

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

Fluid Management Systems
Watertown MA USA



### **FMS**

- FMS Fluid Management Systems
  - Founded in 1986

 Manufactures Total Solution Sample Preparation and Consumables for GC, GC/MS, LC and LC/MS



### Made in the USA







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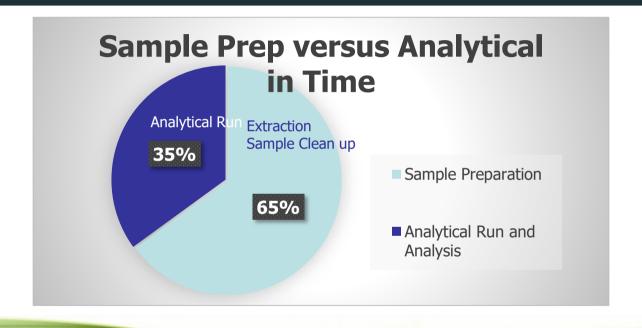


# Class 1000 Cleanroom for Consumables Manufacturing





### Laboratory Workflow Breakdown





#### Introduction

- POPs (PCDD/Fs, PCBs) continue to attract interest around the world due to strict regulations enforced in many countries
- Rapid and quality sample clean up and analysis is needed for many laboratories processing samples
- Processing times and cost are important considerations
- In US EPA methods 1613 and 1668 are used



#### 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:
 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



### Manual Sample 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



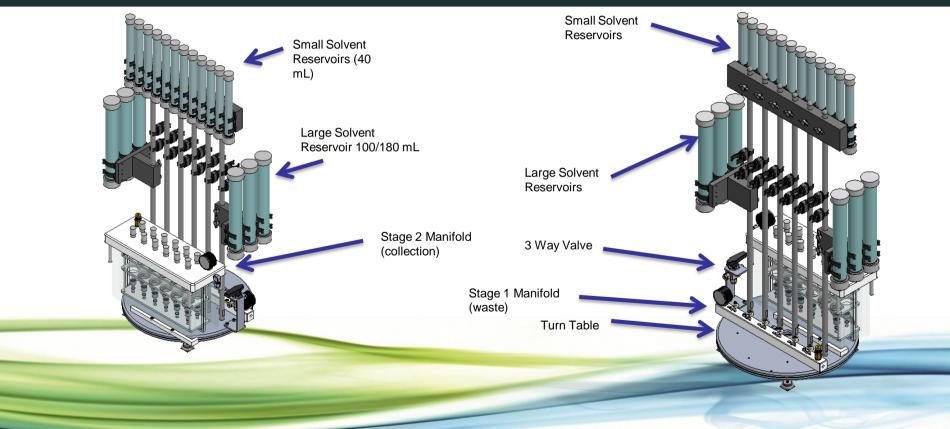
### Semi-Automated System

#### Specification: .

- Simple to Run, Self Installable
- Fast: 35 to 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 160 mL for serum
- Economical column kits, choice of low fat and high fat column kits
- No capital equipment cost
- No electronics, computer or mechanical equipment to fail
- No downtime

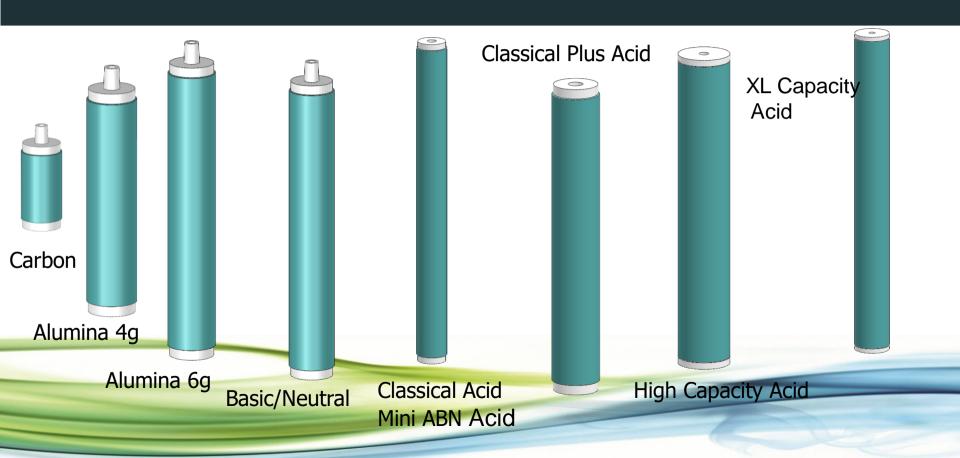


#### Characteristics of Semi-Automated System (EZPrep)



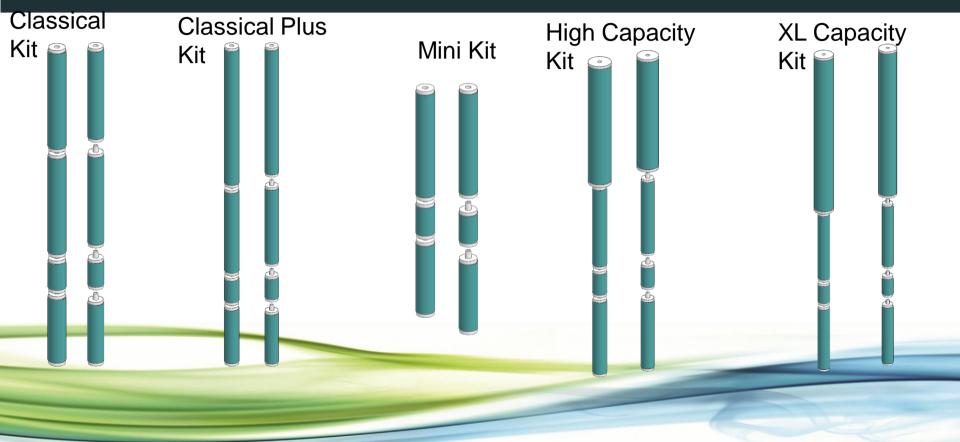


### Columns (1)



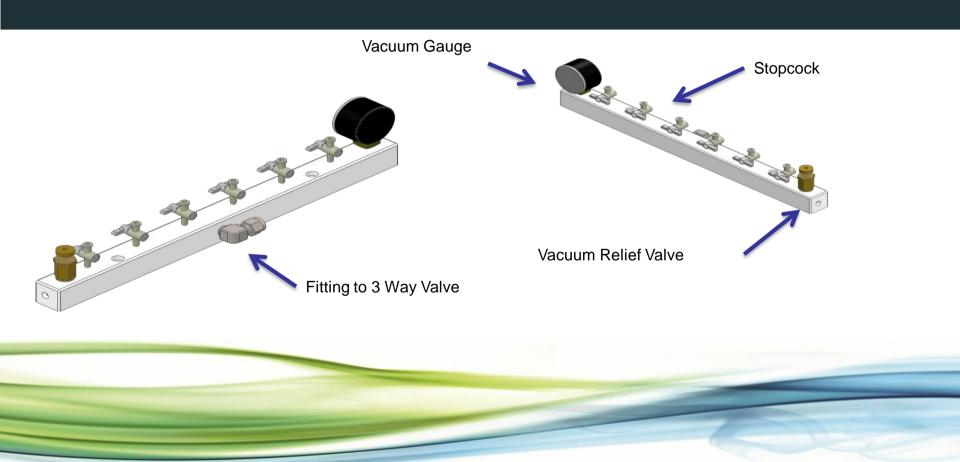


### Columns (2)





#### Stage 1 Manifold



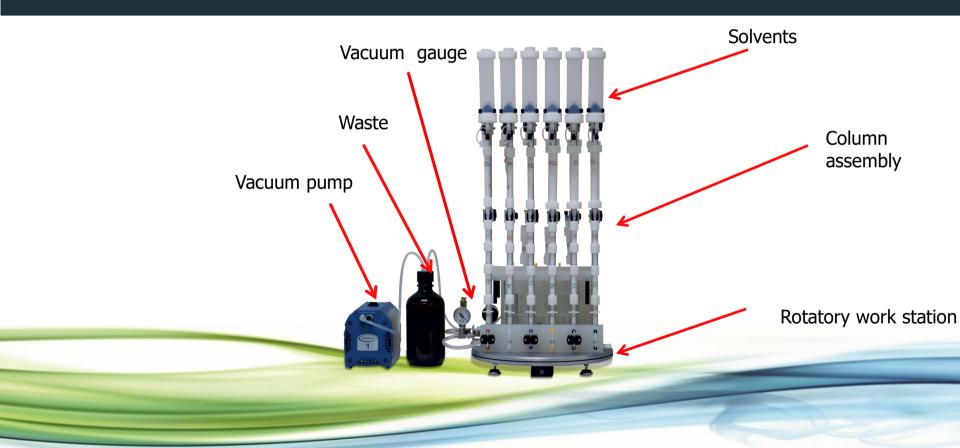


### Stage 2 Manifold





### Stage 1: to waste





### Stage 2: collect





### 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



### Attributes (3)

- > 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
  - Collection vessels can be SuperVap 50ml vessels that go directly to the SuperVap



# Column Kits with various fat removal capacities

		STAGE 1			STAGE 2		
					PCBs	Dioxins	
	Fat Removal	Hexane	Hexane	Hexane	Toluene	Toluene	
Column kits	Capacity	conditioning (mL)	sample volume (mL)	Elute Silica (mL)	Reverse Almina (mL)	Reverse Carbon (mL)	Time (min)
Mini kit	0.15 g	20	10	80	40	40	30
Classical kit	0.5 g	20	10	90	40	40	30
Classical Plus	1.0 g	20	10	100	40	40	40
High Capacity	2.5 g	40	30	160	40	40	60
Extra high Capacity	5.0 g	60	30	180	40	40	75



#### **Direct to Vial Concentration**

# Direct to Vial Concentration SuperVap – Concentration System

- 6 Position 25ml Vessel
- 12 Position 50ml Vessel
- 12 Position 20,40,60 ml vial
- 24 Position 2 and 4 ml vial
- 24 Position PFC 15ml Conical tube







#### SuperVap Features

- Self Installable
  - Video unpacking, installation and training video
- Preprogrammed with most common temperature settings
- 6 (250mL) and 12 (50mL) position models for extractions, direct-to-vial connections
- 24 (2ml) vial
- Dry bath heating element
- Independent secondary heater for extract nipple (can be disabled)
- Sensor or time controlled



### 12 position evaporator 50 mL





### SuperVap Evaporation

- System pre-heated to 55-60 °C.
- 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.



### 24 position Vial evaporator 1-2 mL





### Direct-to-Vial





GC vial



### DFS HRGC/HRMS





#### <sup>13</sup>C PCBs Recoveries EZPrep (%)

	Soil	Feed	Egg yolk	Olive oil	Fish oil	Hexane
	5 g	10 g	18 g	2 g	2 g	Пехапе
DCD 30	93	10 g 104	72	103	100	95
PCB 28		-				
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



### <sup>13</sup>C PCDD/F Recoveries EZPrep (%)

	Soil	Feed	Egg yolk	Olive oil	Fish oil	Hexane
	5 g	<b>10</b> g	18 g	2 g	2 g	
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



## Comparison Native Data with Automated System

#### 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



## <sup>13</sup>C PBDEs Recoveries in Sediment with EZPrep (%)

	Sediment
	<b>1</b> g
BDE-28	67
BDE-47	71
BDE-99	81
BDE-100	80
BDE-153	79
BDE-154	77
BDE-183	80
BDE-209	60



## Free EZPrep Program

- Requirements to get a free EZPrep
  - Purchase 500 sets of columns in 1 and 2 year

• Column Kit pricing is with standard price.



## Free EZPrep Program

- The Column Kits are:
  - Mini kit 0.15gm Fat removal
  - Classical kit 0.5gm Fat removal
  - Classical Plus 1gm Fat Removal
  - High Capacity 2.5gm Fat Removal
  - Extra High Capacity 6gm Fat Removal



# Free EZPrep Program

How to get this offer:

- Contact:
  - sales@fms-inc.com



# EZPrep and SuperVap Conclusions

- ➤ No DCM used in clean up
- > Self Installable, No worries about breakdown or downtime
- No washing needed
- No cross-contamination
- Low cost total solution for Cleanup and Concentration of Samples



### Conclusions (2)

- > 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



# Questions