C STD			82	105
23'44'55'-Hx-PCB 13C STD			95	91
233'44'5-Hx-PCB 13C STD			96	103
233'44'5'-Hx-PCB 13C STD			88	90
33'44'55'-Hx-PCB 13C STD			98	90
233'44'55'-Hp-PCB 13C STD			91	92

Classical  
Up to 0.4 gm  
lipid

285 mL  
Total  
Solvent  
PCB / Dioxin  
Fractions



# Classical PCDD/F data

Compound Name		Olive Oil	Olive Oil
		Module 1 Position 1	Module 1 Position 2
2378-TCDF 13C12 STD		102	99
2378-TCDD 13C12 STD		101	103
12378-PeCDF 13C12 STD		100	94
23478-PeCDF 13C12 STD		103	109
12378-PeCDD 13C12 STD		102	106
123478-HxCDF 13C12 STD		101	94
123678-HxCDF 13C12 STD		109	99
234678-HxCDF 13C12 STD		99	98
123789-HxCDF 13C12 STD		95	97
123478-HxCDD 13C12 STD		80	95
123678-HxCDD 13C12 STD		91	95
1234678-HpCDF 13C12 STD		94	99
1234789-HpCDF 13C12 STD		103	85
1234678-HpCDD 13C12 STD		103	78
OCDD 13C12 STD		90	77

Classical  
Up to 0.4 gm  
lipid

285 mL  
Total  
Solvent  
PCB / Dioxin  
Fractions



# High Capacity PCB data

	Soil	Milk	Olive Oil	Red Palm Canola
PCB_77	68%	66%	92%	91%
PCB_81	70%	54%	90%	90%
PCB_105	86%	59%	93%	88%
PCB_114	61%	54%	93%	88%
PCB_118	78%	75%	90%	86%
PCB_123	76%	71%	94%	86%
PCB_126	80%	70%	87%	81%
PCB_156	67%	90%	98%	96%
PCB_157	65%	89%	99%	94%
PCB_167	71%	109%	97%	94%
PCB_169	89%	111%	104%	96%
PCB_170	76%	93%		95%
PCB_180	65%	79%	97%	92%
PCB_189	72%	93%	91%	91%

High  
Capacity  
Up to 5 gm  
lipid

335 mL  
Total  
Solvent  
PCB / Dioxin  
Fractions



# High Capacity PCDD/F data

	<b>Animal Feed</b>	<b>Peanut Butter</b>	<b>Corn Oil</b>	<b>Olive Oil</b>
2378-TCDF 13C12 STD	78%	73%	61%	66%
2378-TCDD 13C12 STD	78%	73%	63%	66%
12378-PeCDF 13C12 STD	78%	78%	66%	69%
23478-PeCDF 13C12 STD	78%	73%	63%	65%
12378-PeCDD 13C12 STD	78%	77%	65%	72%
123478-HxCDF 13C12 STD	97%	74%	90%	78%
123678-HxCDF 13C12 STD	96%	74%	85%	81%
234678-HxCDF 13C12 STD	95%	64%	81%	86%
123789-HxCDF 13C12 STD	98%	80%	88%	84%
123478-HxCDD 13C12 STD	96%	68%	84%	89%
123678-HxCDD 13C12 STD	95%	64%	76%	83%
1234678-HpCDF 13C12 STD	90%	63%	85%	83%
1234789-HpCDF 13C12 STD	94%	66%	90%	87%
1234678-HpCDD 13C12 STD	91%	65%	80%	81%
OCDD 13C12 STD	80%	62%	84%	78%

High  
Capacity  
Up to 5 gm  
lipid

335 mL  
Total  
Solvent  
PCB / Dioxin  
Fractions

# Conclusions (1)

- Total Econo Prep delivers cheap, efficient, and quick solution for sample extraction and clean up.
- Clean up part is flexible depending on sample requirements: mini, classical or HC.
- Excellent recoveries for both PCDD/Fs and PCBs for various complex matrices.
- Solvent use limited to 140-335 mLs depending on application.



## Conclusions (2)

- Extraction and clean up can be done in 3-4 hours.
- Different plumbing available for food (PCBs in two fractions) and environmental (all PCBs together).
- Same day sample processing and analysis (HRGC/HRMS): can be easily done in one day.

