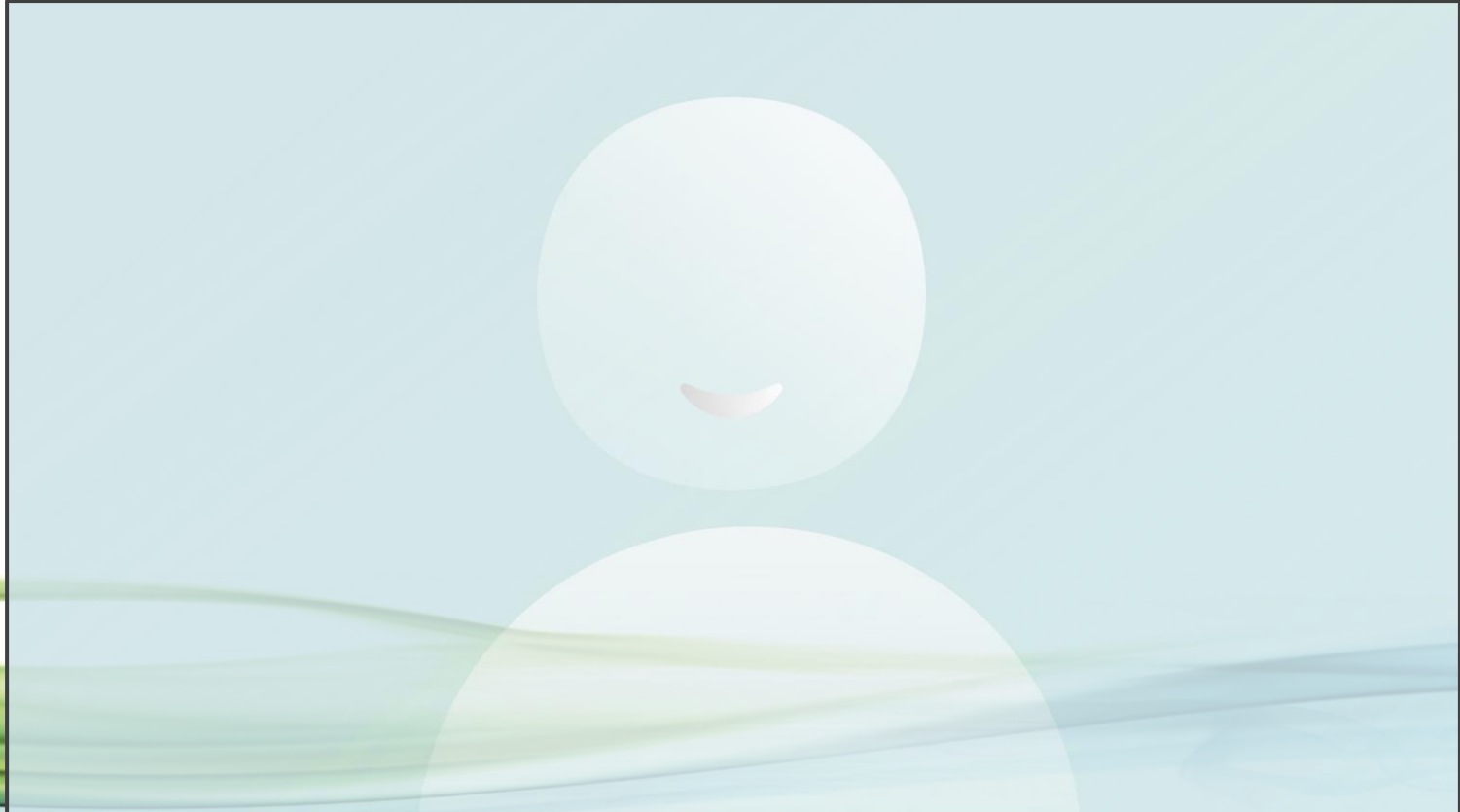# Automated Low Cost Sample Clean-Up System for Dioxin & PCBs

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Fluid Management Systems

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Introduction

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

FMS
Fluid Management Systems

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

EzPrep Automated Sample Cleanup Design

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

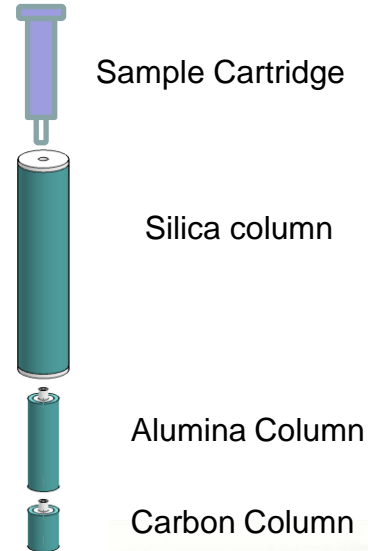➢ Affordable Automation: Low cost

FMS
Fluid Management Systems

FMS Certified Snap-In columns:

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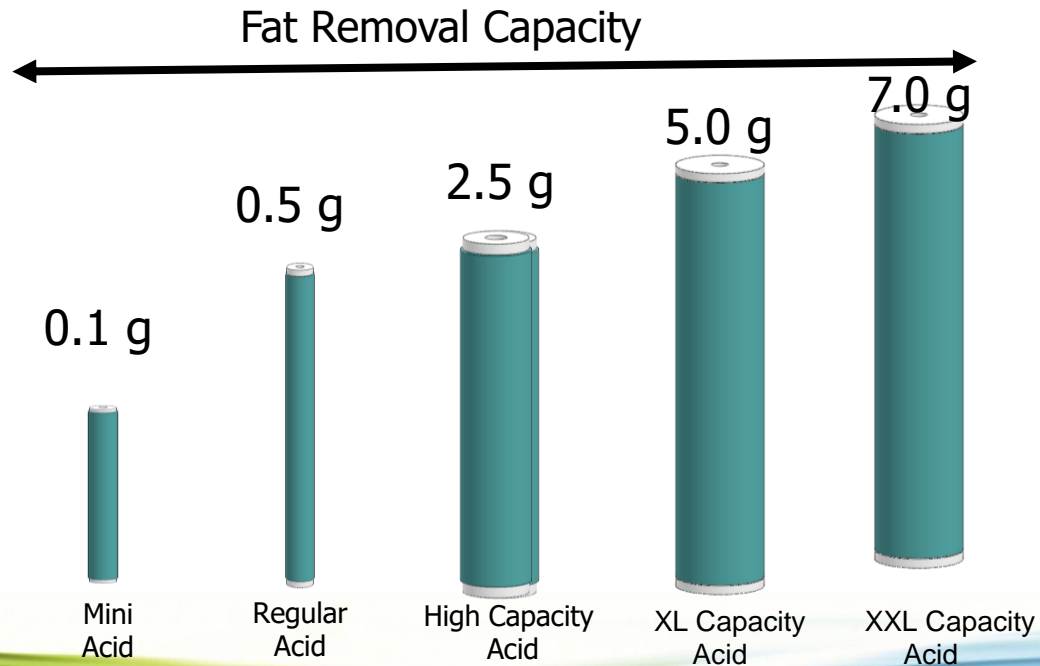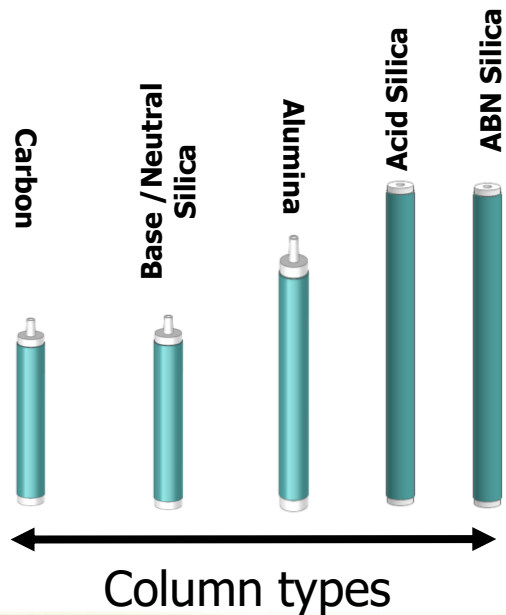- ➤ **Different size for different fat capacity from 0.2 up to 7 gm of Fat**

Disassembled   **Assembled**

Sample Cartridge

Silica column

Alumina Column

Carbon Column

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

- ➢ **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**
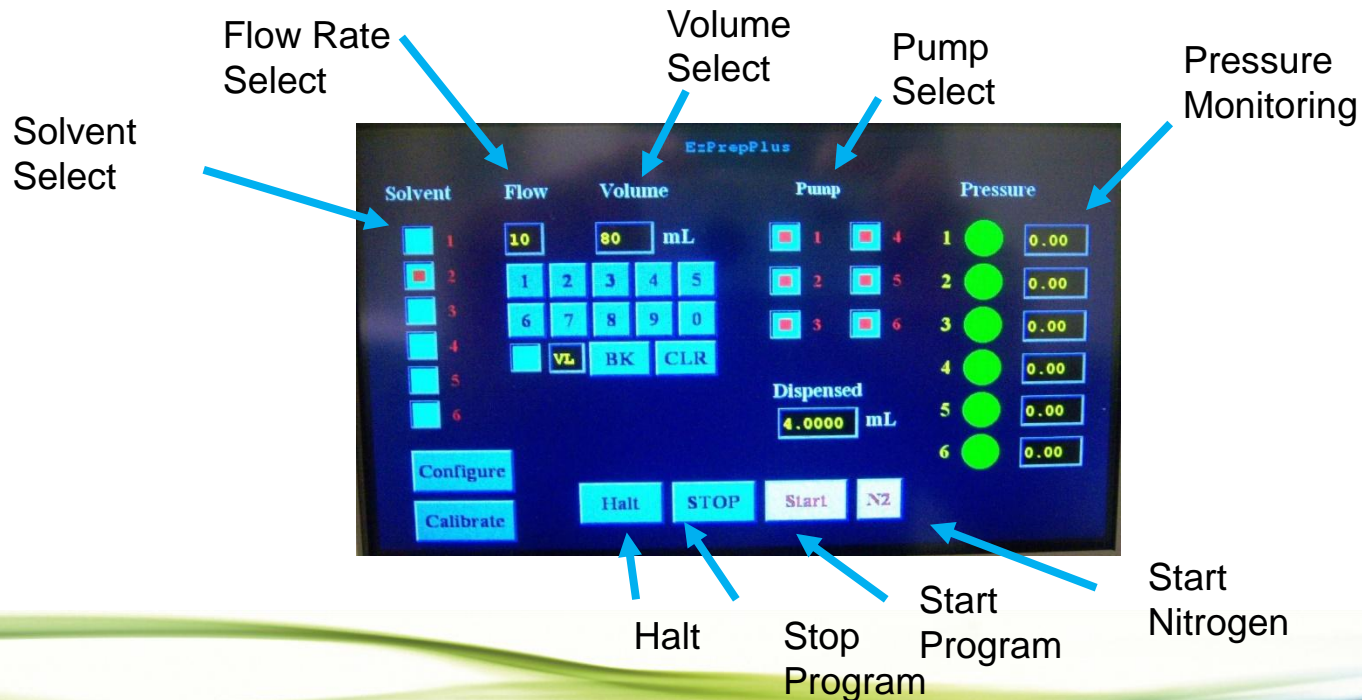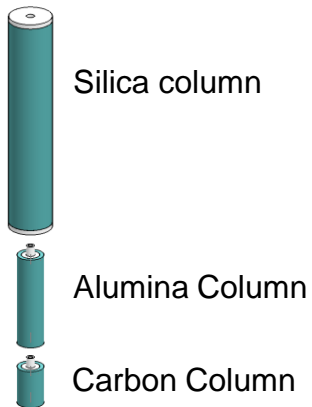
Live Action
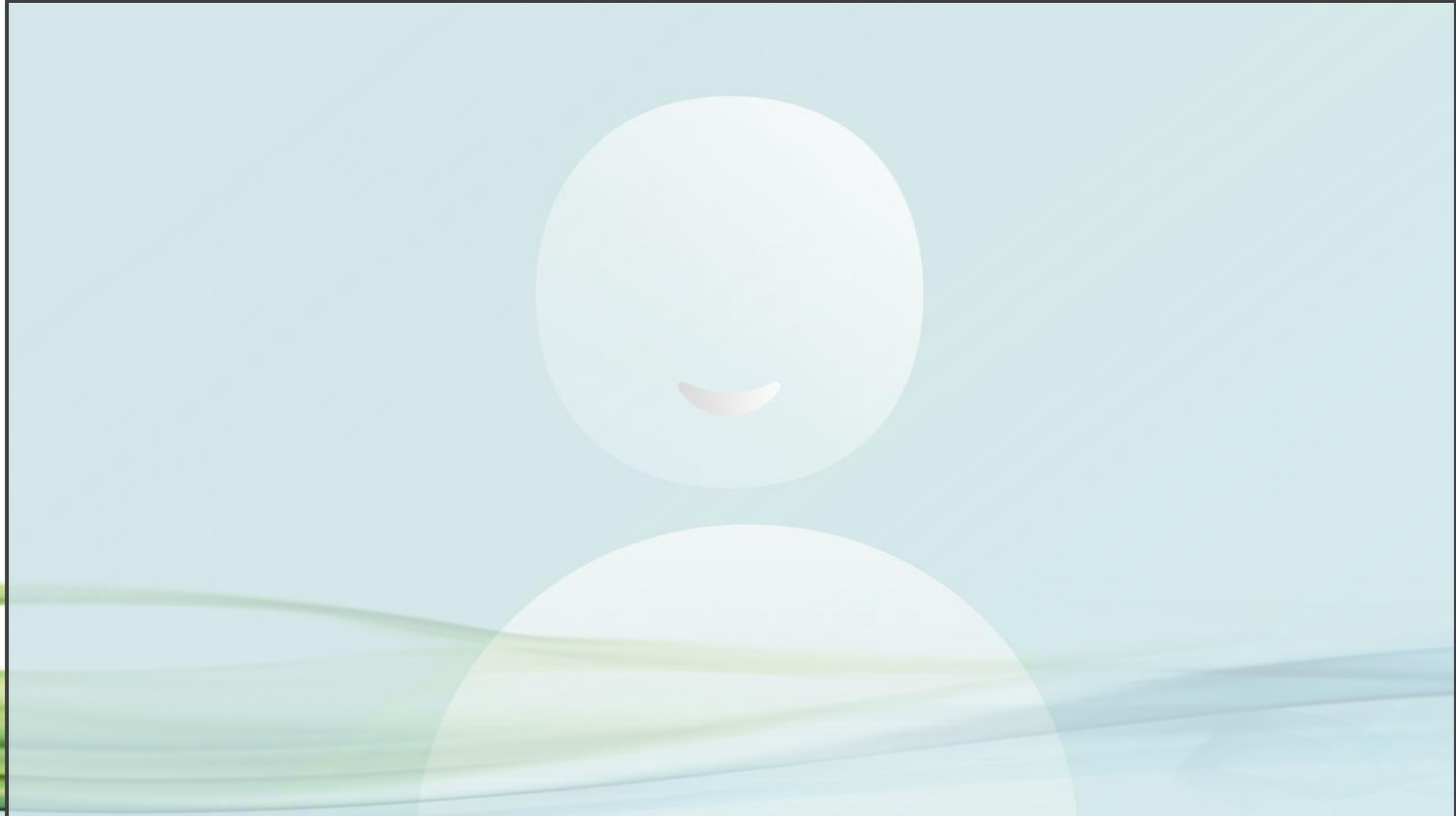
How It Works
System set up

Unpack column

Assemble Snap Columns

Add Samples

Install Columns

Silica column

Alumina Column

Carbon Column

Automated Solvent Delivery System
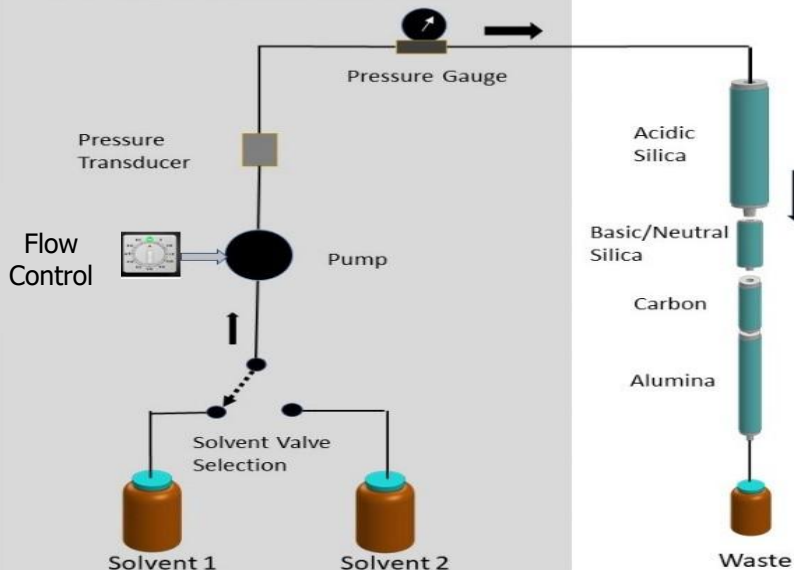
EzPrep – Stage 1

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

FMS Fluid Management Systems

Pressure sessor

Pump

Solvent Select Valve

Nitrogen Select Valve

Nitrogen

Carbon Column

Alumina Column

1   2

Hexane   Toluene
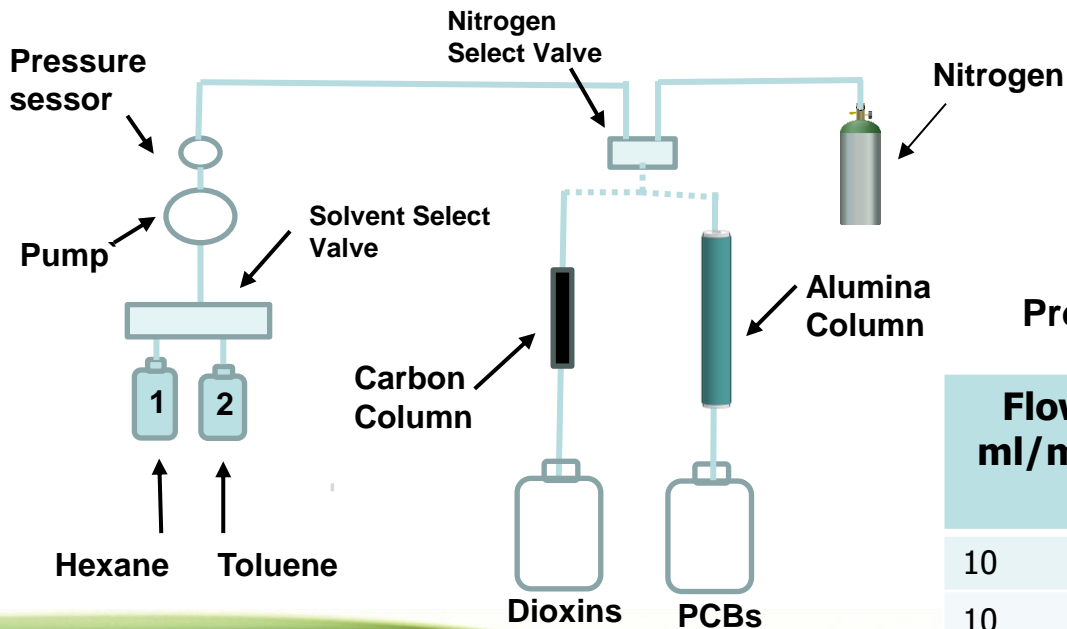
Dioxins   PCBs

Program2: Dioxins & PCBs, Fractionation

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

Live Action

Cycle Time EzPrep/+

FMS
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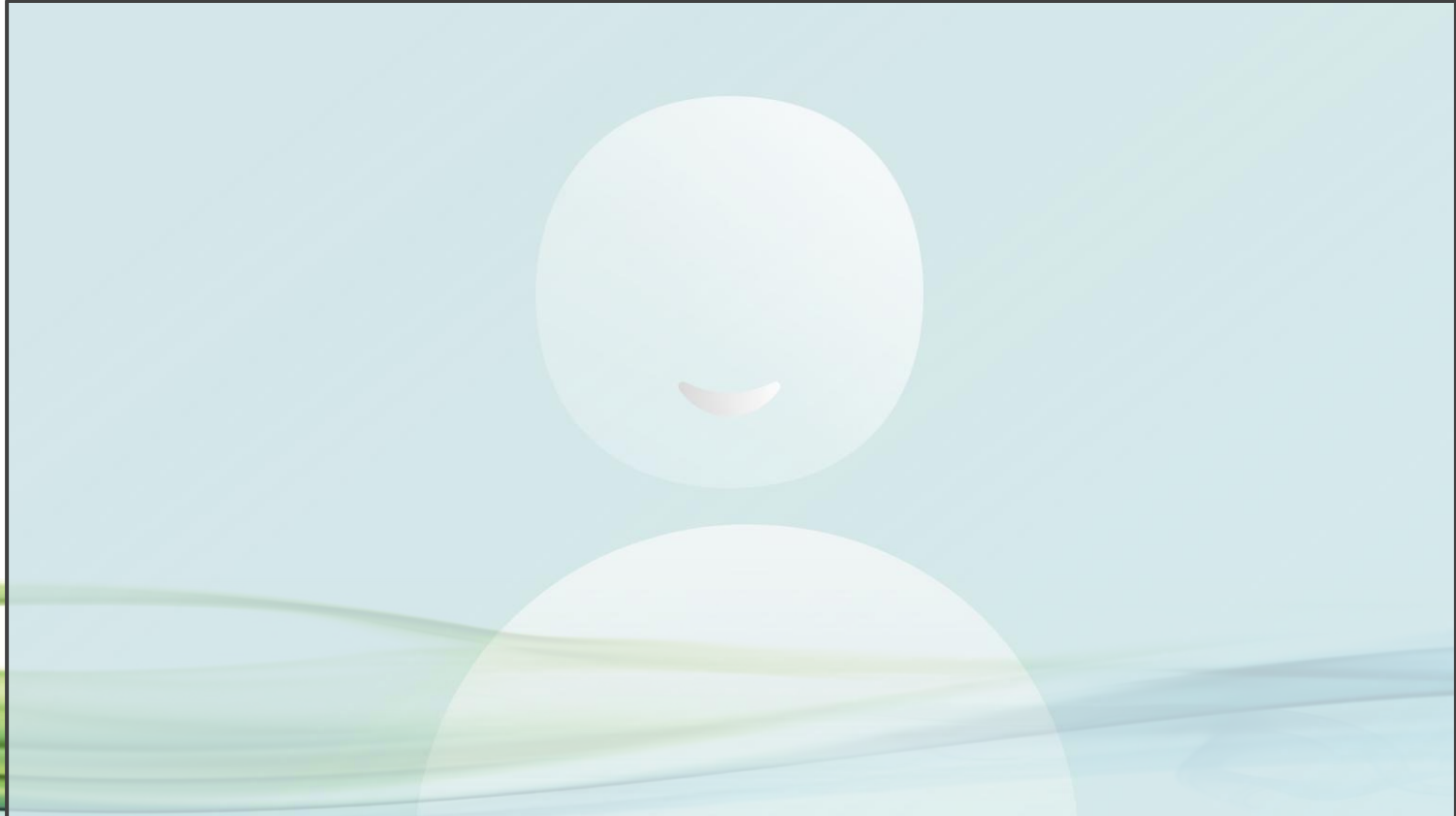
	Automated	Manual

- **Set up time:**
- Assemble & Install acidic silica-carbon-alumina columns on column rack
- Place samples cartridges on top of acidic silica columns — - 10 min

- **Program 1:**
  elute hexane through all three columns ; apply nitrogen to push hexane onto the columns — - 16 min
  to waste
- Disassemble the column set, install carbon and alumina columns on top of manifold — -10 min

- **Program 2:**
- 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 Cross contamination	30~60 min No Tubing	20 ~ 30 min 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/+
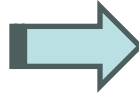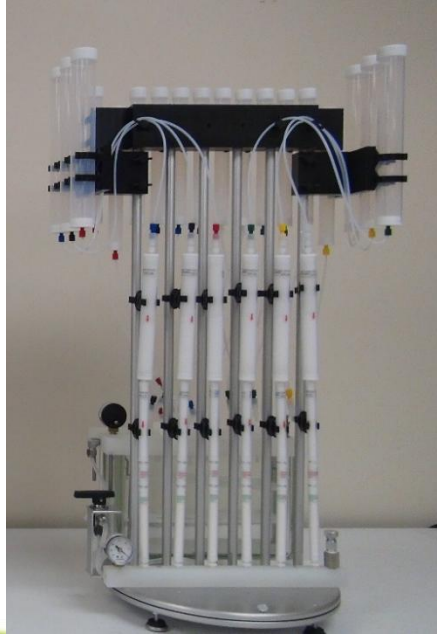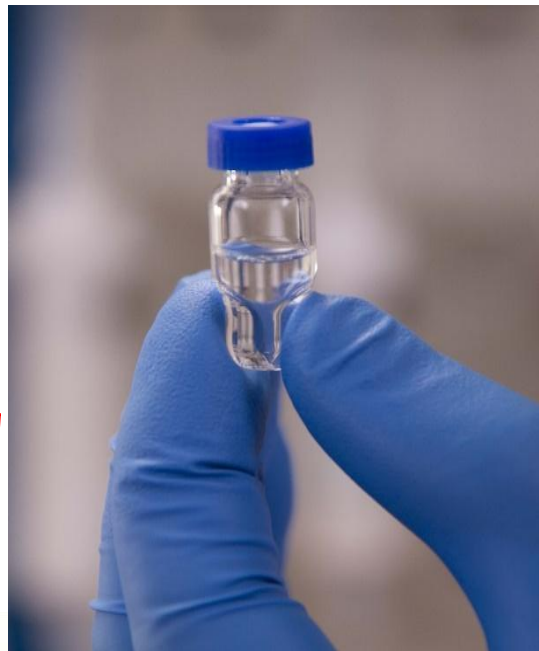
• System pre-heated to 50 °C.

• 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
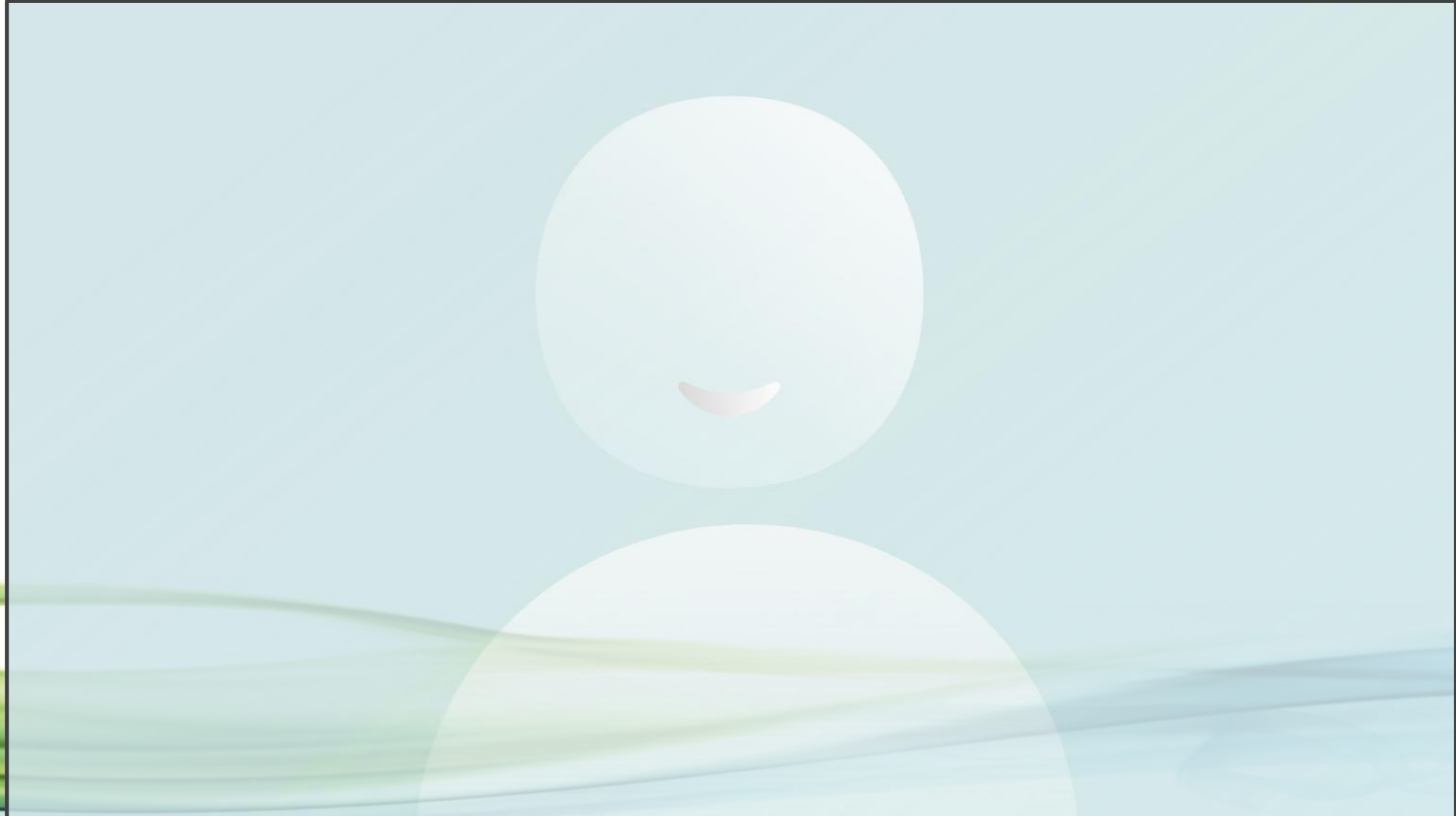
45 Min

Triple Quad

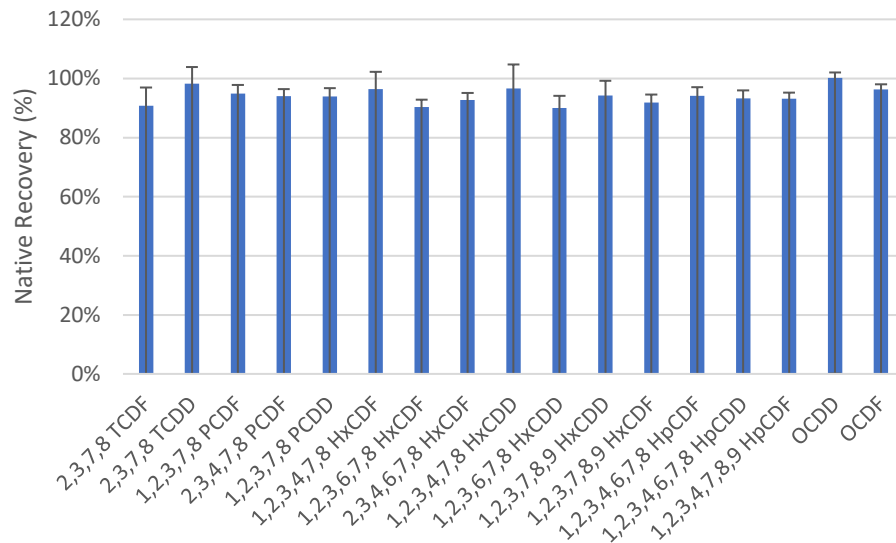**Total Sample Prep Time = 4 hours per batch of 6 samples**

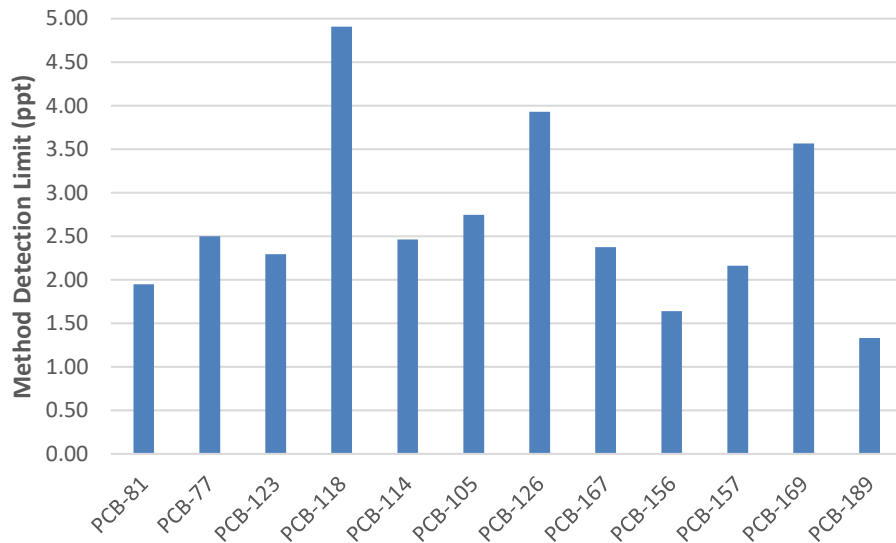# Direct to GC/MS or Triple Quad

Live Action

Native PCDD/F IDC

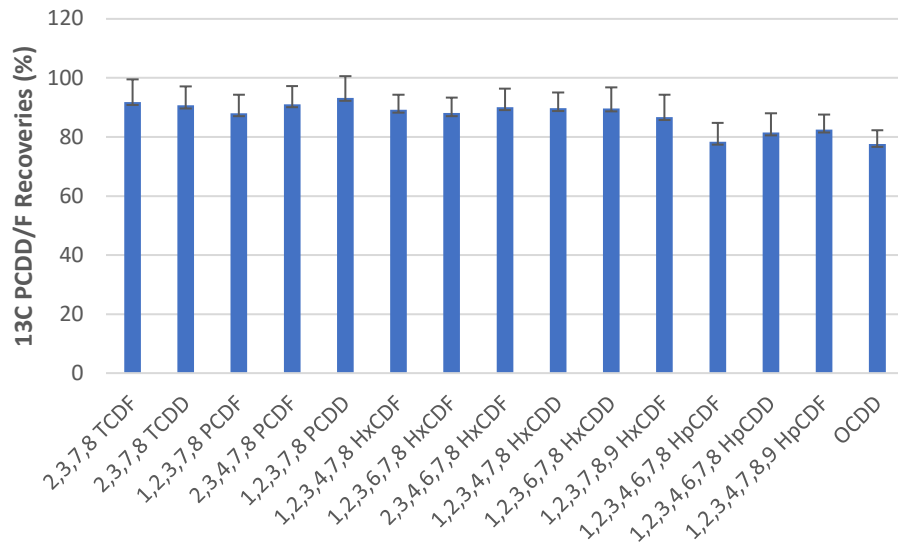PLE-EZP-conc,
400-4000 pg,
n=6

Native PCB MDL

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

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

Native PCBs in oil

Natives in pg	Cod oil		Pumpkin oil		Corn oil	
	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

| 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	0.1	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
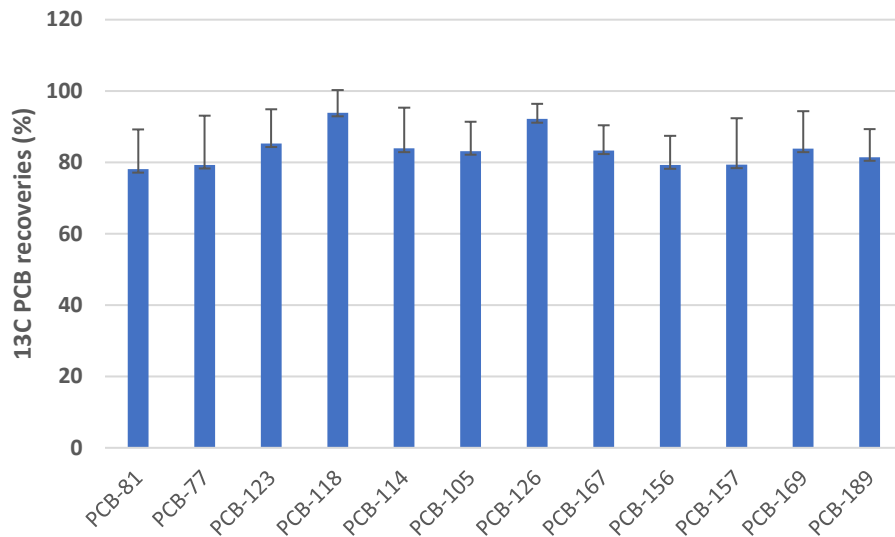
Conclusions

➢ **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

Live Action
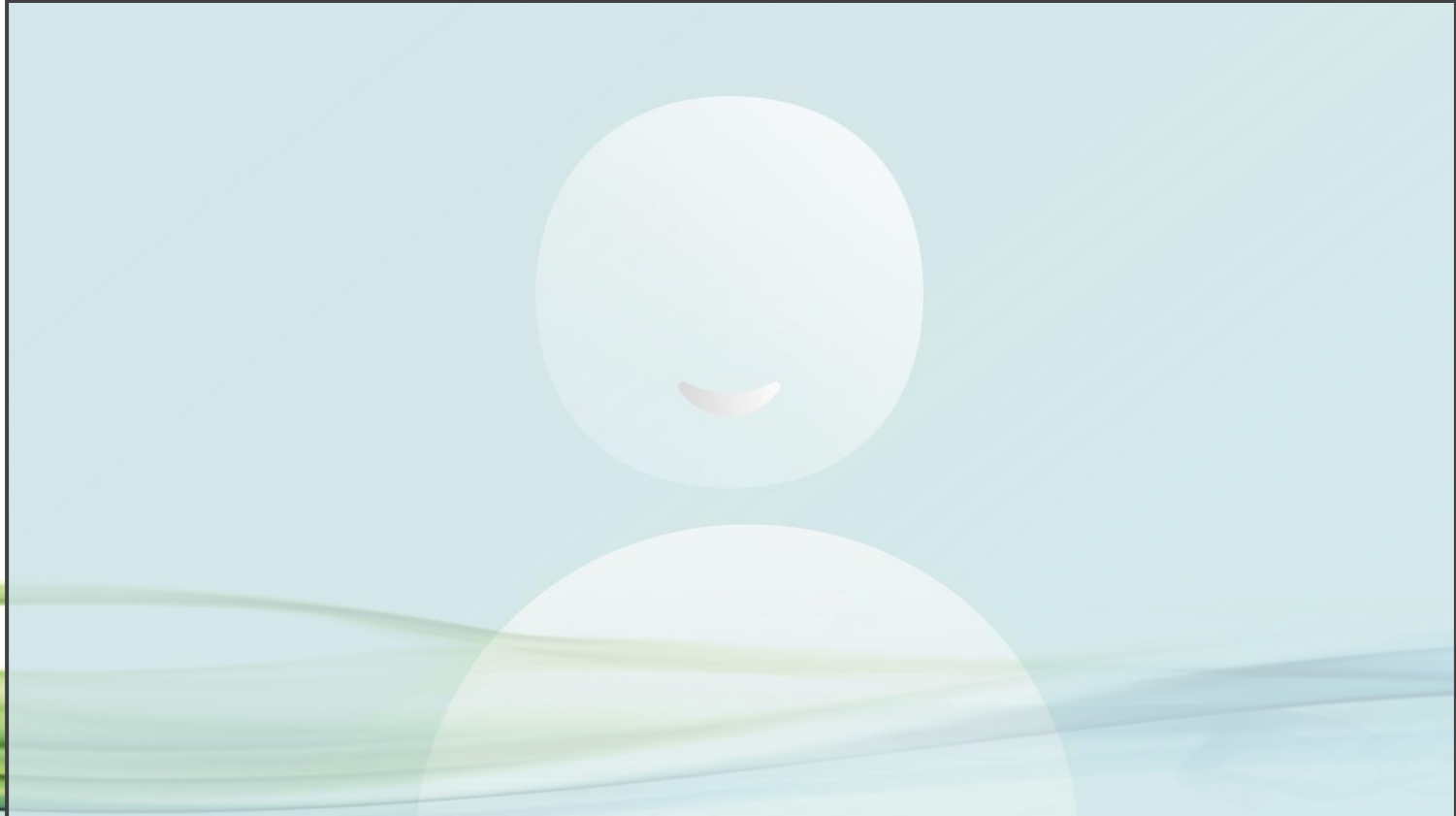
Questions?

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