

Automated High Throughput Dioxin and PCBs Sample Cleanup

Ruud Addink and Tom Hall
Fluid Management Systems
Billerica MA USA



Introduction

- Stockholm Convention on Persistent Organics Pollutants 2004.
- Compounds of interest: polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs), biphenyls (PCBs) and poly brominated diphenyl ethers (PBDEs).
- Known toxicity.
- Strict environmental regulations in force in most countries.
- US EPA and EU methods and regulations; other countries have their own.



Challenges of POPs Sample Prep

- Labor intensive, prone to error
- Compliance with regulatory procedures and accreditation (lengthy method validation)
- Strict QA/QC requirements
- Sample matrix complexity
- Native background and interferences (sometimes orders of magnitude higher than analytes)
- Pico-/femto-gram analyses require ultra-pure extract and excellent instrument sensitivity



Manual Sample Prep

Advantages of Manual Sample Prep

- Flexibility
- Low initial Capital equipment Cost
- Easier to implement
- No electronics or mechanical failure
No down time due to system failure
- No service contract cost

Disadvantages of Manual Sample Prep

- Human Error
- Less Efficiency
- Increased workload
- Inconsistency
- Risk of Cross contamination
- Human Exposure to Chemical
- Lack of Traceability
- Difficult to Scale up

Automated Sample Prep

Advantages Automated Sample Prep

- Efficiency & Speed
- Accuracy & Consistency
- Repeatability & Reproducibility
- Reduction of Manual Labor
- Documentation & Traceability
- Less exposure to Hazardous
- Cleaner Background Interference
- Simpler QA/QC & Accreditation

Disadvantages Automated Sample Prep

- High Initial Cost
- Maintenance & Service contract Cost
- Technical Knowledge required
- System Limited Flexibility
- Down time due to failure
- Sample size limitation



Design of the Ideal Sample Clean-up

Combining The Best Features of Manual & Automated

Advantages of Manual Sample Prep

- Flexibility
- Low initial Capital equipment Cost
- Easier to implement
- No electronics or mechanical failure
No down time due to system failure
- No service contract cost



Advantages Automated Sample Prep

- Efficiency & Speed
- Accuracy & Consistency
- Repeatability & Reproducibility
- Reduction of Manual Labor
- Documentation & Traceability
- Less exposure to Hazardous
- Low Background Interferences
- Simpler QA/QC & Accreditation

Features:

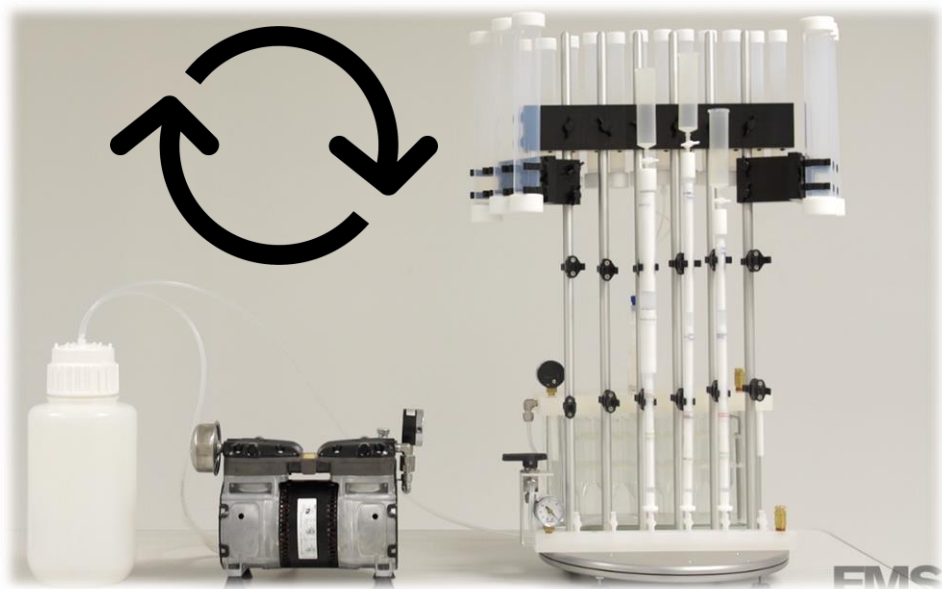
- Rapid Turn Around Time: 45 to 60 Minutes for 6 Samples
- Simple to operate Resemble Manual Sample Clean-up
- Cleaner Background Interferences: Closed Loop System
- Quality Results: Certified Pre-packaged Columns
- Green Technology: Low solvent and power use
- QA/QC & Accreditation Requirements: Easy to manage
- Reliable Little electronic or Electro-Mechanical to fail
- Affordable Automation: Low cost

Combine Best Features (EzPrep Family)

Combine both features:

- Fast: 45-60 min
- Simple to run, no computerized instrumentation
- Closed loop system with clean background, low detection limits
- Use certified pre-packaged columns
- Green technology, uses multi-pump to do cleanup
- Low solvent volumes
- Economical column kits, five choices of low fat to high fat column kits
- Low capital equipment cost
- Little electronics or mechanical equipment to fail
- Little cleaning and no cross-contamination
- Minimal downtime

Design of EzPrep Using Vacuum Pump



Sample Concentration Using FMS SuperVap



Cycle Time EzPrep

Processing 6 Samples

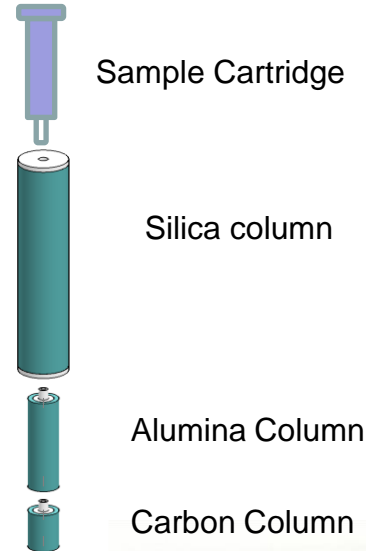
	<u>Automated</u>	<u>Manual</u>
• <u>Set up time:</u>		
• Assemble & Install acidic silica-carbon-alumina columns on column rack		
• Place samples cartridges on top of acidic silica columns , Add Solvents to solvent reserve		- 20 min
• <u>Program 1:</u>		
elute hexane through all three columns ; apply nitrogen to push hexane onto the columns to waste	- 20 min	
• Disassemble the column set, install carbon and alumina columns on top of manifold		-10 min
• <u>Program 2:</u>		
• Dispense Toluene through alumina & Carbon and collect PCBs & Dioxins	-10 min	

Total Cycle Time 60 min

FMS Certified Snap-In columns:

- Easy to connect
- NO fittings
- Designed for easier flow
- Different size for different fat capacity from 0.2 up to 7 gm of Fat

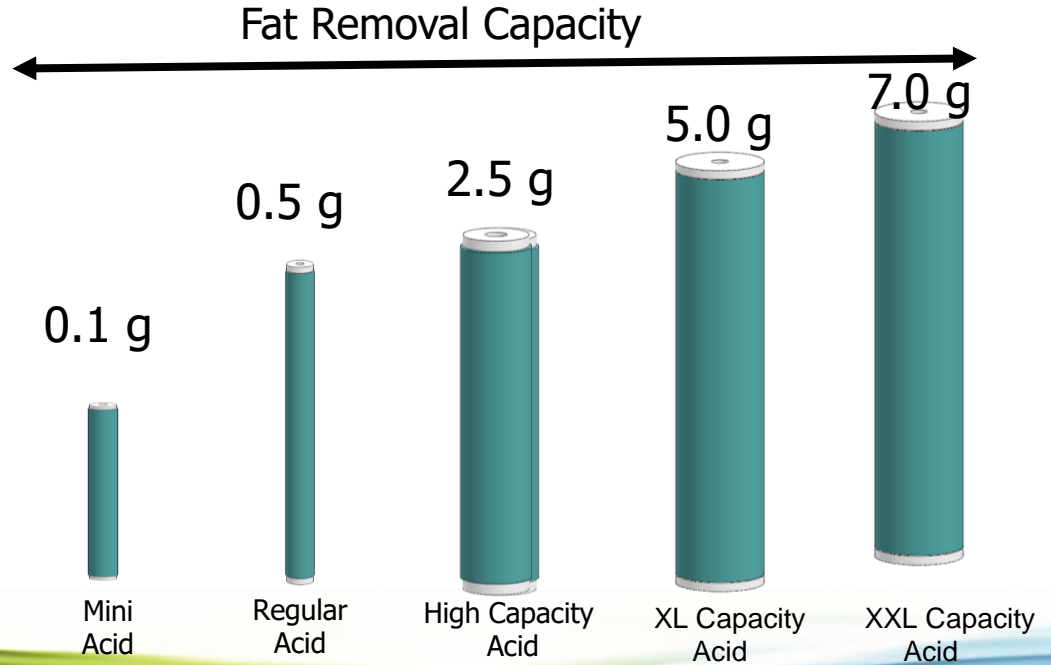
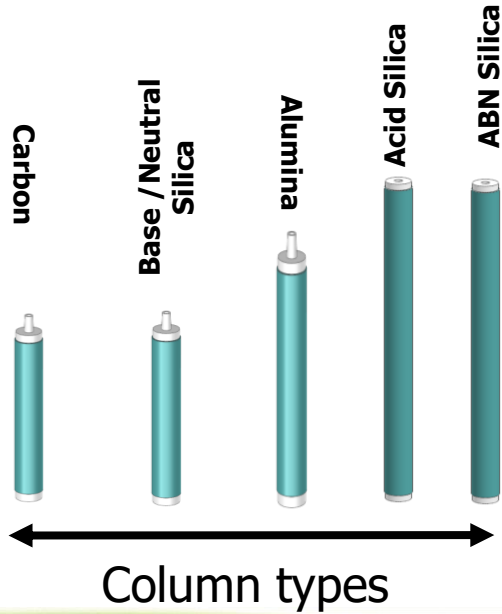
Disassembled



Assembled



Columns/ Fat Removal Capacity



Features:

- Programmable Flow rate and Volume
- Pressure indicator and over pressure alarm
- Real time read-out for dispensed volume and pressure
- Ability to select from 1 to six samples
- Can accommodate up to 4 solvents
- Economical & less expensive automation

Automated EzPrep/ + Sample Clean-up

Benefits:

- Rapid Turn Around Time: 45 to 60 Minutes for 6 Samples
- Simple Programming: Just Select Solvent, Set Flow & Volume
- High Throughput: Process up to 48 samples per day
- Cleaner Background Interferences: Closed Loop System
- Quality Results: Certified Pre-packaged Columns
- Green Technology: Low solvent and power use
- QA/QC & Accreditation Requirements: Easy to manage
- Reliable: Minimal Electronics & Electro-Mechanical

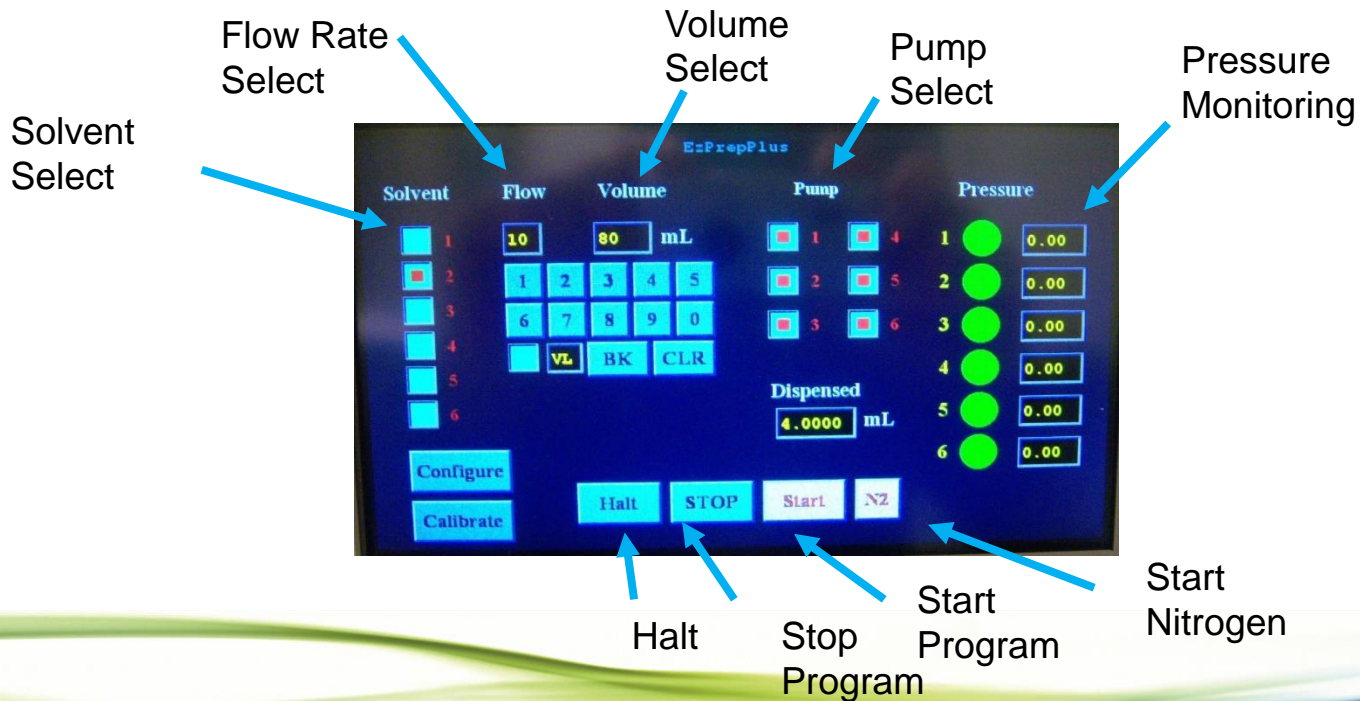
Attributes EZPrep/+

- **Closed loop system, eliminates background contaminants & exposure to chemicals**
- **Optimized for solvent reduction while obtaining highest possible recoveries**
- **Certified disposable Columns with guaranty Low contaminants background and Excellent Recoveries**
- **Quick connect SNAP columns simplifies system set up**
- **Multi pump Solvent Delivery system brings convenient automated solvent selection & dispense with controllable flow & volume**
- **EzPrep/+ designed with Minimum number of electronics and Electromechanical valve to lower cost and simplify the maintenance**

Automated EZprep/+

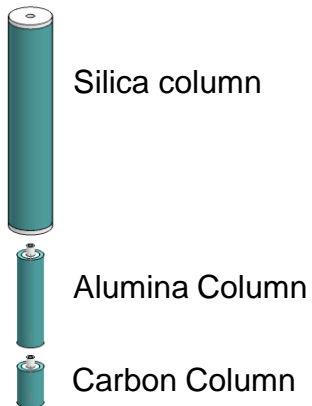


EzPrep /+ Control Panel



How It Works System set up

Unpack column



Assemble Snap Columns



Add Samples



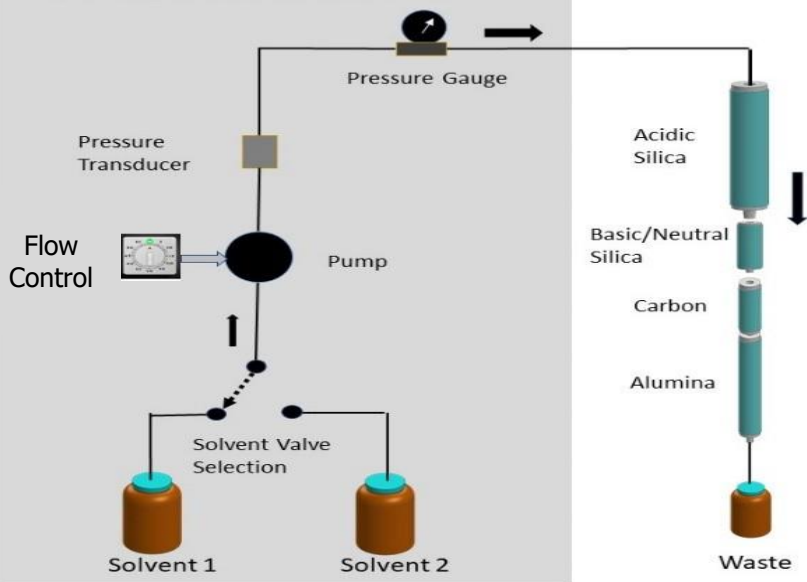
Install Columns



How It Works

Run Sample loading and Elution

Automated Solvent Delivery System



EzPrep – Stage 1

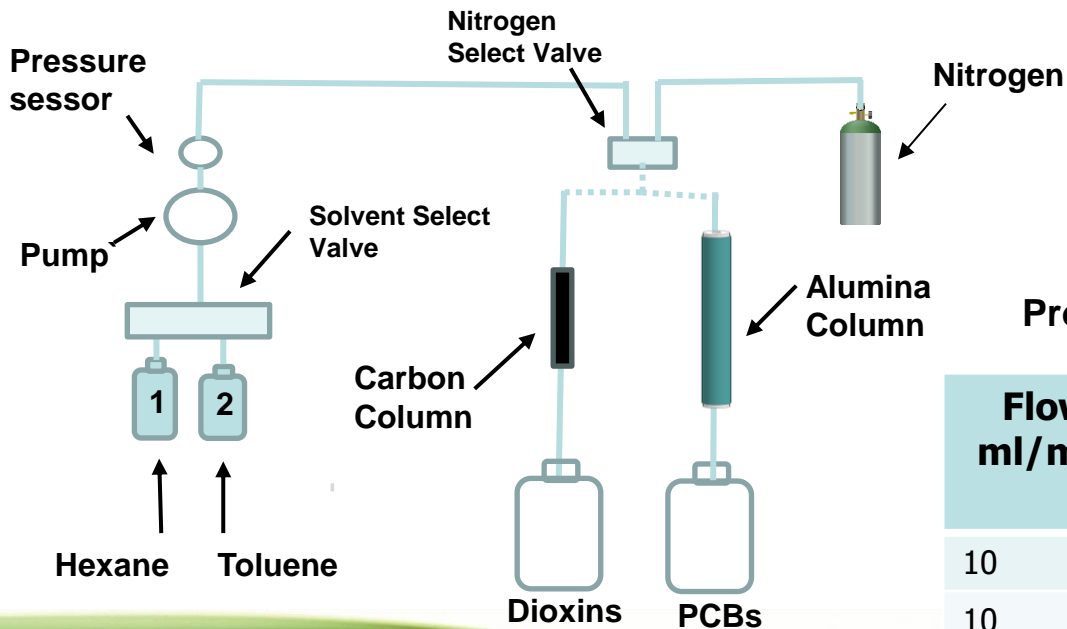
Program1:

- Load Samples
- Elute Dioxins & PCBs

Flow ml/min	Volume ml	Solvent	Description
10	160	1	Load sample
			Elute Dioxins & PCBs

How It Works

Elute Dioxins & PCBs



Program2: Dioxins & PCBs, Fractionation

Flow ml/min	Volume ml	Solvent	Description
10	40	2	Collect Dioxins
10	40	2	Collect PCBs

Cycle Time EzPrep/ +

- | | <u>Automated</u> | <u>Manual</u> |
|--|------------------|---------------|
| • <u>Set up time:</u> | | |
| • Assemble & Install acidic silica-carbon-alumina columns on column rack | | |
| • Place samples cartridges on top of acidic silica columns | | - 10 min |
| • <u>Program 1:</u> | | |
| elute hexane through all three columns ; apply nitrogen to push hexane onto the columns to waste | - 16 min | |
| • Disassemble the column set, install carbon and alumina columns on top of manifold | -10 min | |
| • <u>Program 2:</u> | | |
| Dispense Toluene through alumina & Carbon and collect PCBs & Dioxins | | -10 min |

Total Cycle Time 46 min

Combine best features (EZPrep Family)

Features	EzPrep	EzPrep/+
System run time for 6 samples	45 ~60 min	30 ~ 40 min
Fat Removal Capacity	.1 ~ 5g	.1 ~7g
Programmability	Minimal	Fully programmable
Pumping method	Vacuum	Pressurized
Use of certified pre-pack column	yes	yes
Use of electronics, electromechanical valve	No	Minimal
Labor required time to run 6 samples	30~60 min	20 ~ 30 min
Cross contamination	No Tubing	No Tubing

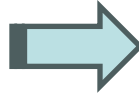
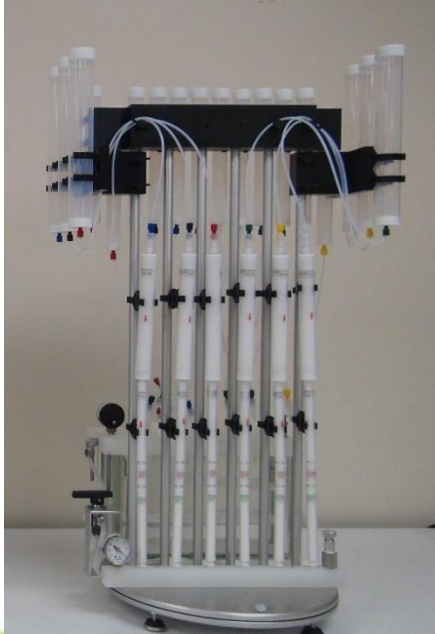
Comparison of Manual, Automated vs EzPrep Family

Task	Manual Sample Prep	Automated Sample Prep	EzPrep Semi-Automated	EzPrep/ + Automated
Labor Time	Hours	1 Hour	1 Hour (up to 2.5g fat) 2 Hour (2.5 to 5.0 g fat)	Less than 1 Hour
Accreditation	Slow	Fast	Fast	Fast
Accuracy & Precision	Varies	Excellent	Excellent	Excellent
Matrix	Dependent	Many	Many	Many
Instrument Maintenance	None	Required	Minimal	Minimal
Instrument Down Time	None	Some Times	none	Minimal
Fat Removal Capacity gram	Minimal	0.1 ~ 7.0 Gram	0.1 to 5.0 gram	0.1 to 7.0 g
Human Exposure	High	Minimal	Minimal	Minimal
Cost	5 x	50 x	10 x	25x

Automated EZprep expandable to EzPrep/+



EzPrep Expandable to EzPrep/+



SuperVap 12 Concentrator 50 mLs

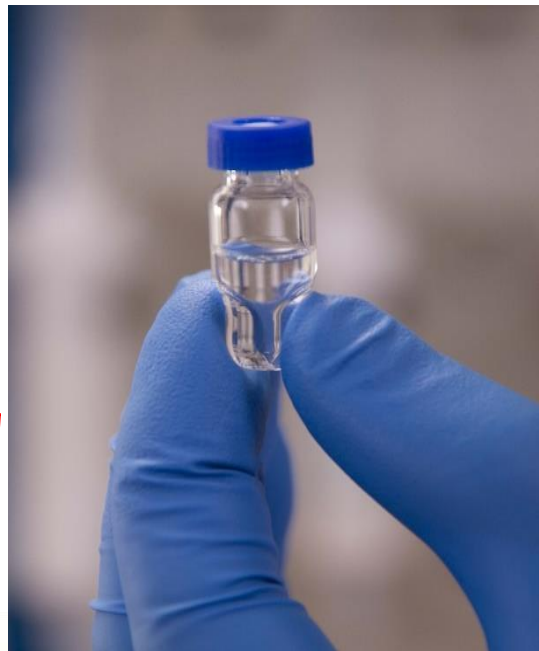


SuperVap Concentration/Evaporation

- System pre-heated to 50 °C.
- Samples evaporated at stable T under 8 psi nitrogen (sensor).
- 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 temp.



Direct-to-Vial



GC vial

Sample Analysis Work Flow



PLE Extraction

45 Min

+



Concentration

30 Min

+



**Sample Cleanup/
Concentration**

120 MIN

+



**Vial
Concentration**

45 Min

→



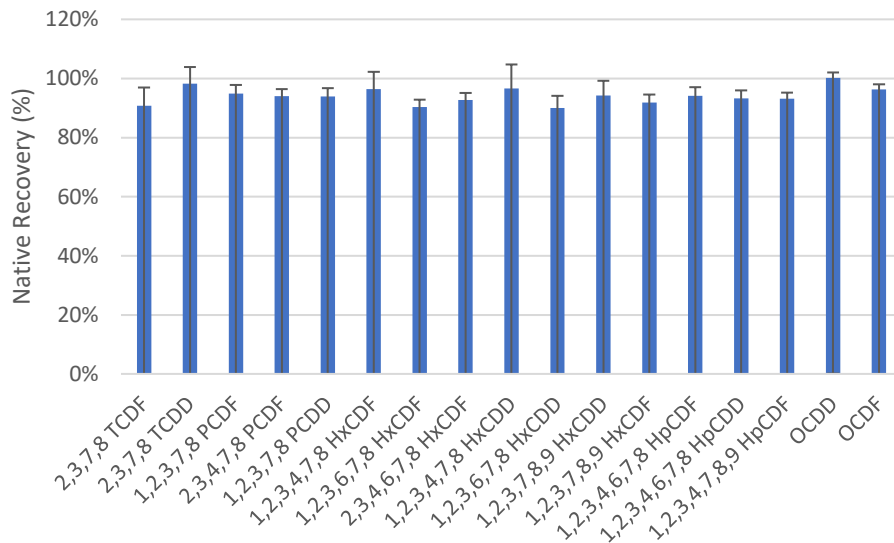
**Triple
Quad**

Total Sample Prep Time = 4 hours per batch of 6 samples

Direct to GC/MS or Triple Quad

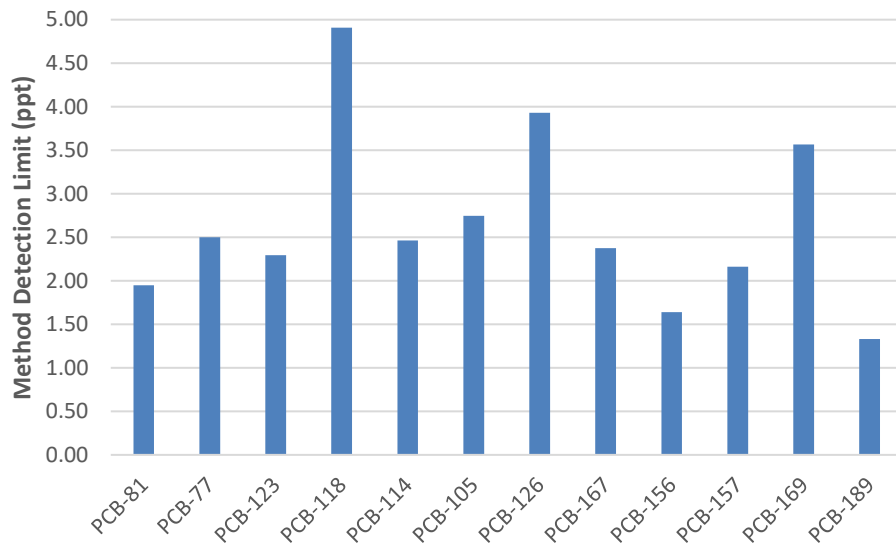


Native PCDD/F IDC



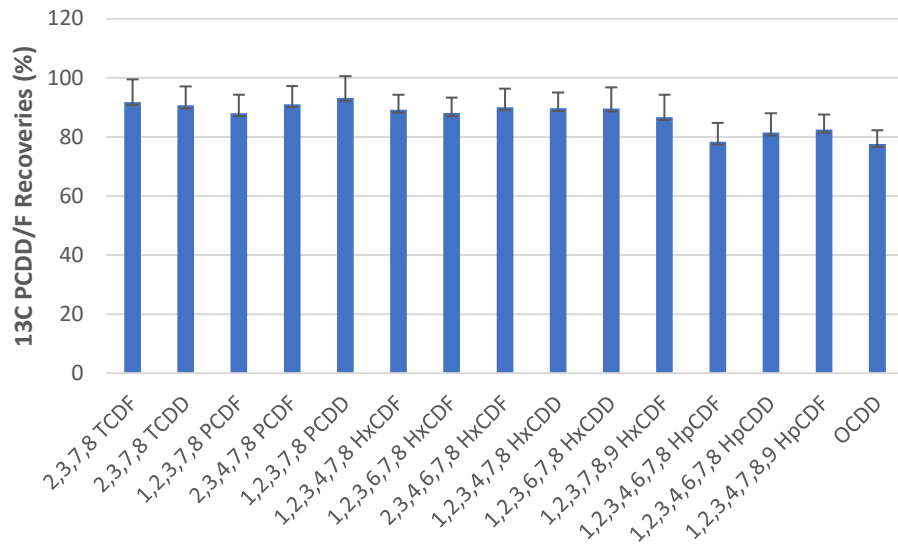
PLE-EZP-conc,
400-4000 pg,
n=6

Native PCB MDL



MDL, PLE-EZP-
conc, 10 ppt spike,
n=7

^{13}C PCDD/F recoveries no matrix



PLE-EZP-conc,
Ottawa Sand
matrix, n=6

Native PCBs in oil

	Cod oil		Pumpkin oil		Corn oil	
Natives in pg	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

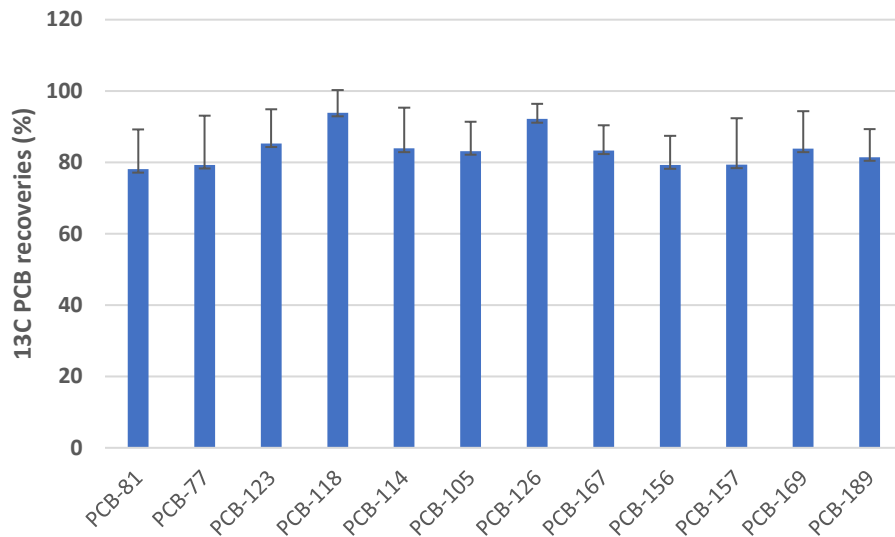
EZP - conc, 2.5
g oils

Native PCDD/Fs in feed and soil

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

PLE-EZP-conc,
5-10 g matrix

^{13}C PCBs in soil



PLE-EZP-conc,
10 g soil, n=6

Conclusions

- **EzPrep family of products designed to combine the advantages of Manual & Automated Sample prep**
- **EzPrep family of products designed to eliminate disadvantages of Automated and Manual system**
- **EzPrep/+ designed for ease of use, and lowering cost by using a minimum number of electronics and Electromechanical valves**
- **EzPrep family of products uses certified proprietary consumables design to speed up the sample prep workflow**
- **EzPrep family of products process 6 sample Clean-up per hour & 48 samples per day**
- **Combining EzPrep family of products with PLE (pressurized Liquid Extraction) allows laboratories to perform up to 48 samples from samples to vial**

Conclusions ...

- **Closed loop system, eliminates background contaminants & exposure to chemicals**
- **Optimized for solvent reduction while obtaining highest possible recoveries**
- **Certified disposable Columns with guaranty Low contaminants background and Excellent Recoveries**
- **Multi pump Solvent Delivery system brings convenient automated solvent selection & dispense with controllable flow & volume**
- **Little washing needed**
- **No cross-contamination**



Come see us at booth # A01

Questions?

raddink@fms-inc.com

thall@fms-inc.com