

Simple, Quick & Low Cost 6 -Parallel Channel, High Throughput Semi-Automated, Expandable to Automated, Sample Cleanup for POPs Analysis

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Introduction

- Stockholm Convention on Persistent Organics Pollutants 2004.
- Compounds of interest: polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs), and biphenyls (PCBs), PFAS (Added 2009 and later)
- 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

Why Manual Sample Prep is the preference for most labs?

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 Chemicals
- Lack of Traceability
- Difficult to Scale up

Automated Sample Prep Pros & Cons

Advantages Automated Sample Prep

- Efficiency & Speed
- Accuracy & Consistency
- Repeatability & Reproducibility
- Reduction of Manual Labor
- Documentation & Traceability
- Less exposure to Hazardous compounds
- Cleaner Background less 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

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
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FMS Solution

Minimize Disadvantages of Automation and Enhance the Advantages of Manual Sample prep

Disadvantages Automated Sample Prep

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

FMS Solutions " EZprep 6 sample parallel system"

EzPrep uses minimum number of electronics and mechanical valves reduces the cost

EzPrep /+ has one electronic module & 6 pumps which easily can be maintained & converted to SPE for PFAS

Simple to operate. no more than few hours training needed

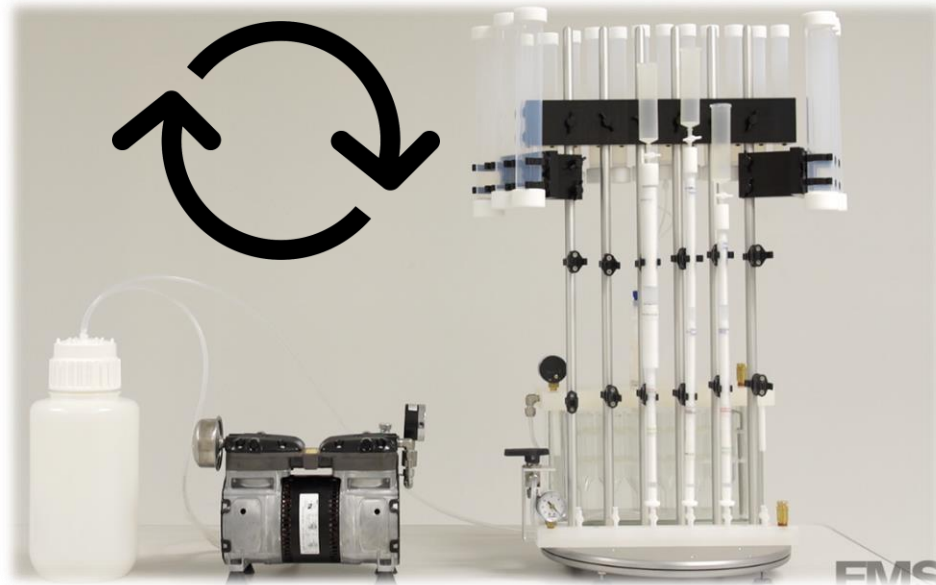
Minimum down time due to modularity and minimal electronics & electromechanical valves

EzPrep can run Dioxins, PCBs, PBDEs, PAHs, OCPs, EPH, TPH

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 No electronic or Electro-Mechanical to fail
- Affordable Automation: Low cost: fits the smallest of budget
- Expandable: Transition to automated sample prep

Design of EzPrep Using Vacuum Pump

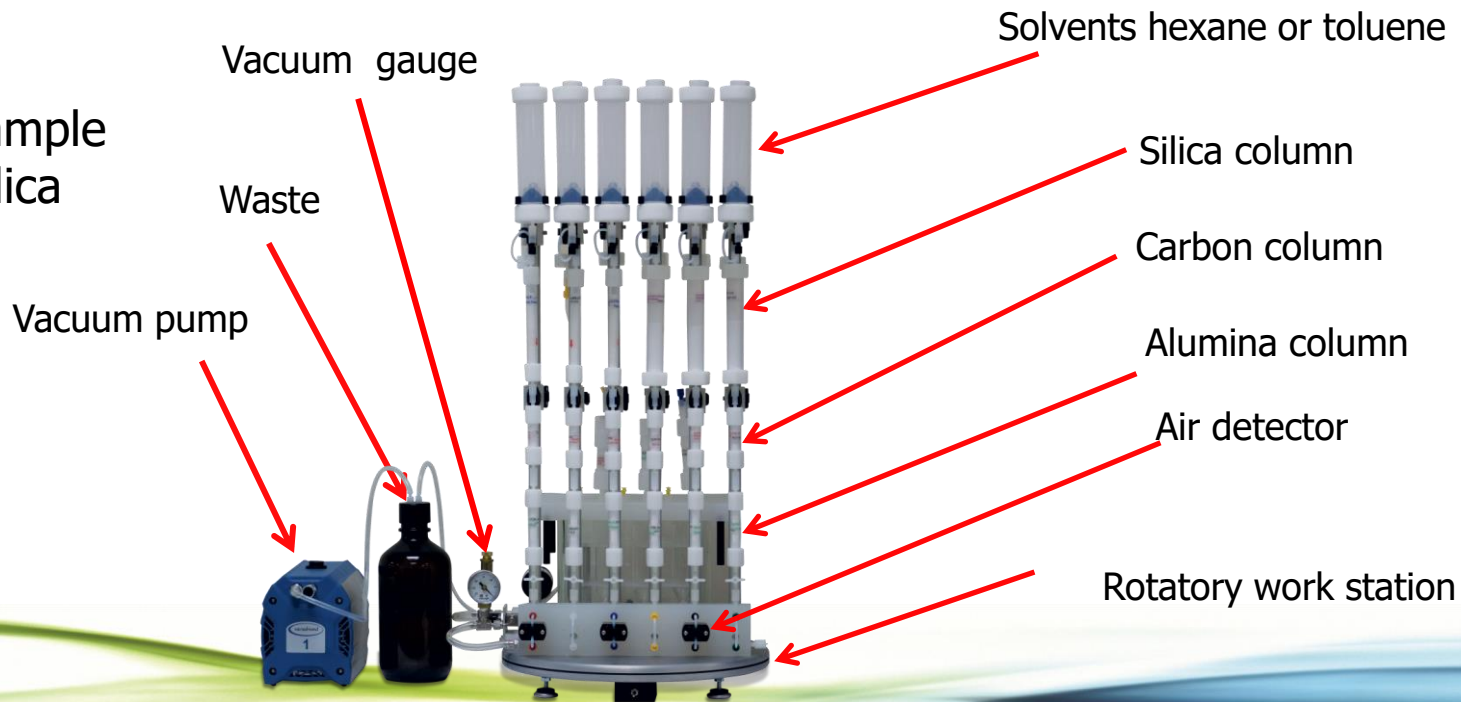
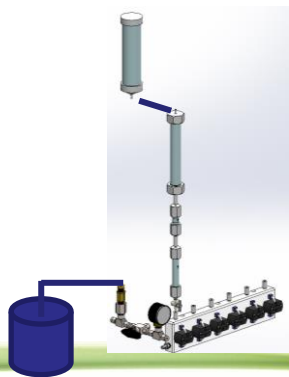


Design of Semi-automatic EZprep

Stage 1

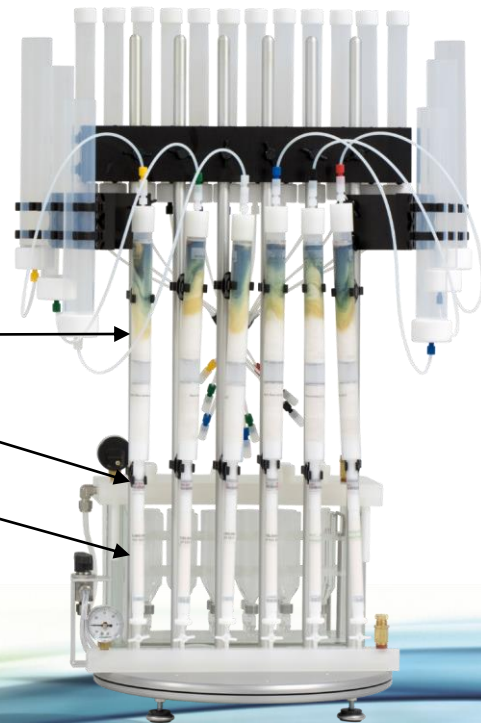
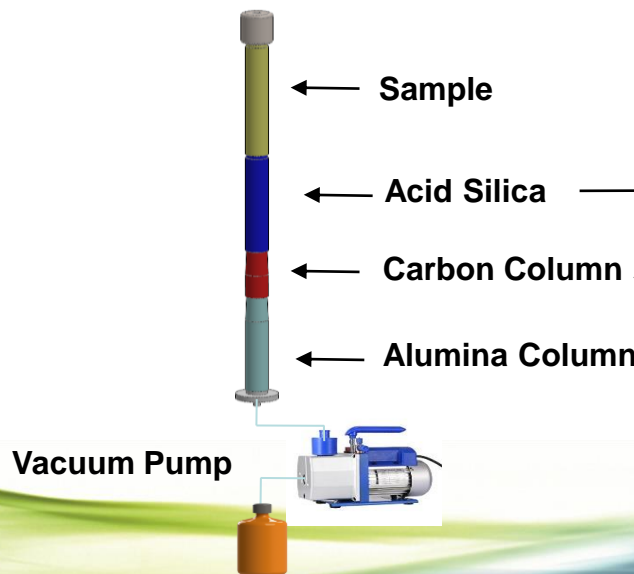
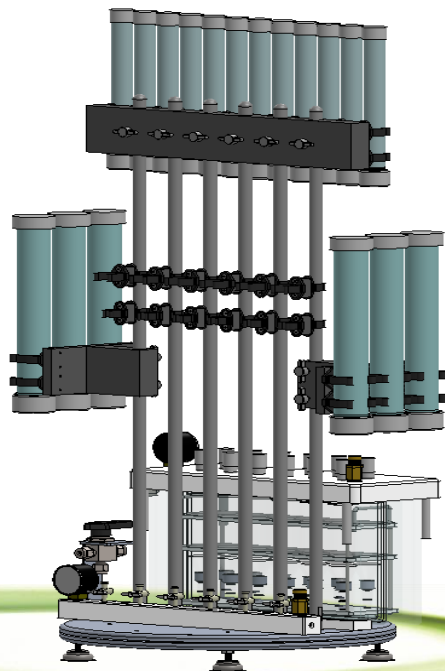
Step 1: Load Sample

Step 2: Elute Silica



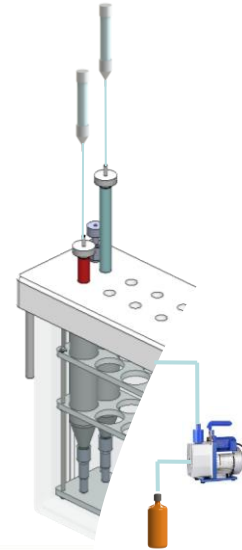
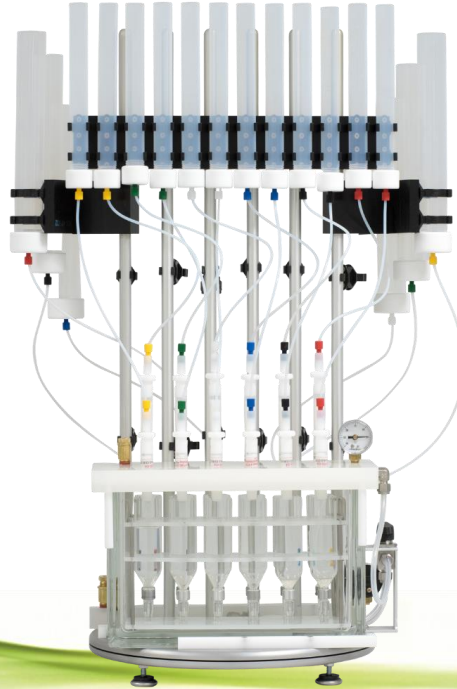
EzPrep System

Stage 1: sample loading & Elution

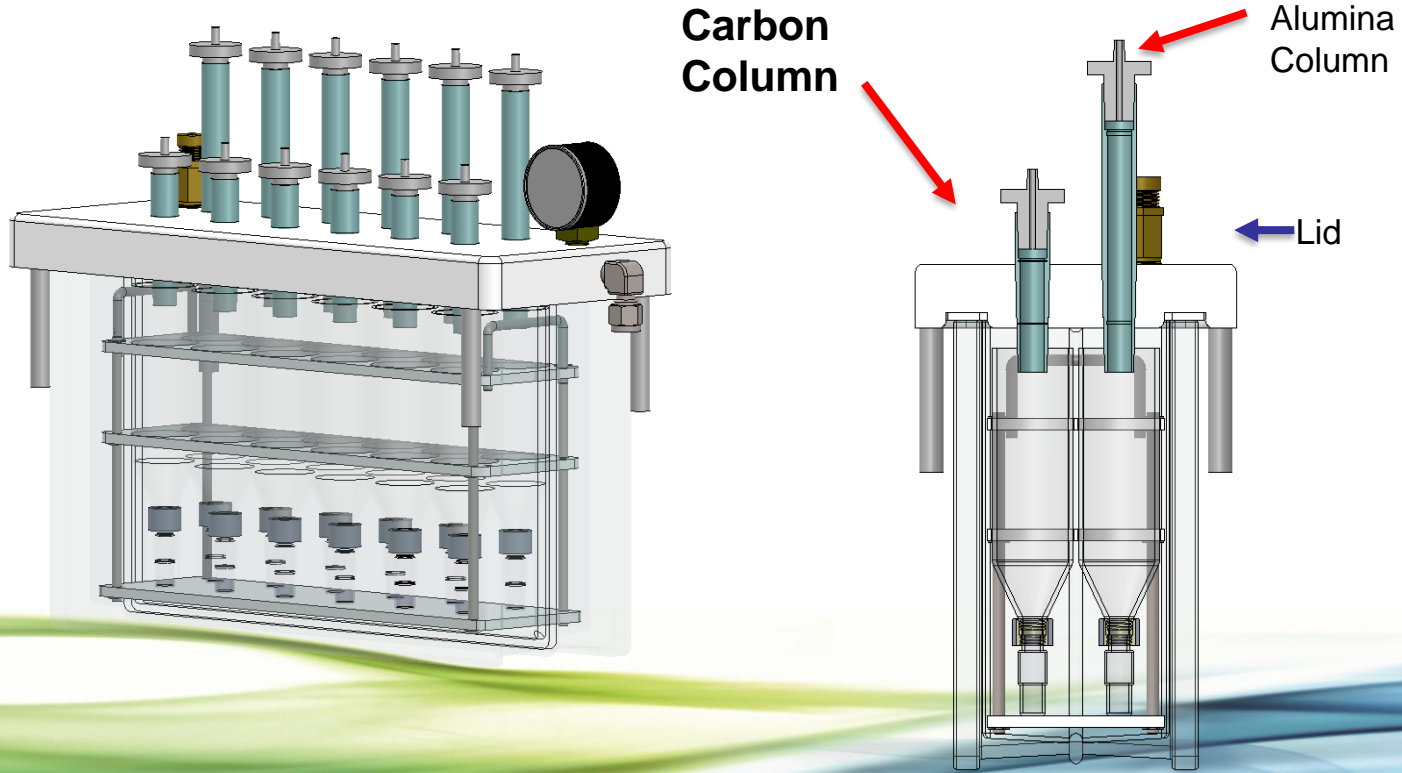


Ezprep System

Stage 2: Elution & Fractionation



Stage 2 : Fractionation



Sample Concentration Using FMS SuperVap



Cycle Time EzPrep

Processing 6 Samples

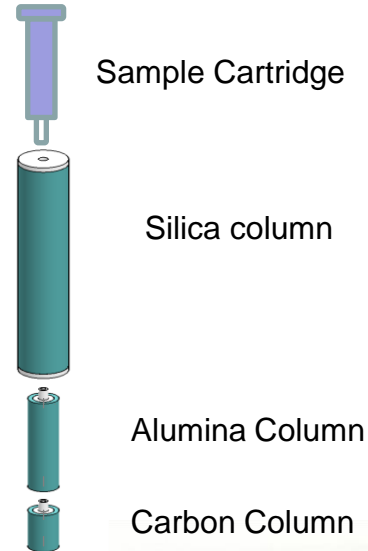
	<u>Automated</u>	<u>Manual</u>
• <u>Set up time:</u>		
• Assemble & Install acidic silica-carbon-alumina columns on column rack		
• Place samples cartridges on top of acidic silica columns , Add Solvents to solvent reserve		- 20 min
• <u>Program 1:</u>		
elute hexane through all three columns ; apply 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

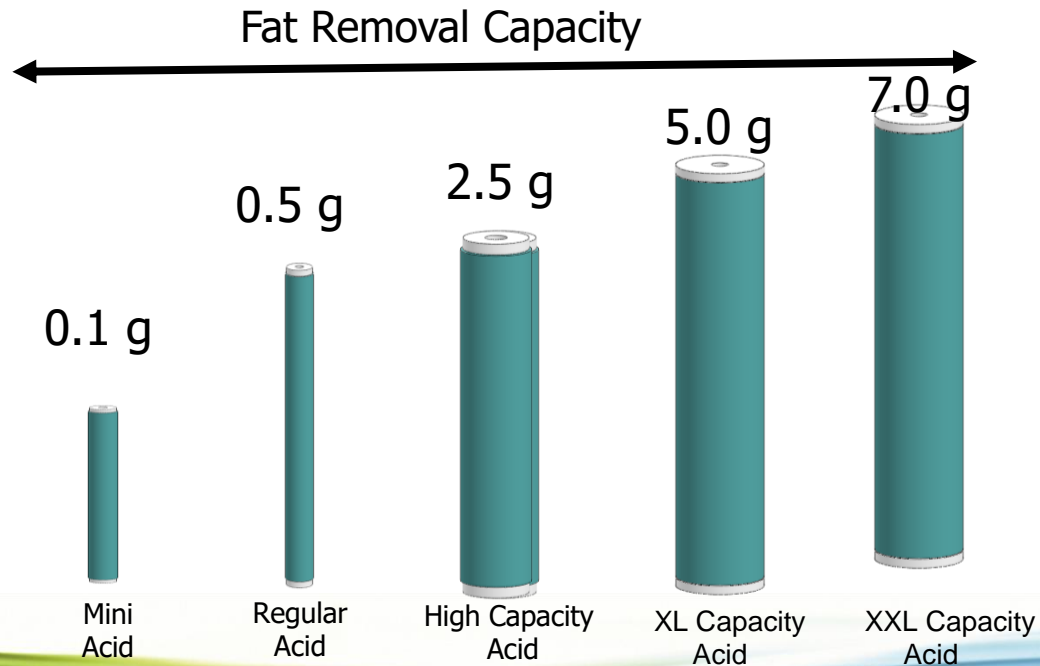
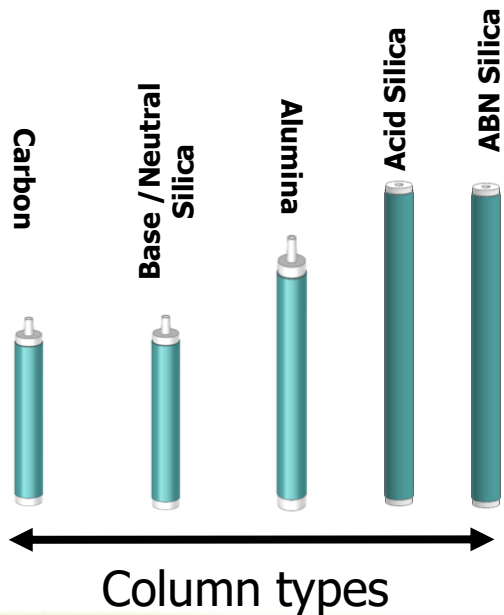
Disassembled



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

Benefits:

- Rapid Turn Around Time: 35 to 45 Minutes for 6 Samples
- Simple Programming: Just Select Solvent, Set Flow & Volume
- High Throughput: Can 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

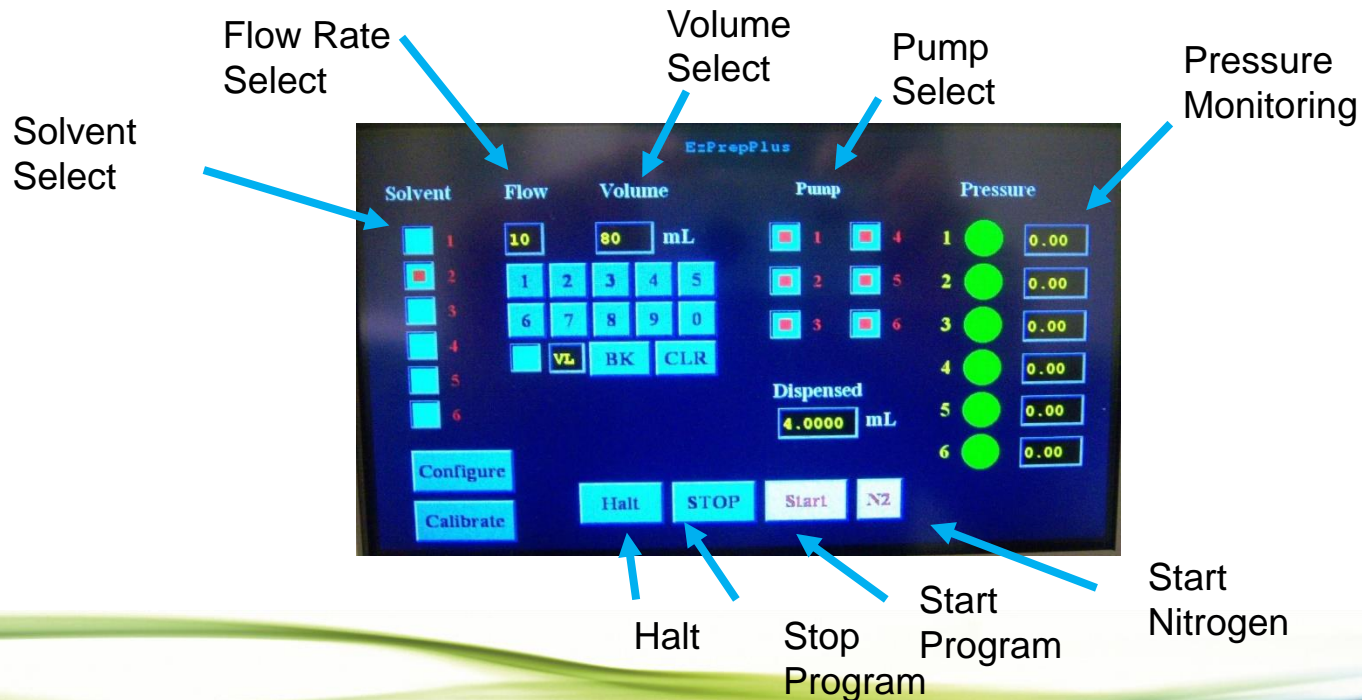
Attributes EZPrep/+

- **Optimized for solvent reduction while obtaining highest possible recoveries**
- **Certified disposable Columns with guaranteed Low native 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/+ PFAS conversion kit allows solvent delivery with no Teflon components**
- **EzPrep/+ designed with Minimum number of electronics and Electromechanical valve to lower cost and simplify the maintenance**

Automated EzPrep/+ For Dioxin & PCBs

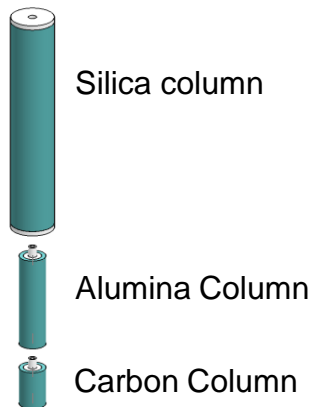


EzPrep /+ Control Panel



How It Works: System set up

Unpack column



Assemble Snap Columns



Add Samples

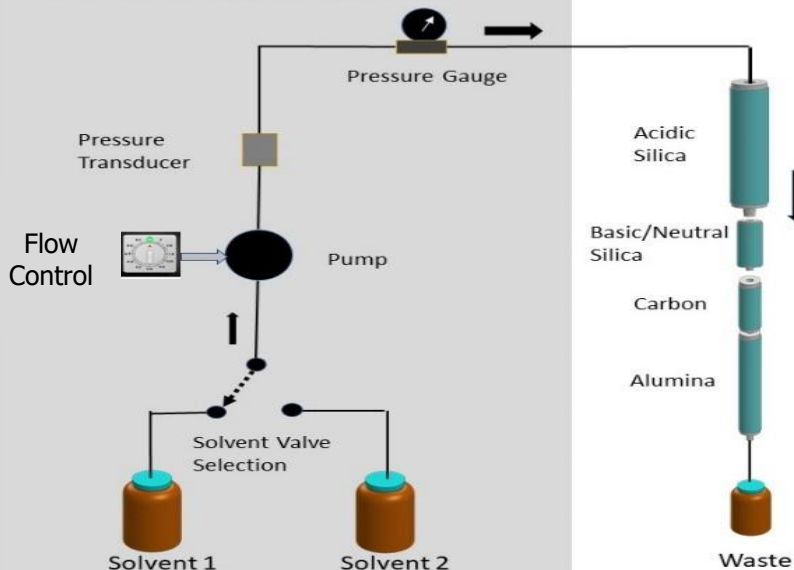


Install Columns



How It Works: Run Sample loading and Elution

Automated Solvent Delivery System



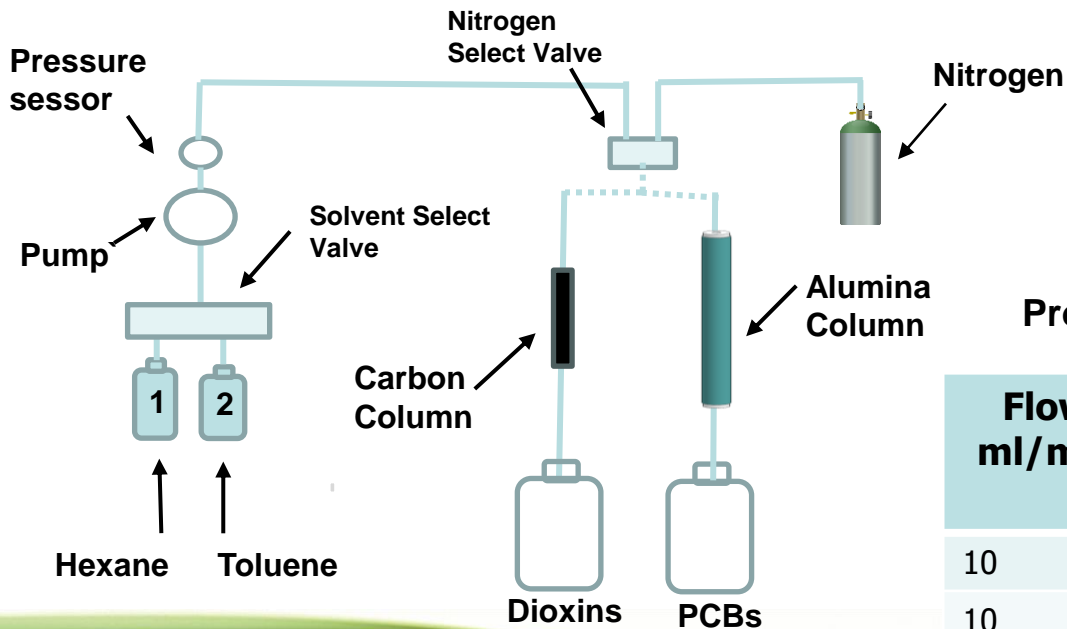
EzPrep – Stage 1

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

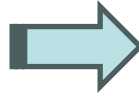
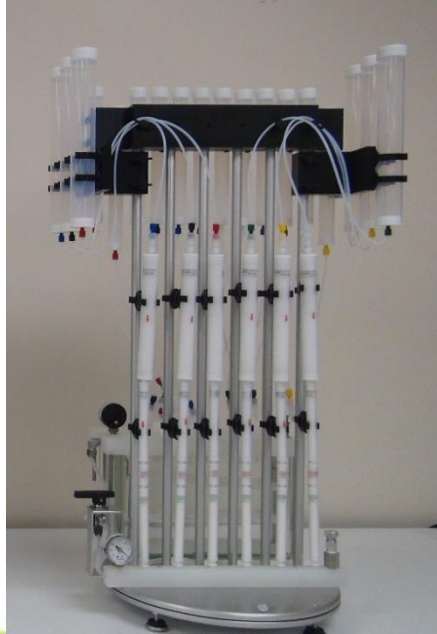
Combine Best Features (EZPrep Family)

Features	EzPrep	EzPrep/+
System run time for 6 samples	45 ~60 min	35-45 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
Technician presence time to run 6 samples	30 min	20 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 (on multiple days)	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	Sometimes	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

EzPrep Expandable to EzPrep/+ Add EzPrep/+ Control Module



SuperVap 12 Concentrator 50 mLs



Direct-to-Vial



GC vial

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 temperature.



Sample Analysis Workflow



PLE Extraction

45 Min

+



Concentration

30 Min

+



**Sample Cleanup/
Concentration**

120 MIN

+



**Vial
Concentration**

45 Min

→



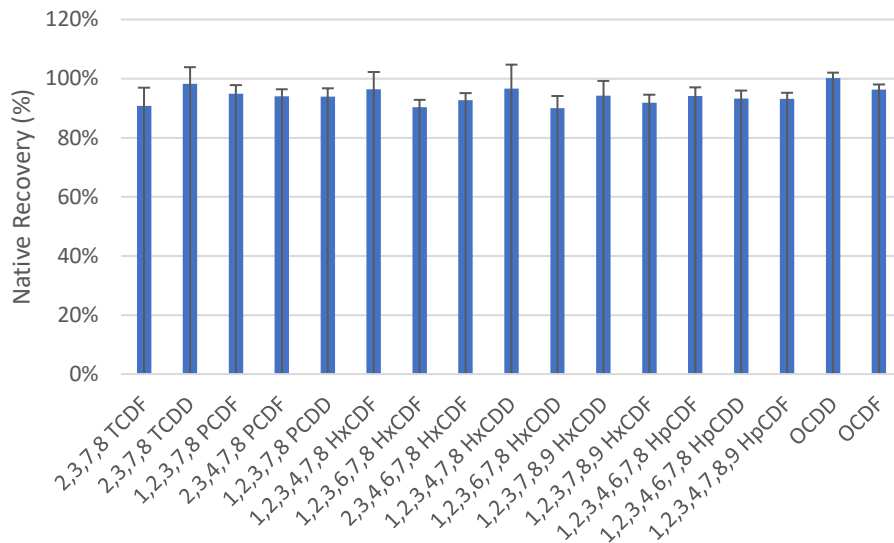
**Triple
Quad**

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

Direct to GC/MS or Triple Quad

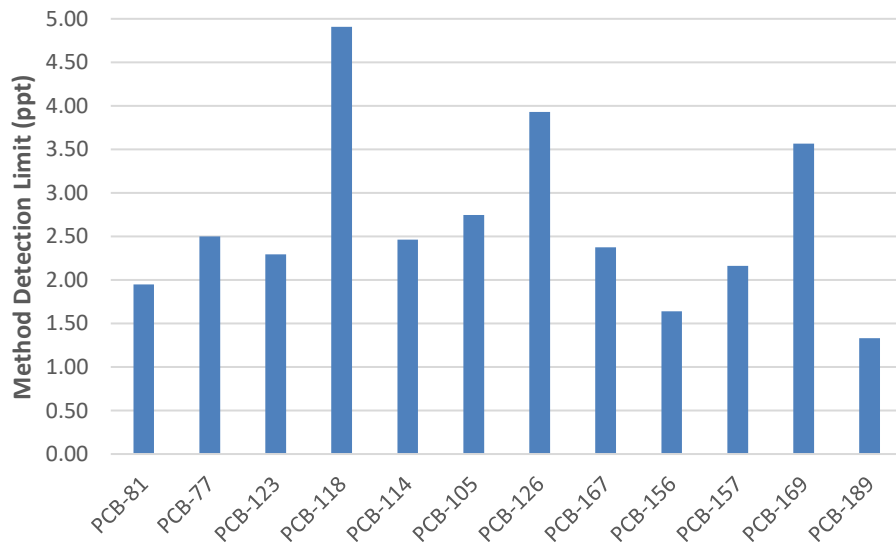


Native PCDD/F IDC



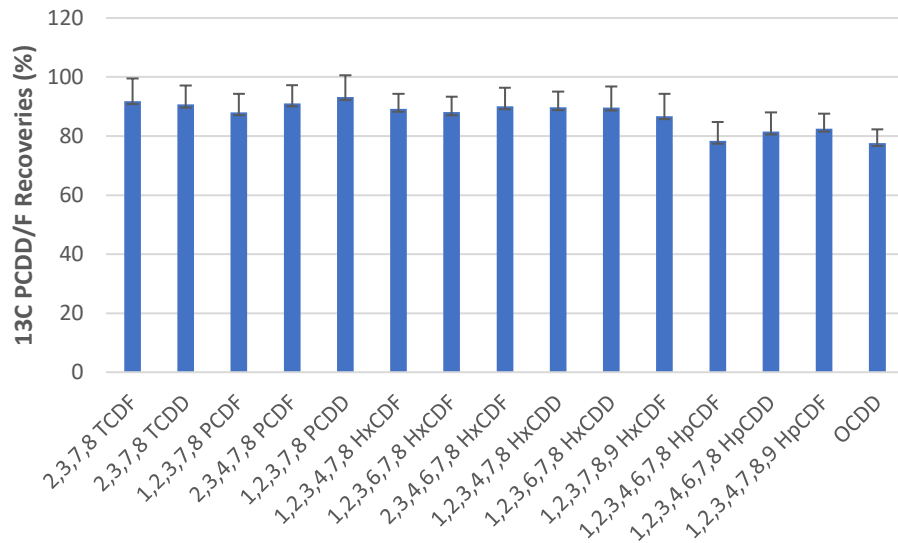
PLE-EZP-conc,
400-4000 pg,
n=6

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

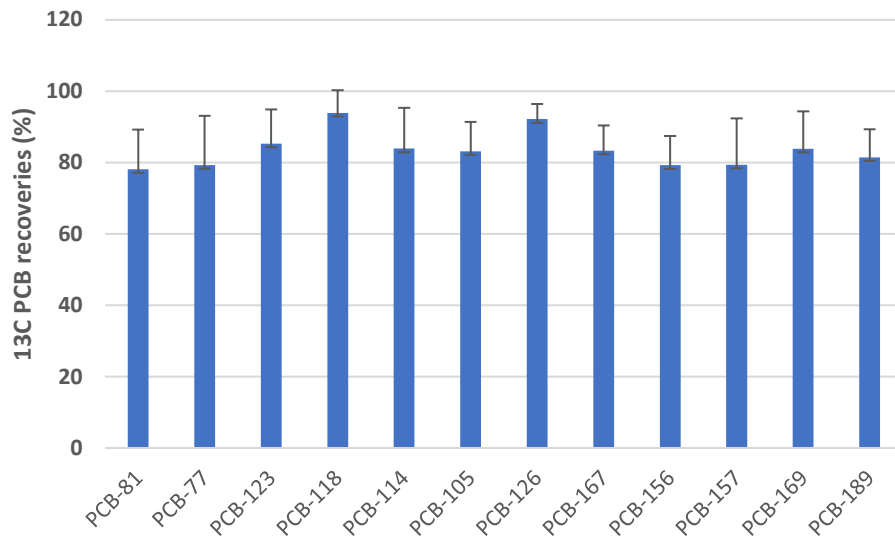
EZP - conc, 2.5
g oils

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 automated and eliminate the disadvantages of Manual & Automated Sample prep**
- **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 processes 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 sample to vial**

Conclusions ...

- **Closed loop system, eliminates native background contaminants & exposure to chemicals**
- **Optimized for solvent reduction while obtaining highest possible recoveries**
- **Certified disposable Columns with guaranteed Low contaminants background and Excellent Recoveries**
- **Multi pump Solvent Delivery system brings convenient automated solvent selection & dispenses with controllable flow & volume**
- **EzPrep/+ with Conversion kit produces very low PFAS background and excellent**
- **Extraction and cleanup recoveries for PFAS**



Come see us at booth # A8

Questions?

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