![FMS - Fluid Management Systems]

Fast, Flexible, High Throughput Sample Preparation for the Analysis of Dioxins and PCBs

Overview

- Introduction

- Sample Prep Workflow

- Automated Hyphenated System Configurations for different Matrices and Extractions

- Semi Automated Systems

Introduction

- Stockholm Convention on Persistent Organics Pollutants 2004

- Compounds of interest:
  - Polychlorinated biphenyls (PCBs)
  - Polychlorinated dibenzo-p-dioxins (PCDDs), and furans (PCDFs)
  - PBDEs

- 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.

Background

- POPs (PCDD/Fs, PCBs) continue to attract interest around the world due to strict regulations enforced in many countries

- Rapid, quality sample clean up and analysis is needed for many laboratories processing samples

- Processing times and cost are important considerations

- In the US, EPA methods SW-846, 1613, 1668 and 8082A are used for PCBs and PCDD/Fs work

- Sample extracts in DCM, hexane or toluene

- Cleanup for analysis of all 209 PCBs in common in North America - extracts are often in toluene after Soxhlet Extraction

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 (can be orders of magnitude higher than analytes)**

- **Pico/femto-gram analyses require ultra pure extract and excellent instrument sensitivity**

Automated Sample Prep

- **Advantages of Automated Sample Prep**

  – Rapid Turn Around Time: 30 to 45 Minutes for 6 Samples

  – Cleaner Background Interferences: Closed Loop System

  – Quality Results: Certified Pre-packaged Columns
  –
  – Green Technology: Lower solvent and power use

  – QA/QC & Accreditation Requirements: Easier to Manage

  – Computerized Method: Instrumentation based prep

- **Advantages of Manual Sample Prep**
  - Most labs use a Manual Methods for the following reasons:

    - No electronics or mechanical components to fail
    - No down time due to the system failure
    - No service contract
    - No capital equipment cost

Sample Processing Workflow

- Analysis of various matrices for PCDD/Fs and PCBs using extraction, clean up and concetration.

- Soxhlet extraction (typically up to 24-36 h).

- Concentration step

- 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 H_2SO_4 acid mixed with silica; NaOH mixed with silica Neutral Silica

- Fractionation and Concentration

Sample Prep and Analysis WorkFlow in Hours vs Days

Automated Sample Prep Time

1 up to 8 samples

= 2hrs

Extraction	+	Concentration	+	Sample Cleanup	+	Concentration	+	GC/MS
30 Min		30 Min		30 Min		30 Min		45 Min

Manual Sample Prep Time   = 2 to 4 days

Extraction	Concentration	Sample Cleanup	Concentration	GC/MS
24 hours	60 Min	24 to 48 hours	60 Min	45 Min

Sample Preparation consist of three main instruments:

1- Extraction   2- Sample Clean-Up   3- Concentration

= Total Prep

PLE Extraction

PowerPrep Sample Cleanup

SuperVAP Concentration

Total Sample Prep

Extraction Procedure

- 1 g sample mixed with Hydromatrix^{TM} to dry, transferred to extraction cells

- Spiked with ^{13}C PCDD/Fs and PCBs standards.

- Void volume filled with Hydromatrix^{TM}.

- Sample Cells filled with 50% mixture Hexane/Methylene Chloride.

- Cells pressurized to 1500 PSI and heated to 120 ºC.

- Temperature held for 20 minutes.

- Extraction cells cooled, flushed with solvent (50% cell volume), and nitrogen

- Sent directly to the SuperVap collected in 250 mL tubes and automatically concentrated
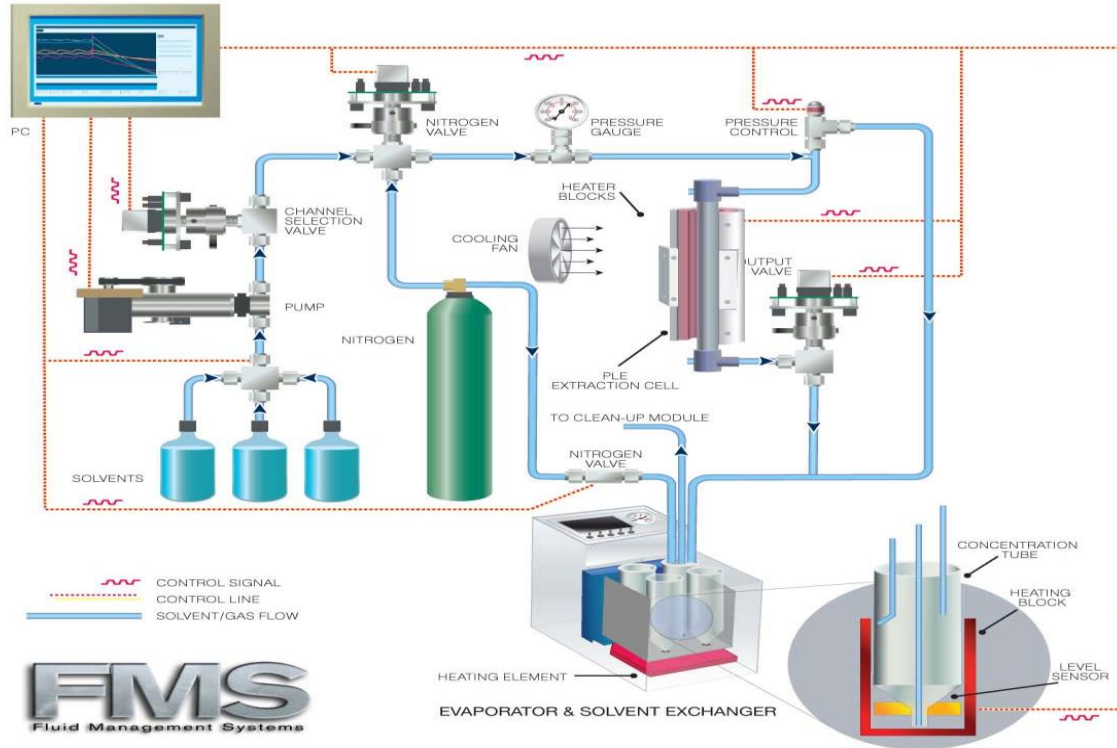
Automation

- Advantages of automated sample prep are:

- Reduced time:
  - Automated Pressurized Liquid Extraction (PLE) takes 60 min start-to-finish
  - Manual 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.

PLE® Fast Extraction & Concentration System

SuperVap Evaporation

- System pre-heated to 45-60 ⁰C.

- 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.

Automating Sample Prep

- Automated FMS Pressurized Liquid Extraction (PLE) for sample extraction is fast (60 min), efficient (120 ^{o}C, 1500 psi), green (less power), reliable (long track record).

- FMS 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.

- Users' choice of FMS fully automated or semi automated clean up system: fast (20-60 min), low solvent usage

PowerPrep Clean-up

- Dioxin / PCB / PBDE Cleanup and Fractionation
  - Silica (Jumbo / Classic/ ABN)
  - Alumina
  - Carbon

- Florisil cleanup

- Custom Clean-up
 - Ability to combine and manufacture any mix of columns

System Flexibility

- With the combination of the PLE, SuperVap and PowerPrep NG you are given the flexibility to automate your extraction, concentration and clean-up for a wide variety of compounds.

- This is an ideal system for:
  - Laboratories that receive varying, different sample types and compounds of interest

  - High volume labs with a consistent daily sample amount load

  - Research laboratories looking to improve efficiency, recoveries and reproducibility
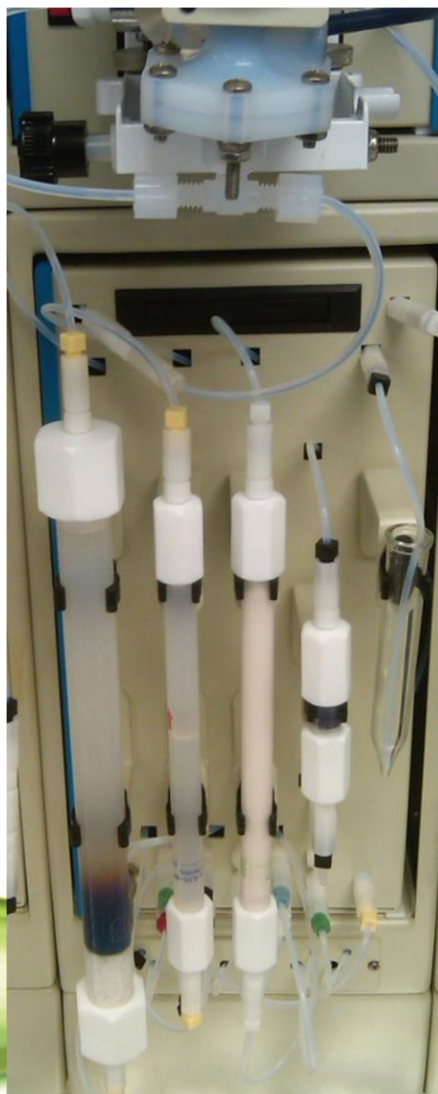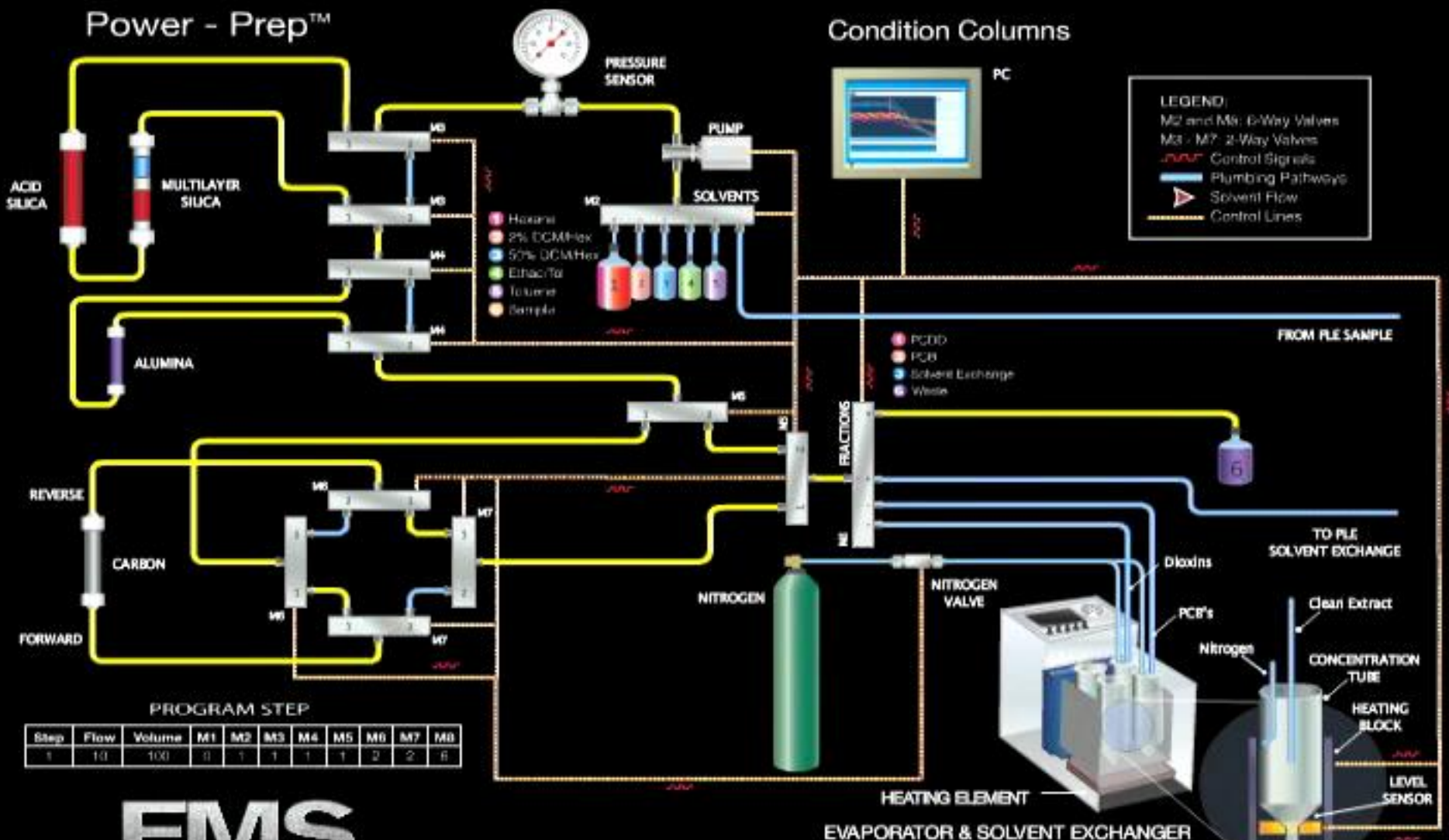
System characteristics

- 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 up to 10-15 mL/min are used.

- Modular and Expandable 1 to 6 modules (samples)

- Up to 3 Fractions

Columns

- Silica - PCB/PBDE-free Acid, Base and Neutral silica gel column (mini, classical, classical plus, high capacity, XL).

- Alumina – PCB/PBDE-free basic alumina column.

- Carbon – PCB/PBDE-free carbon/celite column.

- Packed in disposable Snap Teflon tubes; individually sealed in Mylar packaging; production in clean room environment.

Program

- Condition columns with hexane (step 1-3).
- Load sample in hexane onto silica (step 4).
- Elute silica column with hexane, analytes onto alumina (step 5).
- Flush with 10% DCM/hexane (step 6).
- Elute alumina with 10% DCM/hexane, collect all PCBs (F1, step 7).
- Flush system with DCM (step 8).
- Elute alumina with DCM, PCDD/Fs onto carbon (step 9).
- Flush with toluene (step 10) and elute carbon with toluene (step 11). Collect all PCDD/Fs (F2).
- Hexane purge (step 12).
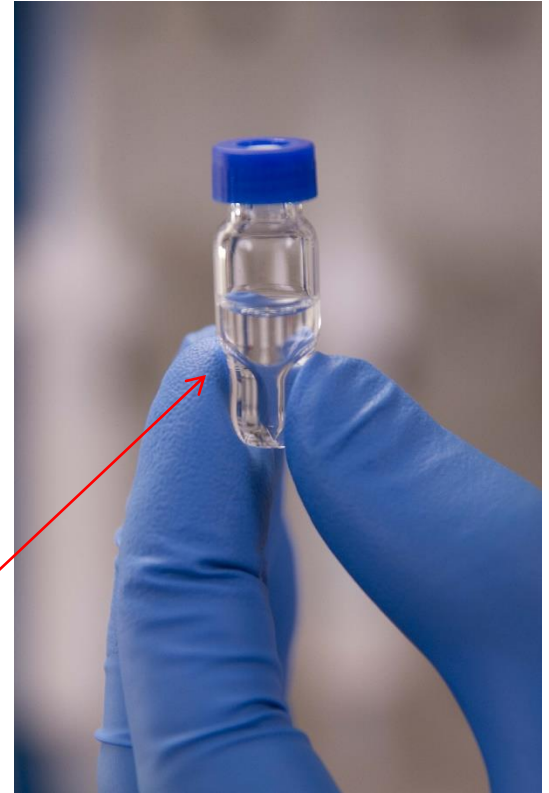
SuperVap Concentrator

SuperVap Evaporation

- System pre-heated to 45-60 ºC.

- Samples evaporated at stable T under 8 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.
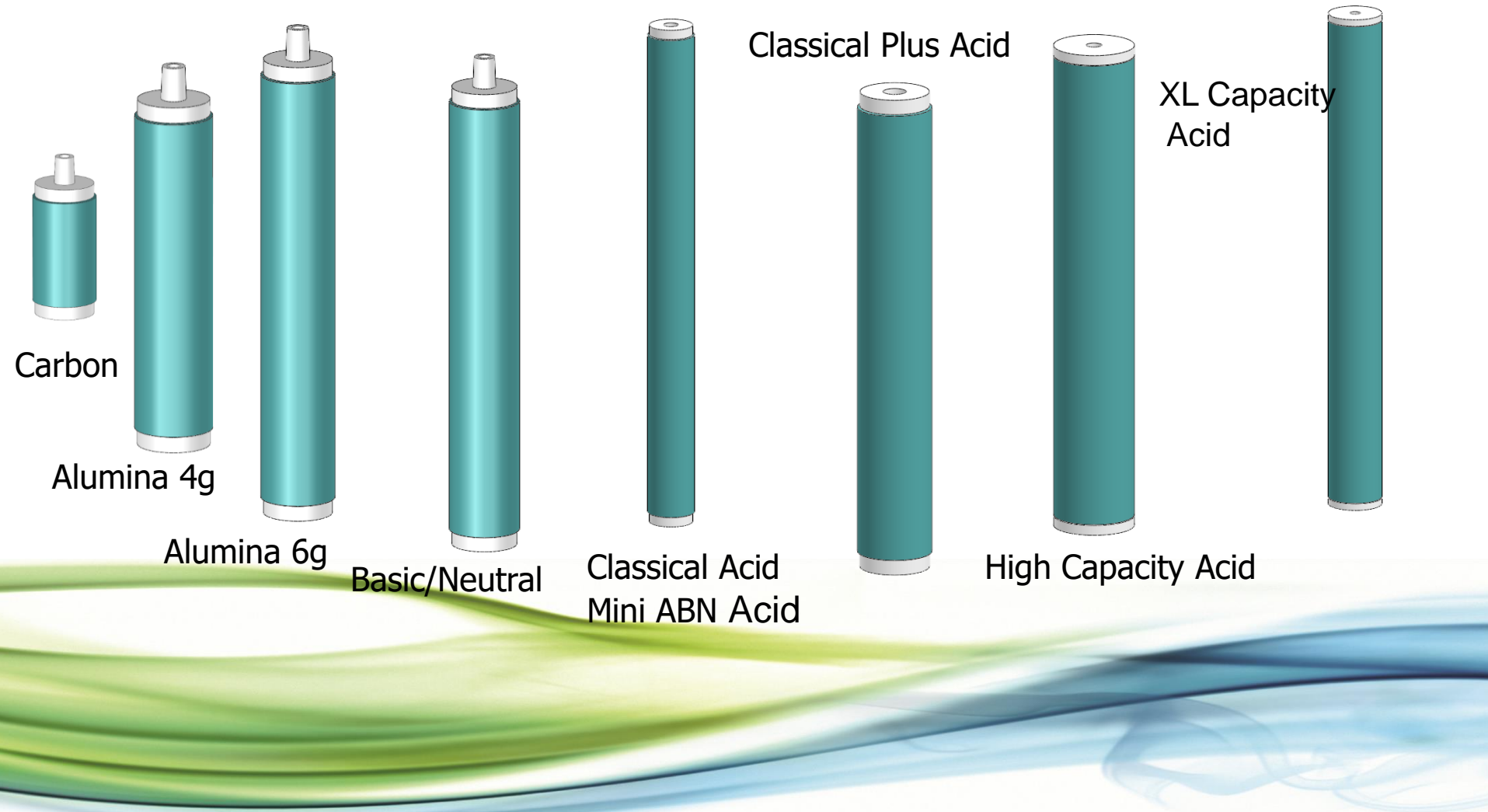
Glass Evaporation tube

GC vial

Prepped Samples

- Various Oil matrices obtained (Lard, Olive Oil, Corn Oil, Cod Oil, Red Palm Oil, Unrefined Pumpkin Oil, Unrefined Vegetable Oil).

- Aliquots of 5 gram samples were spiked with 13C labeled surrogate standards.

- Samples were diluted into n-hexane and drawn up into a gas tight syringe.

Columns

Carbon

Alumina 4g

Alumina 6g

Basic/Neutral

Classical Acid
Mini ABN Acid

Classical Plus Acid

High Capacity Acid

XL Capacity Acid

DFS HRGC/HRMS

Mean PCDD/F recoveries (6 edible oils)

Analyte	Mean	Dev	Blk Conc.
2378TCDF	70	8.5	< .1 pg/g
2378TCDD	78	8.6	< .1 pg/g
12378PeCDF	83	13.5	< .5 pg/g
23478PeCDF	81	10.7	< .5 pg/g
12378PeCDD	81	11.6	< .5 pg/g
123478HxCDF	70	7.1	< .5 pg/g
123678HxCDF	62	3.6	< .5 pg/g
234678HxCDF	71	10.0	< .5 pg/g
123789HxCDF	66	6.9	< .5 pg/g
123478HxCDD	81	11.3	< .5 pg/g
123678HxCDD	77	9.4	< .5 pg/g
123789HxCDD	NA	NA	< .5 pg/g
1234678HpCDF	73	5.0	< .5 pg/g
1234789HpCDF	85	9.0	< .5 pg/g
1234678HpCDD	75	7.1	< .5 pg/g
OCDD	70	3.6	< 1 pg/g
OCDF	NA	NA	< 1 pg/g

Mean PCBs recoveries (6 oils)

	Mean	Dev	Blk Conc.
PCB-77	73	14.9	< .5 pg/g
PCB-81	64	11.0	< .5 pg/g
PCB-105	75	15.2	< .5 pg/g
PCB-114	73	11.4	< .5 pg/g
PCB-118	73	8.5	< .5 pg/g
PCB-123	72	8.0	< .5 pg/g
PCB-126	88	19.7	< .5 pg/g
PCB-156	63	7.4	< .5 pg/g
PCB-157	53	8.7	< .5 pg/g
PCB-167	63	6.1	< .5 pg/g
PCB-169	75	10.4	< .5 pg/g
PCB-170	79	9.4	< .5 pg/g
PCB-180	77	14.2	< .5 pg/g
PCB-189	80	9.8	< .5 pg/g

Conclusions

- Analysis of the 6 Oil matrices processed yielded acceptable recoveries for all analytes with standard deviations below 20%.

- Analysis of an n-Hexane blank sample resulted in no detectable target analytes measured within the calibration range of each respective compound.

- With a total processing time of less than 2.5 hours, the FMS PowerPrep® and SuperVap® Concentrator delivers an efficient, totally automated sample prep process for edible oils.

TotalPrep®

Touch Screen Control

Real Time Display of Program Steps

Online evaporator starts during collection
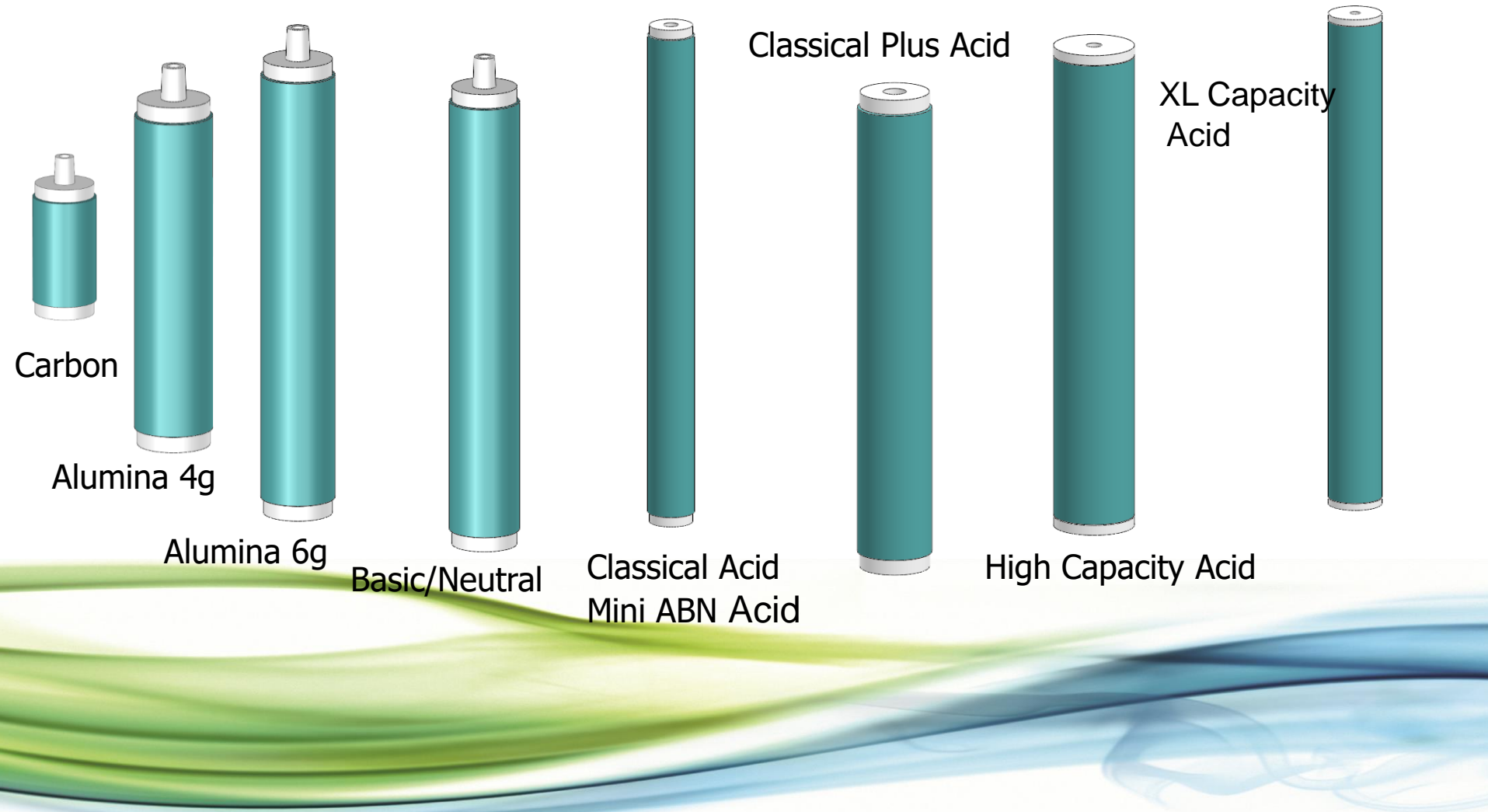
Up to 4 modules, 8 samples parallel

System Characteristics

- Uses Zero DCM

- 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.

- Modular and Expandable 1 to 4 modules (2 samples per module up to 8 samples total)
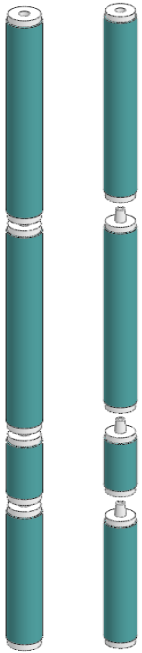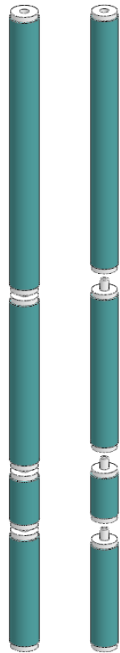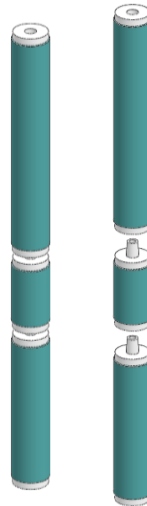
Carbon

Alumina 4g

Alumina 6g

Basic/Neutral

Classical Acid
Mini ABN Acid

Classical Plus Acid

High Capacity Acid

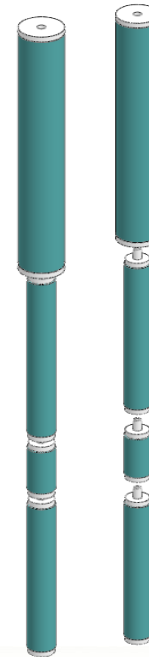XL Capacity Acid

Classical Kit

Classical Plus Kit

Mini Kit

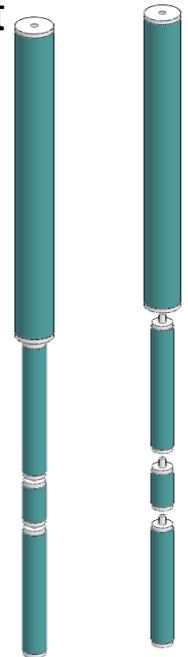**High Capacity Kit**

XL Capacity Kit

Columns

- Silica - PCB/PBDE-free Acid, Base and Neutral silica gel column (mini, classical, classical plus, high capacity, XL).

- Alumina – PCB/PBDE-free basic alumina column.

- Carbon – PCB/PBDE-free carbon/celite column.

- Packed in disposable Teflon tubes; individually sealed in Mylar 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 20-45 min.

- Low volumes 100-250 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 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.

Direct to GC Vial
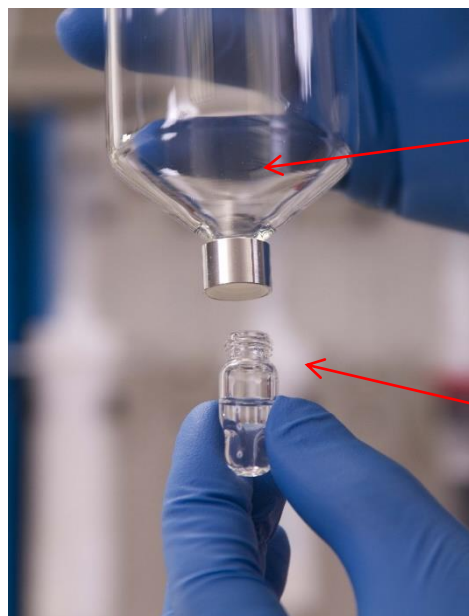
Glass Evaporation tube

GC vial
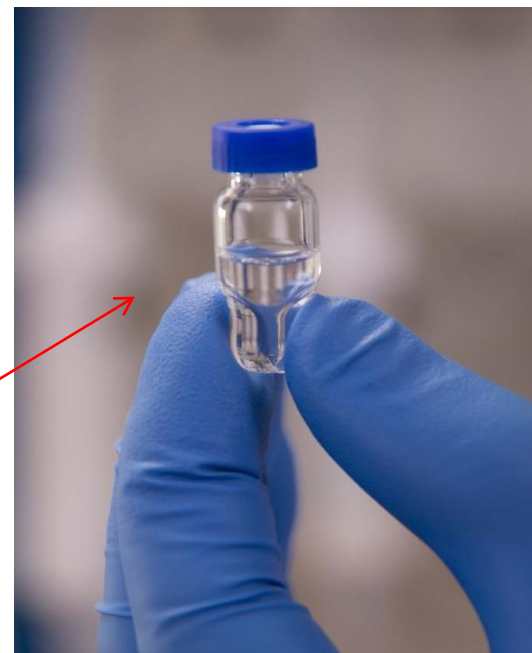
Mini Kit Data

- Used automated SPE for serum extraction

- Clean up with mini silica, carbon, mini alumina

- Total time for clean up 40 min

- Total solvent volume 100 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 45 min

- Total solvent volume 250 mLs

- No DCM

13C Recoveries DD/F Matrices

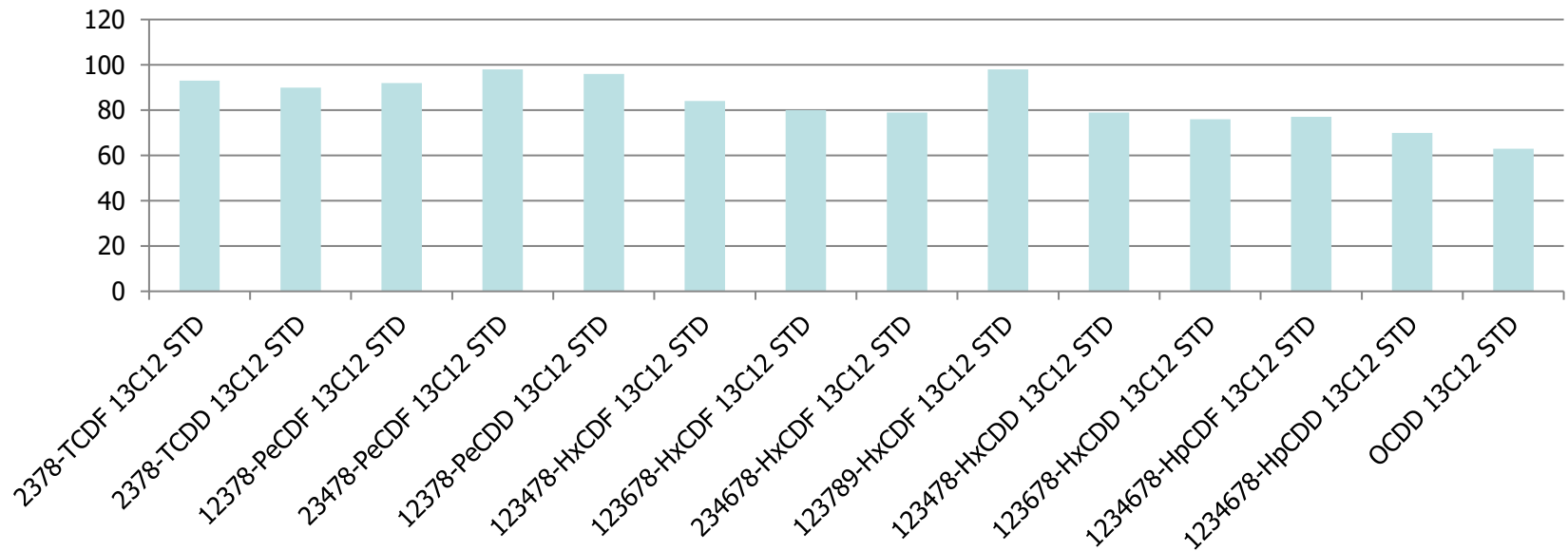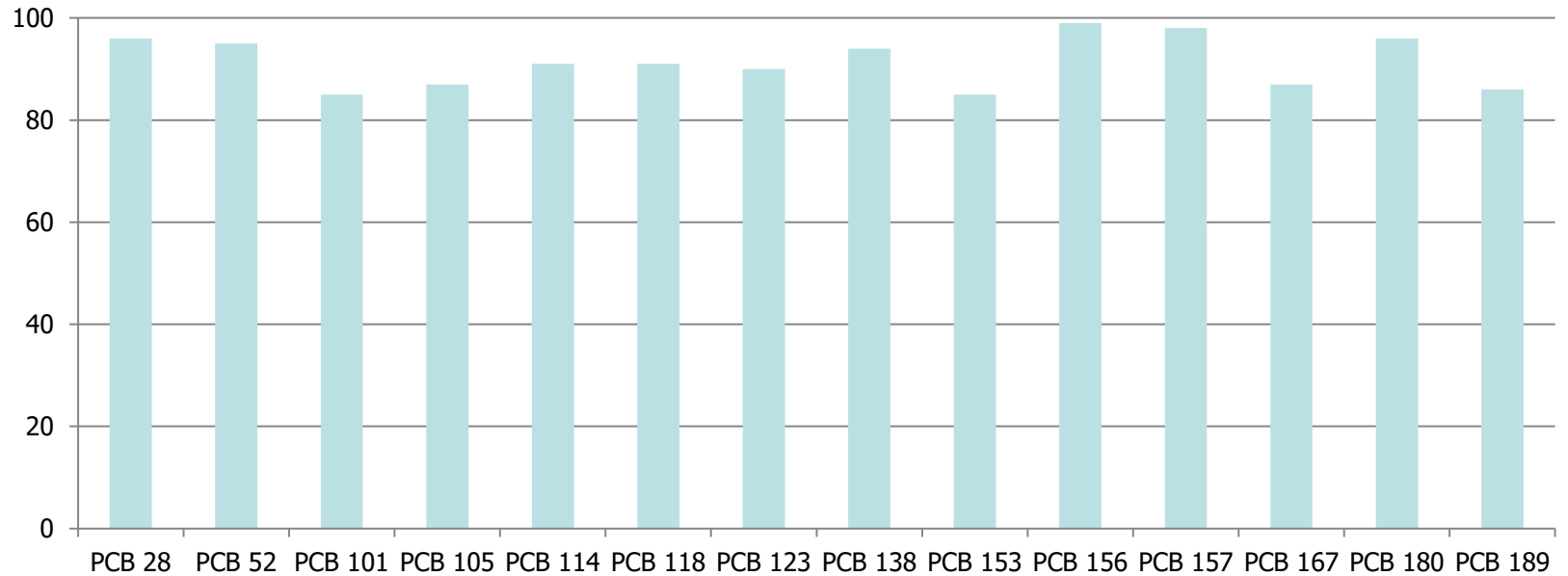| Compound Name	Sediment 1g	Feed 2g	Egg 8 g	Fish Oil 40 mg	Fatty Acid 2 g	Hexane
2378-TCDF 13C12 STD	78	89	75	69	93	78
2378-TCDD 13C12 STD	92	100	86	96	92	93
12378-PeCDF 13C12 STD	80	91	70	91	97	82
23478-PeCDF 13C12 STD	81	90	70	92	103	80
12378-PeCDD 13C12 STD	91	100	75	105	104	90
123478-HxCDF 13C12 STD	79	95	74	93	92	84
123678-HxCDF 13C12 STD	78	81	76	96	90	85
234678-HxCDF 13C12 STD	83	91	78	87	96	86
123789-HxCDF 13C12 STD	88	93	80	99	90	83
123478-HxCDD 13C12 STD	84	92	77	86	97	88
123678-HxCDD 13C12 STD	73	72	67	83	93	81
1234678-HpCDF 13C12 STD	69	79	68	87	88	69
1234789-HpCDF 13C12 STD	82	71	76	80	92	79
1234678-HpCDD 13C12 STD	87	95	80	98	92	79
OCDD 13C12 STD	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	
PCB 28	66	67	68	60	86
PCB 52	68	69	70	71	86
PCB 77	94	90	101	87	86
PCB 81	88	83	95	93	75
PCB 101	77	78	80	77	87
PCB 105	98	112	90	88	95
PCB 114	108	109	87	89	97
PCB 118	92	110	88	90	89
PCB 123	112	115	82	86	98
PCB 126	92	89	88	79	77
PCB 138	75	74	75	72	90
PCB 153	71	71	71	65	84
PCB 156	100	95	98	94	98
PCB 157	98	90	87	92	91
PCB 167	92	89	87	87	86
PCB 169	na	93	106	103	100
PCB 170	99	94	90	98	100
PCB 180	96	85	88	89	88
PCB 189	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
Dioxins & PCBs in Serum	45	30	30	60	60	225 min
Dioxins & PCBs in Soil	60	30	45	60	60	255 min
Dioxins & PCBs in Fatty Foods	60	30	45	60	60	255 min
Dioxins & PCBs in Oil	0	0	45	60	60	165 min

Free EP-110 Program requirements

- EP-110 Controller and Module
  - Capable of running 2 samples in parallel

- Requirements 2 sample system
  - Purchase 1,200 Classic Kits
  - Purchase 1,000 High-Capacity Kits
    - Contact us for pricing

Free EP-110 Program requirements

- Free EP110 4 sample system
  - Purchase 2400 Column Kits

- Free EP110 6 sample system
  - Purchase 3600 Column Kits

- One-year warranty included
- Service Plans are available
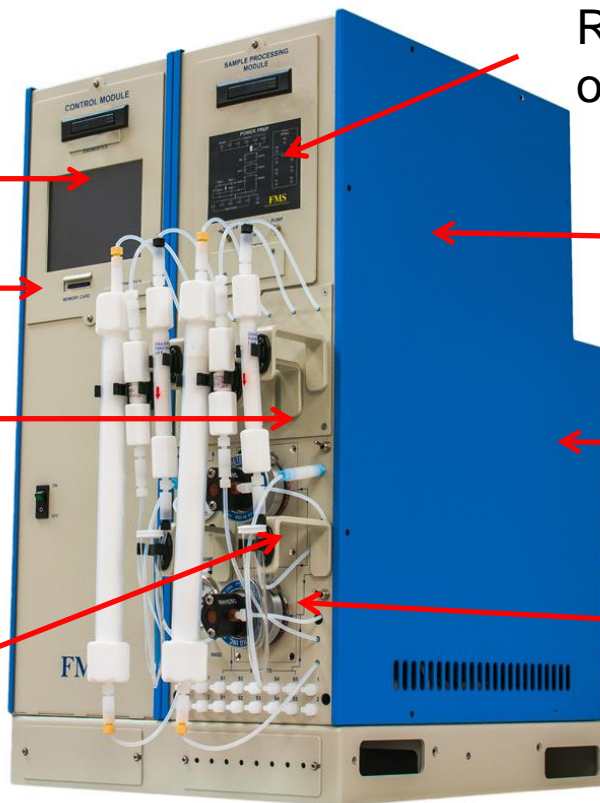- Installation and Training is not included.
  - $6,500

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 power use.**

- **Clean up step time between 30 and 45 min.**

- **EP-110 uses no DCM.**

- **Low solvent use 100-250 mLs.**

- **Total time from sample till data between 3-4.5 hrs.**

- **PLE/SPE and EP-110 can be purchased in one system.**

EconoPrep® for traditional Pops Sample Cleanup

FMS Fluid Management Systems

Real Time Graphical Display of Extraction Steps

Touch Screen Control

Clean Up and Fractionate up to Eight Extracts in Parallel

SD Card for Storing and Transferring Methods

2 Sample Positions per module

Direct to Vial Concentration

Automatic and Manual Sample Loading

Positive Pressure Pumps for precise, consistent delivery of Sample and Solvent

- **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

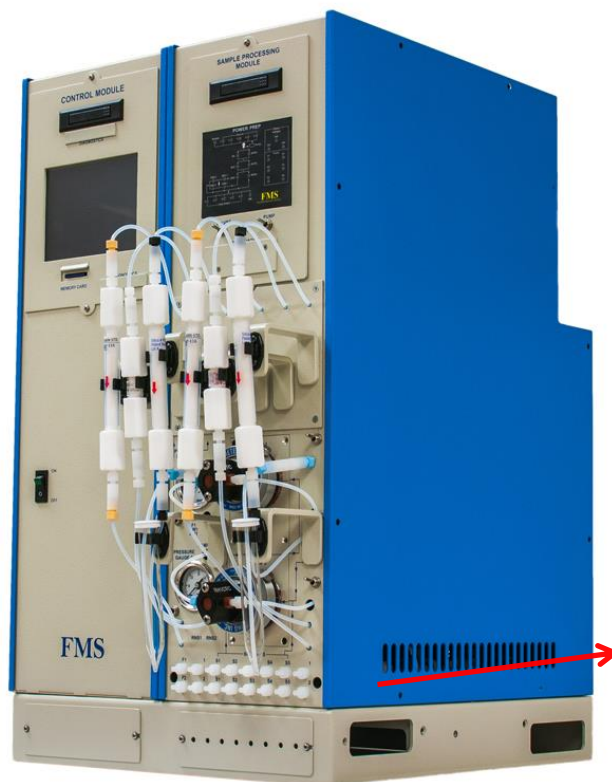**EconoPrep 4
Capable of running
8 Samples in Parallel**

Columns

- Silica - PCB/PBDE-free Acid, Base and Neutral silica gel column (mini, classical, classical plus, high capacity, XL).

- Alumina – PCB/PBDE-free basic alumina column.

- Carbon – PCB/PBDE-free carbon/celite column.

- Packed in disposable Teflon tubes; individually sealed in Mylar packaging; production in clean room environment.

EconoPrep Classic

Total run time
30 minutes

Classical
Up to 0.4 gm
lipid

PCB / Dioxin
Fractions

EconoPrep HC

Total run time
40 minutes

High
Capacity
Up to 5 gms
lipid

PCB / Dioxin
Fractions

- 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).

- Hexane, DCM and toluene used as solvents.

- Solvent use between 100-335 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.

Glass Evaporation tube

GC vial

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

PCB / Dioxin Fractions

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

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

PCB / Dioxin
Fractions

High Capacity PCDD/F data

	Animal Feed	Peanut Butter	Corn Oil	Olive Oil
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

PCB / Dioxin Fractions

EconoPrep Systems

- Reduced Solvent

- Reduced Power Consumption

- Lower Cost System and Consumables

- Simultaneously perform eight sample extractions, cleanups, and concentrations in up to 2 hours

- Unattended sample preparation

- Same day turn around for multiple samples

EconoPrep Systems

- Automates EPA SW846 and 40 CFR 136 methods

- Reduce solvent and glassware usage

- Dramatic savings in time and resources

- Automated Closed System produces Low background

- Economical Disposable Column Kits used for Dioxins and PCBs, PBDEs analysis

Conclusions (1)

- Total EconoPrep delivers low cost, 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 100-335 mLs depending on application.

- Extraction, Cleanup and Concentration 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.

Simple, Quick & Low Cost Semi-Automated Clean-Up for Dioxins/PCBs

Automated Sample Prep

- **Advantages of Automated Sample Prep**
  - Rapid Turn Around Time: 30 to 60 Minutes for 6 Samples
  - Cleaner Background Interferences: Closed Loop System
  - Quality Results: Certified Pre-packaged Columns
  - Green Technology: Lower solvent and power use
  - QA/QC & Accreditation Requirements: Easier to Manage
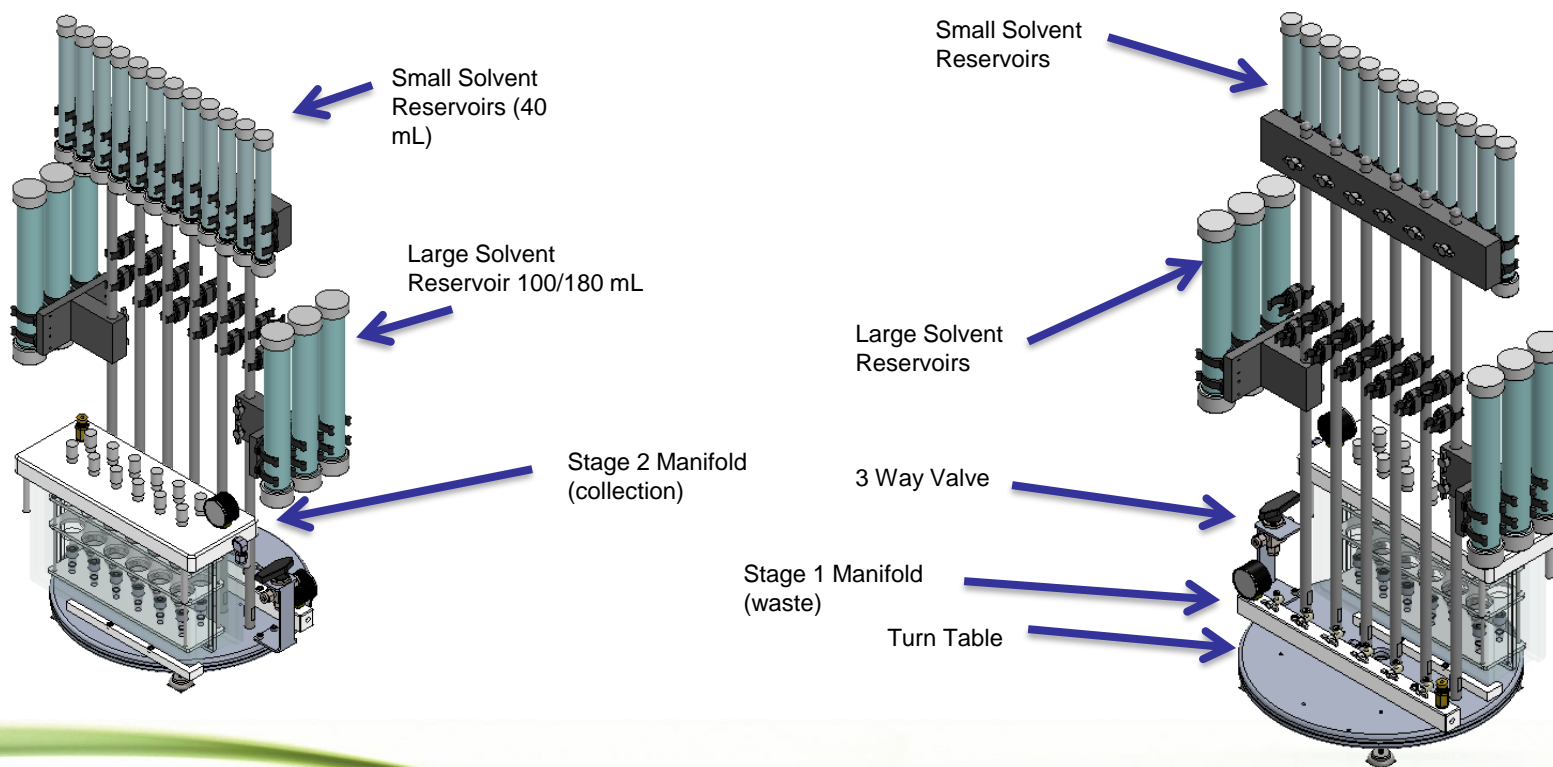 - Computerized Method: Instrumentation based prep

- **A**dvantages of Manual Sample Prep
  - Most labs use a Manual Methods for the following reasons:

    - No electronics or mechanical components to fail
    - No down time due to the system failure
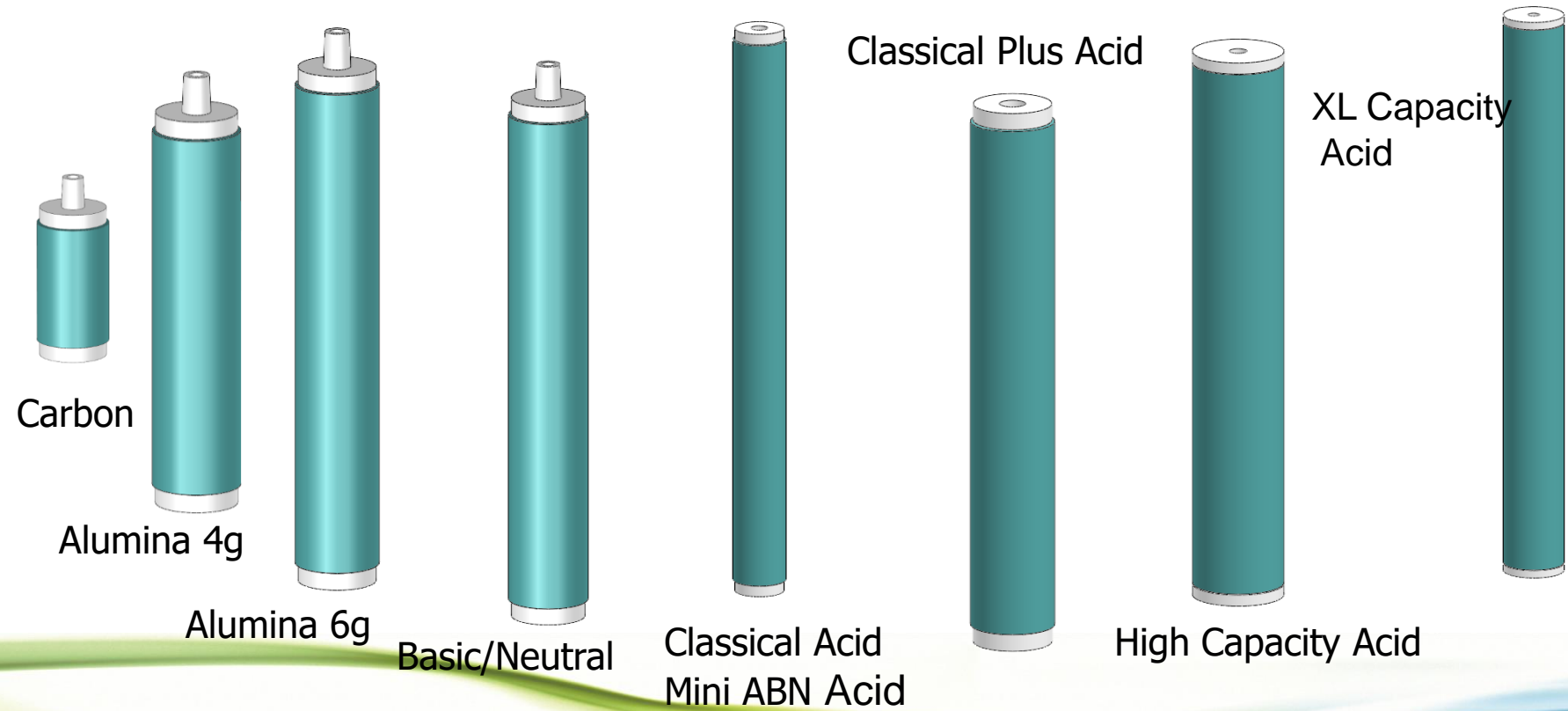   - No service contract
    - No capital equipment cost

Specification:

- Simple to run, no computerized instrumentation
- Fast: 60 min
- Closed loop system to give a clean background, low level detection
- Use certified pre-packaged columns
- Green technology, only vacuum pump uses power
- Low solvents, as low as 100 mL for serum
- Economical column kits, choice of low fat and high fat column kits
- No capital equipment cost
- No electronics or mechanical equipment to fail
- No downtime

Small Solvent Reservoirs (40 mL)

Large Solvent Reservoir 100/180 mL

Stage 2 Manifold (collection)

Small Solvent Reservoirs

Large Solvent Reservoirs

3 Way Valve

Stage 1 Manifold (waste)

Turn Table

Carbon

Alumina 4g

Alumina 6g

Basic/Neutral
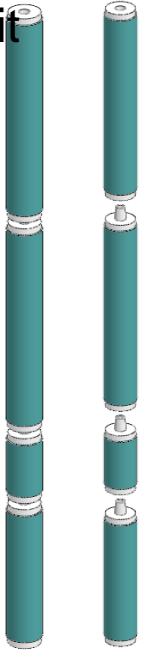
Classical Acid
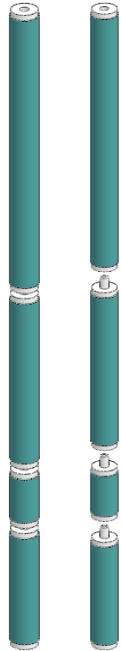Mini ABN Acid

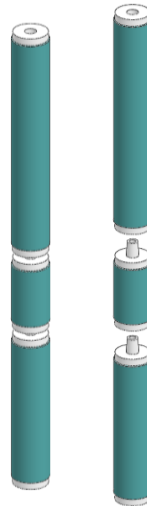Classical Plus Acid

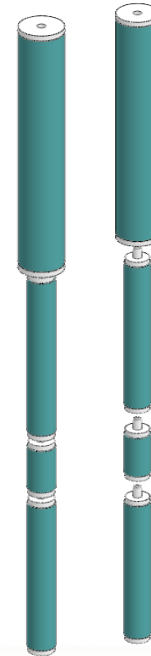High Capacity Acid

XL Capacity Acid

Columns (2)

Classical Kit

Classical Plus Kit

Mini Kit

High Capacity Kit
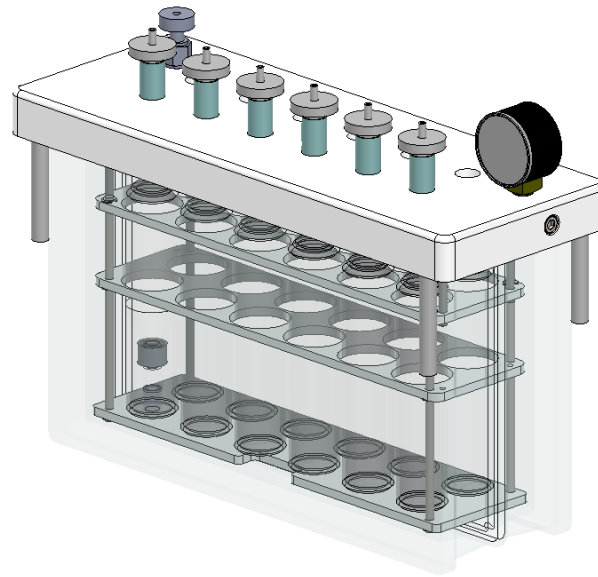
XL Capacity Kit

Vacuum Gauge

Stopcock

Fitting to 3 Way Valve

Vacuum Relief Valve

Solvents

Vacuum gauge

Waste

Column assembly

Vacuum pump

Rotatory work station

Columns ready for elution

Manifold with collection tubes
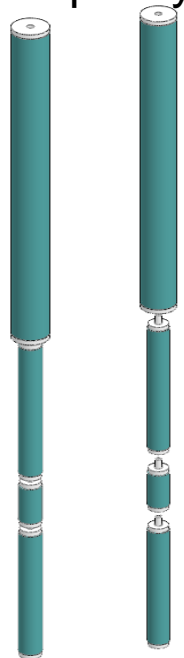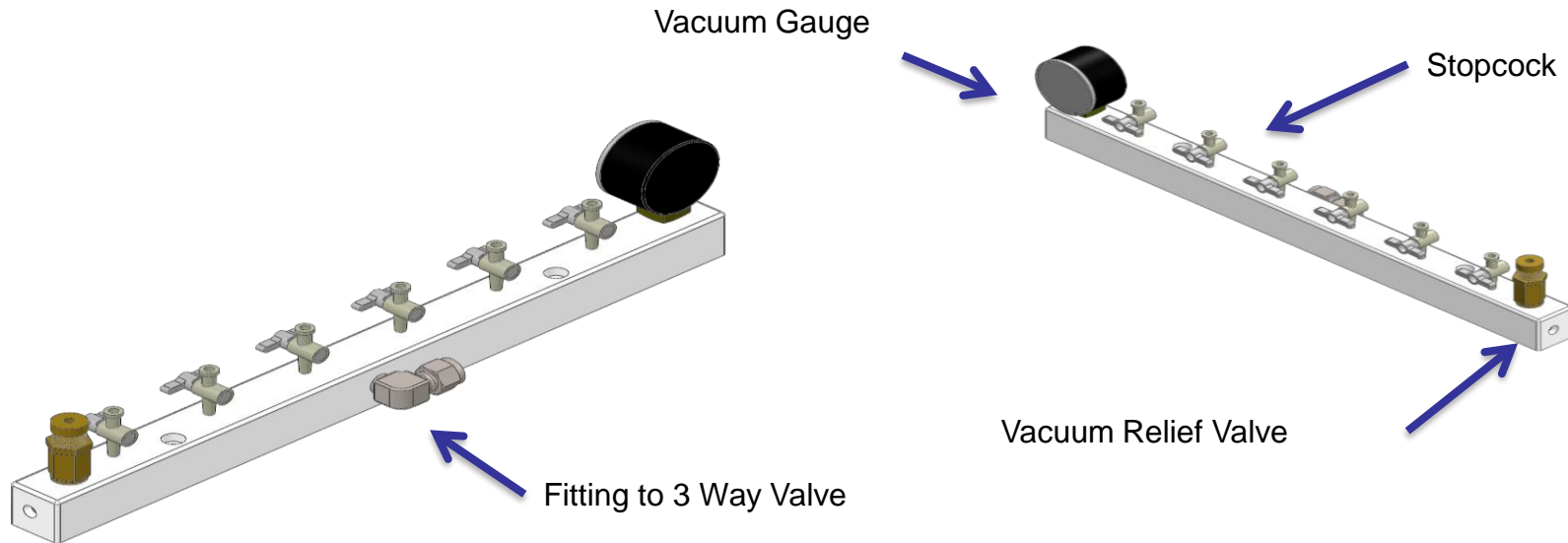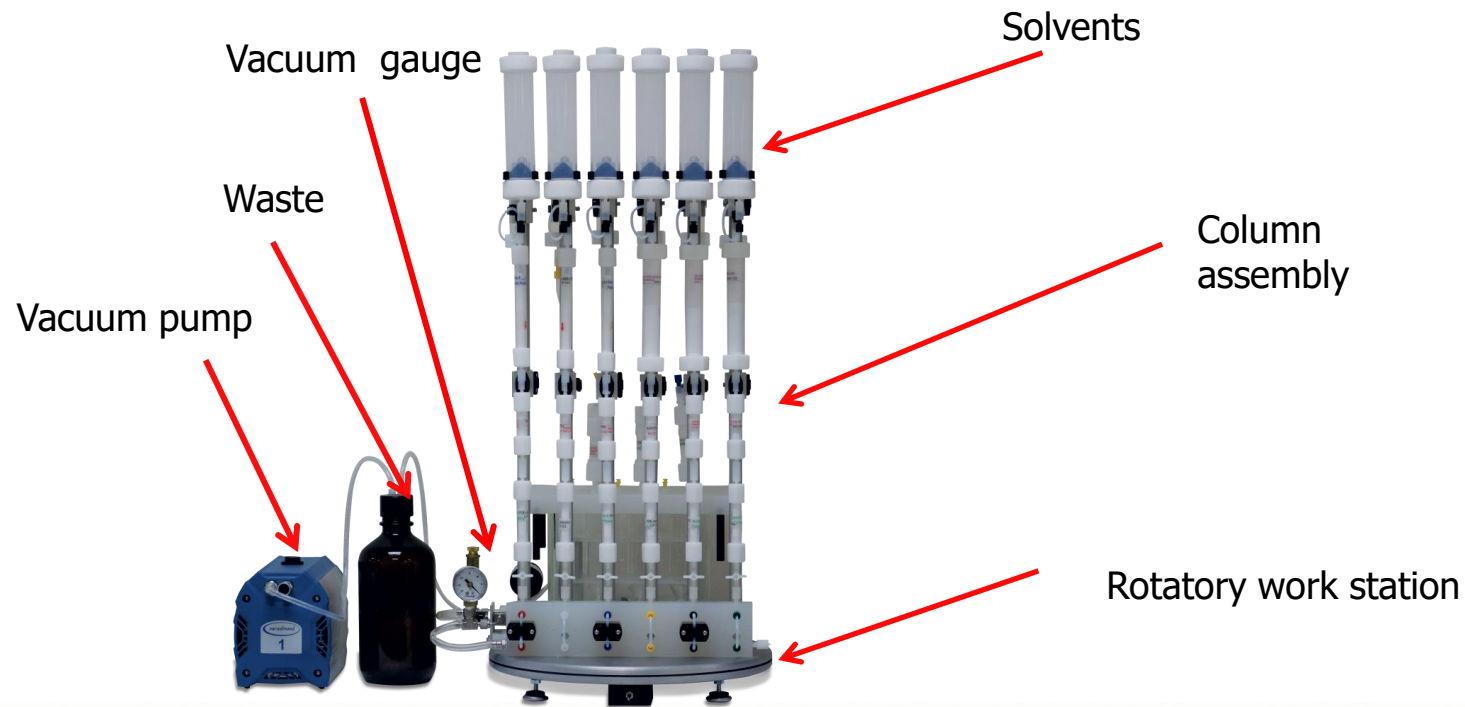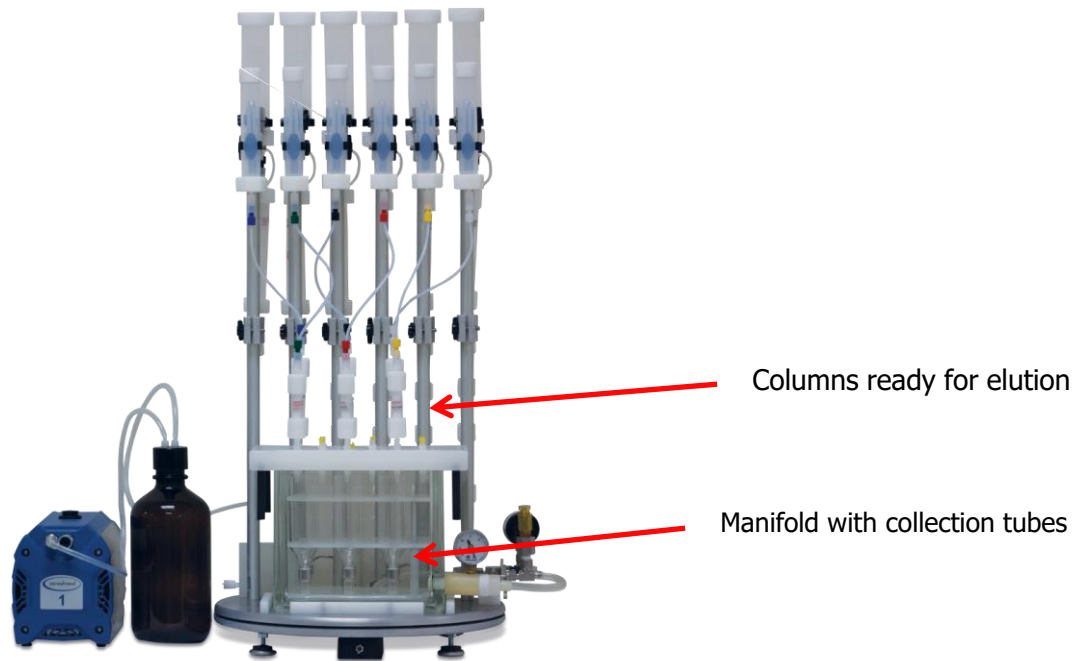
Attributes (1)

- **Closed loop system:**
  - **Eliminates background contaminants**
  - **No washing needed.**
  - **Capped solvent reservoirs**

- **Optimized for solvent reduction while obtaining highest possible recoveries**

- **Uses Hexane and Toluene, no Dichloromethane**

- **Easy sample loading on top of silica column via syringe vial**

- **Columns connect easy with SNAP connections**

Attributes (2)

- **Order of columns is Silica-Carbon-Alumina**

- **Columns are assembled and vacuum turned on**

- **Conditioning on top of silica column via syringe vial (Stage 1 to waste, use 20-60 mL hexane through all columns)**

- **Load sample into syringe vials and pull through column assembly (Stage 1, waste)**

- **Elute columns with hexane (80-180 mL) and transfer all target compounds to carbon and alumina (Stage 1, waste)**

- **Discard silica columns and remove carbon and alumina columns**

- **Rotate turntable (Stage 2)**
  - Install carbon and alumina columns
  - Elute carbon and alumina columns in reverse each individually with 40 mL toluene and collect
  - Collect Carbon Fraction 1 with PCDD/F and co-PCBs
  - Collect Alumina Fraction 2 with mono- and di-ortho PCBs (and PBDEs)
  - Collection step ~ 3-5 min

SuperVap Evaporation

- System pre-heated to 55-60 ºC.

- Samples evaporated at stable T under 6-8 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.

Direct-to-Vial

GC vial

^{13}C PCBs Recoveries EZPrep (%)

		Soil	Feed	Egg yolk	Olive oil	Fish oil	Hexane
		5 g	10 g	18 g	2 g	2 g	
PCB 28		93	104	72	103	100	95
PCB 52		90	108	70	100	97	95
PCB 77		90	103	120	98	102	108
PCB 81		92	99	62	102	98	92
PCB 101		93	110	73	106	102	98
PCB 105		108	101	62	110	104	106
PCB 114		111	102	65	105	97	104
PCB 118		86	103	60	91	89	102
PCB 123		106	97	70	92	96	93
PCB 126		107	102	90	102	98	115
PCB 138		104	96	77	92	110	111
PCB 153		101	102	69	102	114	102
PCB 156		102	99	60	113	104	105
PCB 157		93	97	61	103	99	108
PCB 167		119	106	60	105	105	107
PCB 169		98	98	81	96	96	117
PCB 170		103	107	80	103	105	117
PCB 180		98	106	85	102	102	107
PCB 189		108	97	63	95	88	107

^{13}C PCDD/F Recoveries EZPrep (%)

	Soil 5 g	Feed 10 g	Egg yolk 18 g	Olive oil 2 g	Fish oil 2 g	Hexane
2378-TCDF	95	92	83	96	89	89
2378-TCDD	104	101	70	101	96	101
12378-PeCDF	86	92	85	97	78	80
23478-PeCDF	102	94	69	102	98	91
12378-PeCDD	85	93	75	60	71	100
123478-HxCDF	88	105	79	92	92	95
123678-HxCDF	103	109	80	102	94	99
234678-HxCDF	73	66	80	60	95	104
123789-HxCDF	107	92	92	95	89	95
123478-HxCDD	107	95	79	95	92	95
123789-HxCDD	82	84	87	81	86	91
1234678-HpCDF	76	82	82	83	87	87
1234789-HpCDF	91	84	93	84	81	84
1234678-HpCDD	76	80	87	82	79	74
OCDD	60	67	60	60	91	70

2 g Fish Oil pg/g

	EP-110	EZPrep
PCB 28	6398	6324
PCB 52	9549	10150
PCB 118	7566	7542
PCB 138	17816	19270
PCB 156	657	616
PCB 157	208	227
PCB 167	540	501
PCB 170	2013	1994
PCB 189	64	50

EZPrep Conclusions

FMS Fluid Management Systems

- EZprep123 is low solvent and fast clean up system (30 - 75 min)
  - 160 mL for low fat (~ 0.15 g fat, mini kit serum)
  - 260 mL for high fat (up to 5 g fat, Extra-HC kit)

- High sample throughput → 18 samples/hour
  - 6 samples in parallel per station
  - 3 stations fit in one hood

- System gives excellent recoveries for PCDD/F, PCB and PBDEs comparable to FMS automated systems

- Use of certified pre-packaged columns guarantees low native background

EZPrep Conclusions

- No DCM used in clean up

- No worries about breakdown or downtime

- No washing needed

- No cross-contamination

- Low cost

FMS Flexible Dioxin, PCB and PBDE Sample Cleanup Solutions

- There are many different requirements in POPs Sample Prep for Analysis

- FMS has Total System Solutions to meet all or any of those requirements

- PLE (Pressurized Liquid Extraction)
  - Reduced time:
    - Automated Pressurized Liquid Extraction (PLE) takes 60 min start-to-finish
    - Manual Soxhlet up to 36 h.
    - Up to 8 samples in Parallel
  - Reduced cost: less labor involved, shorter turnover time per sample, less electricity use for PLE than Soxhlet.
  - Reduced volume: less solvent used.

- **PowerPrep NG**
  - Millions of POPs samples have been processed on the PowerPrep platform
  - Superior Flexibility for Chemistry types, Analyte Fractionation and Programabilty

- With the combination of the PLE, SuperVap and PowerPrep NG you are given the flexibility to automate your extraction, concentration and clean-up for a wide variety of compounds.

- This is an ideal system for:
  - Laboratories that receive varying, different sample types and compounds of interest
  - High volume labs with a consistent daily sample amount load
  - Research laboratories looking to improve efficiency, recoveries and reproducibility

- **EP110**
  - Uses No DCM
  - Low solvent use 100-250 mLs
  - Total time from sample till data between 3-4.5 hrs
  - Clean up step time between 30 and 45 min
  - Modular 2 to 8 sample configurations in parallel

- **PLE/SPE and EP-110 can be configured in one system**

- **Rental Programs**

- **Economically Priced automated solutions for any Sample Matrix type**

FMS Flexible Dioxin, PCB and PBDE Sample Cleanup Solutions

- **EconoPrep**
  - Extraction, Cleanup and Concentration can be done in 3-4 hours
  - Different plumbing available for food (PCBs in two fractions) and environmental (all PCBs together)
  - 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
  - Low solvent use 100-250 mLs
  - Clean up step time between 30 and 45 min
  - Modular 2 to 8 sample configurations in parallel

- **PLE/SPE and EconoPrep can be configured in one system**

- **Rental Programs**

- **Economically Priced automated solutions for any Sample Matrix type**

- **EZPrep 123**
    - No DCM used in clean up
    - No worries about breakdown or downtime
    - No washing needed
    - No cross-contamination
    - Low cost
    - Low solvent use 100-250 mLs
    - Clean up step time between 30 and 45 min
    - 6 samples in parallel
    - System gives excellent recoveries for PCDD/F, PCB and PBDEs comparable to FMS automated systems
    - Use of certified pre-packaged columns guarantees low native background
- **Rental Programs**

- **Economically Priced Solutions for any Sample Matrix type**