Quick and Reliable Method for Cleanup of All 209 PCBs in One Fraction in Environmental Samples

Fluid Management Systems
Watertown MA

Introduction (1)

- **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 the US, EPA methods SW-846, 1668C and 8082A are used for PCBs**

Introduction (2)

- **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**
- **Dioxins and furans can also be run**

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

Manual Sample Prep

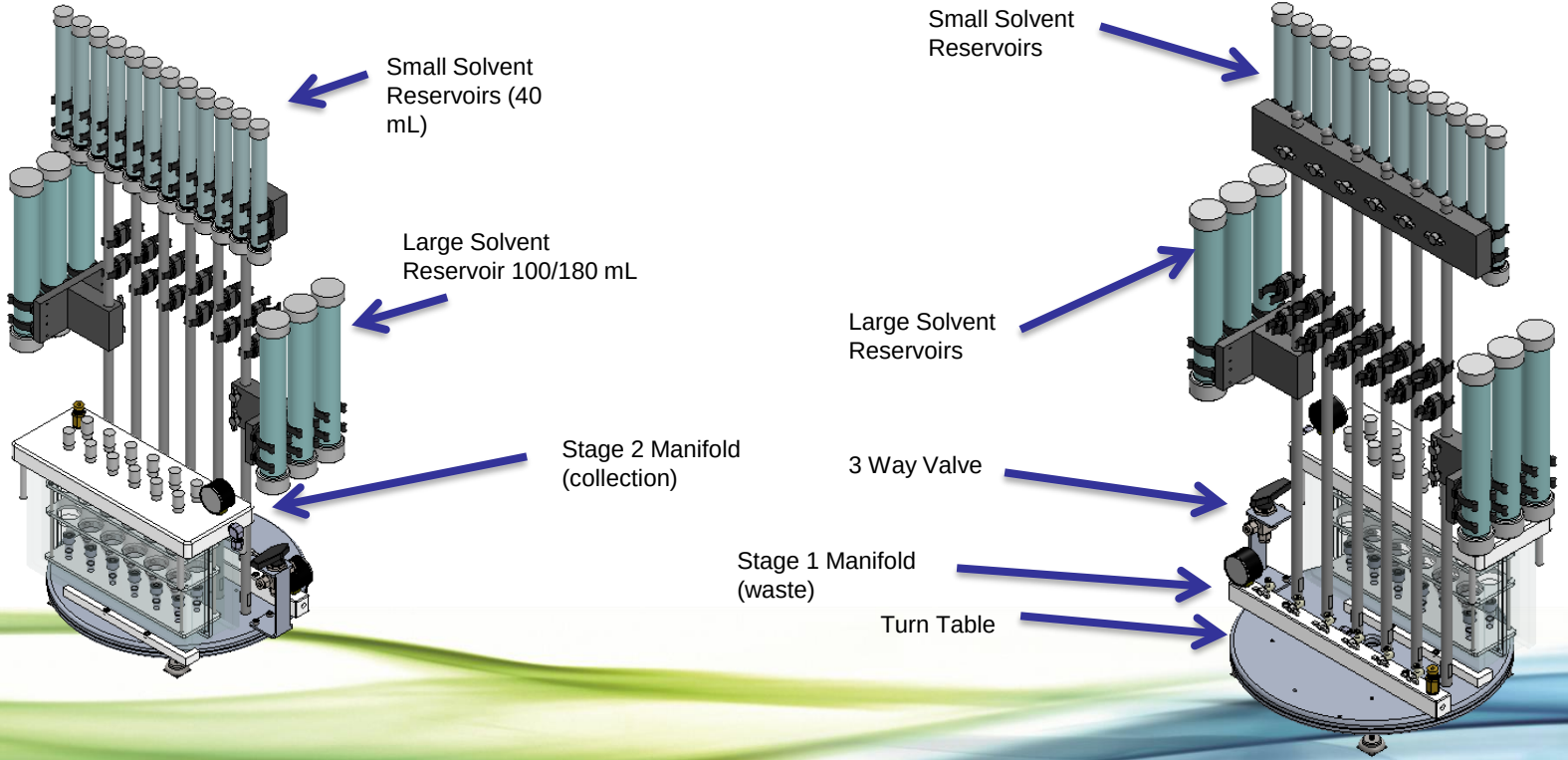
- **Advantages of Manual Sample Prep**
  - Most labs use a Manual Methods for the following reasons:
    - No electronics or mechanical components to fail
    - No down time due to the system failure
    - No service contract
    - No capital equipment cost

Semi-Automated System

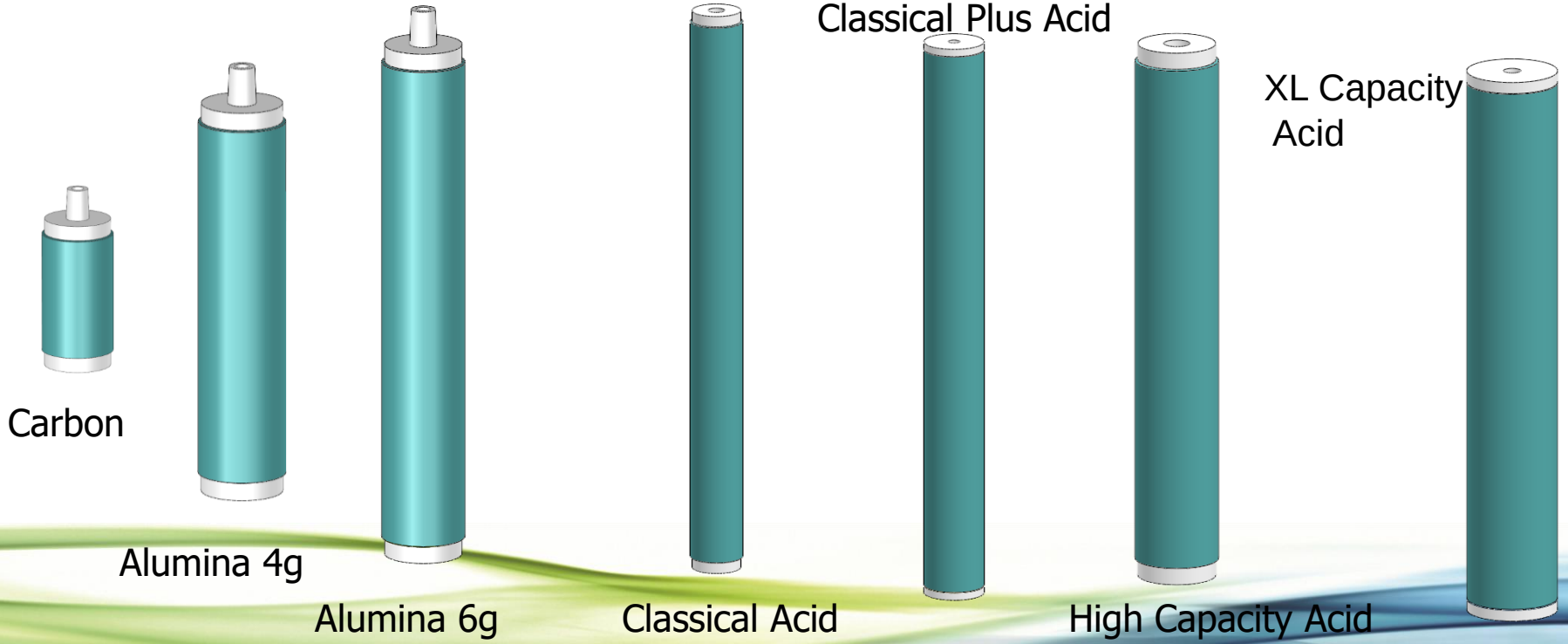
Specification:

- Simple to run, no computerized instrumentation
- Fast: 30 to 45 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
- 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

Characteristics of Semi-Automated System (EZPrep)

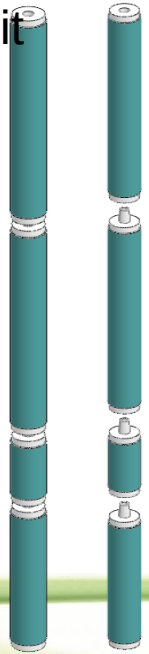


Columns (1)

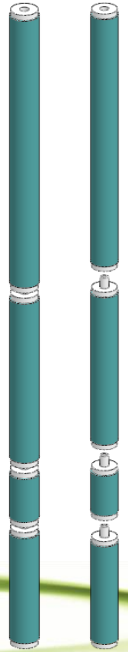

Carbon
Alumina 4g
Alumina 6g
Classical Acid
Classical Plus Acid
High Capacity Acid
XL Capacity Acid

Columns (2)

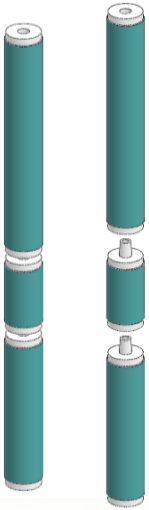
Classical Kit



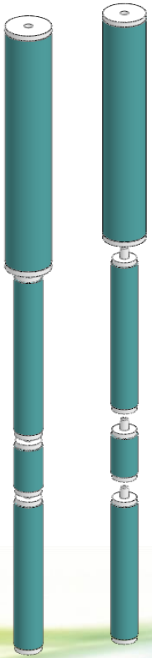
Classical Plus Kit



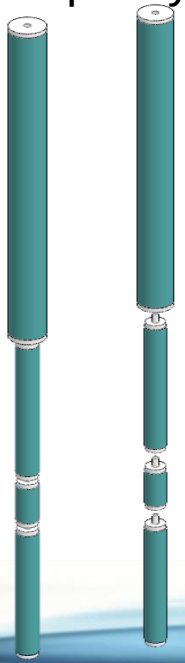
Mini Kit



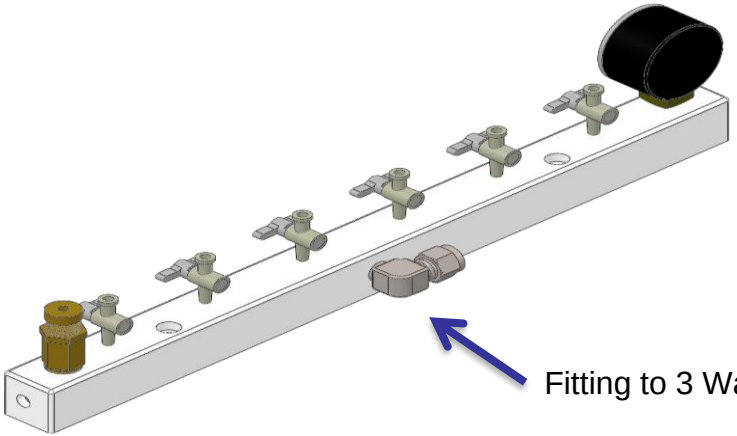
High Capacity Kit



XL Capacity Kit

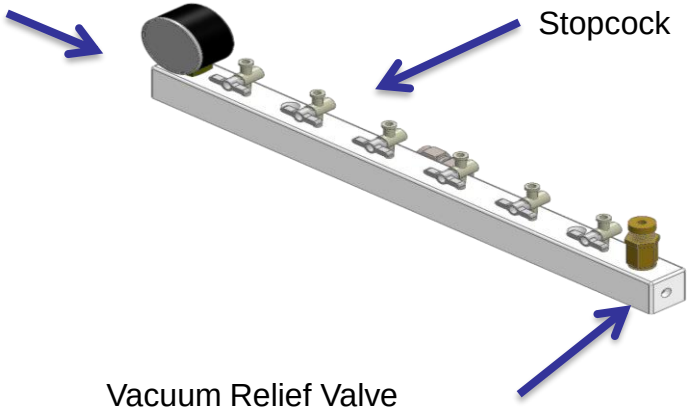


Stage 1 Manifold



Fitting to 3 Way Valve

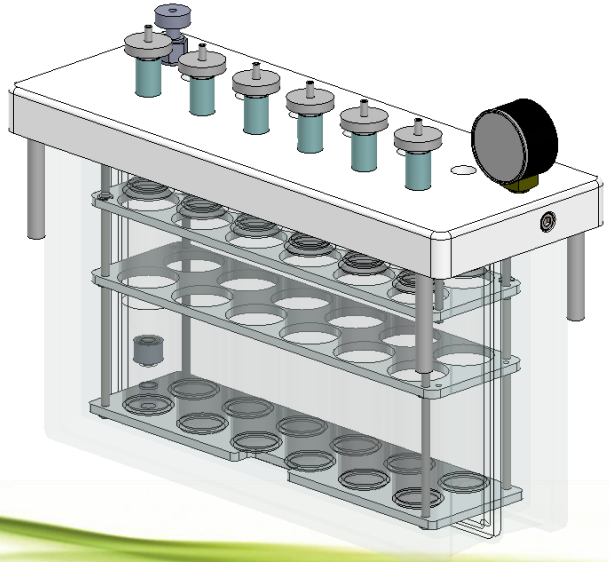
Vacuum Gauge



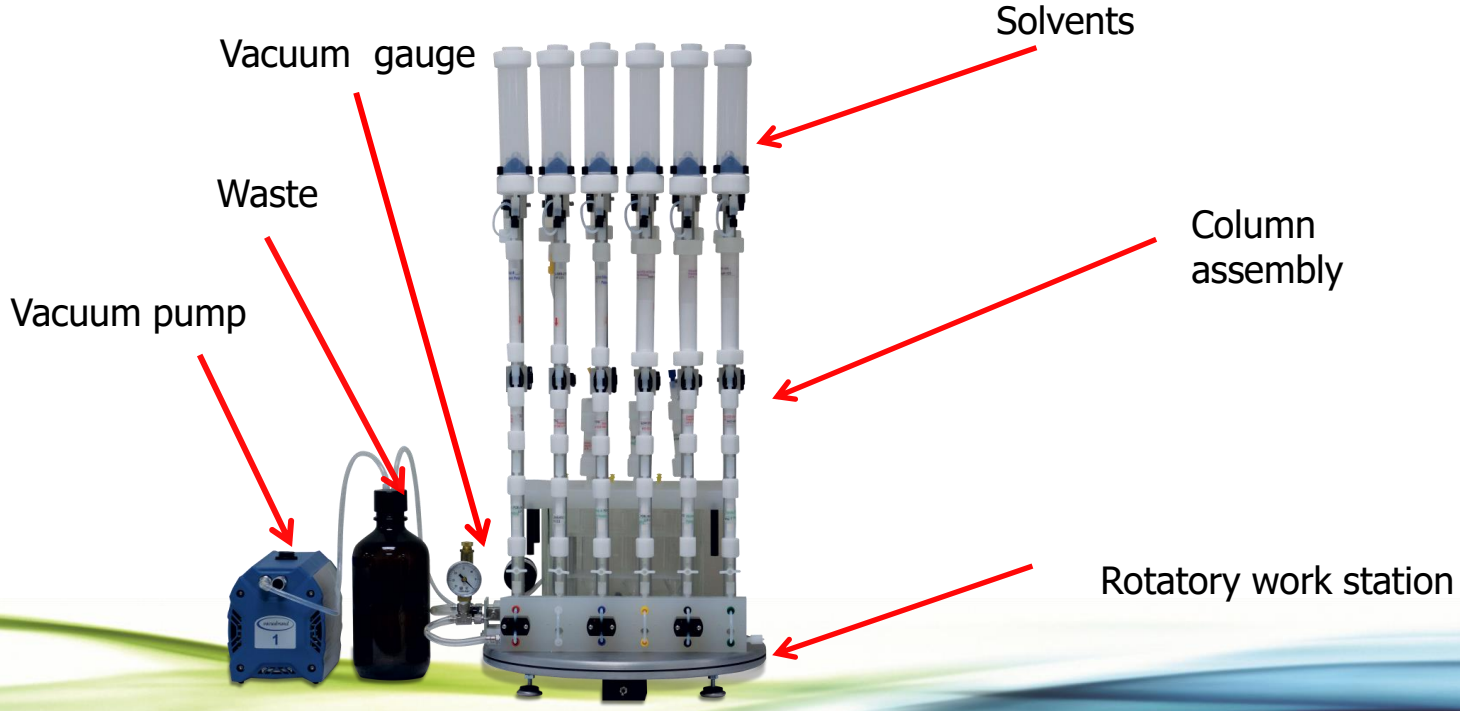
Stopcock

Vacuum Relief Valve

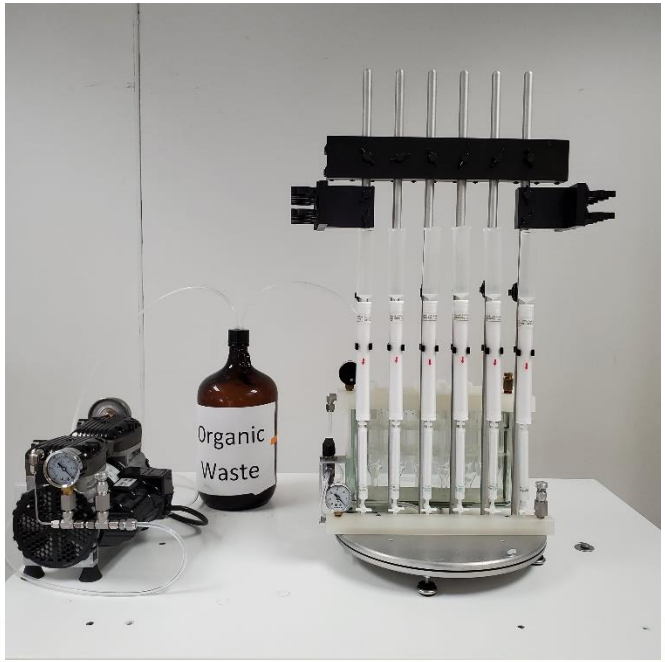
Stage 2 Manifold



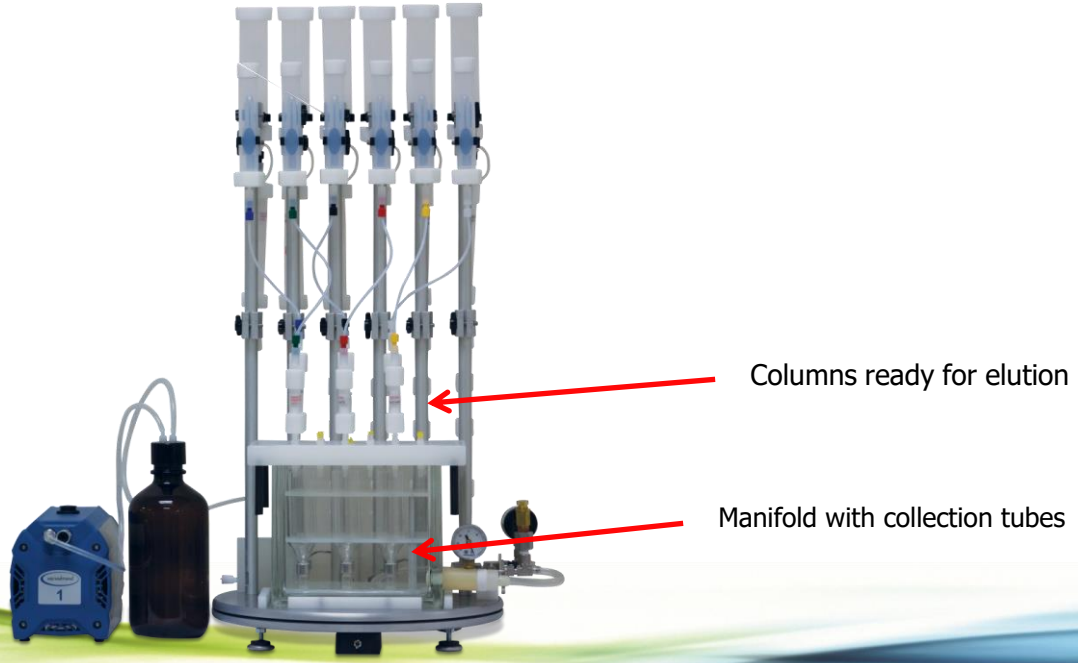
Stage 1: to waste



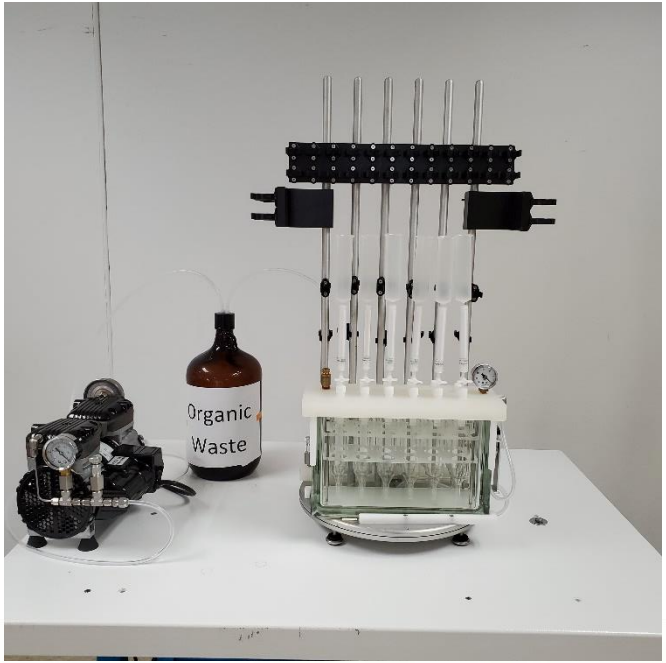
EZPrep Stage 1



Stage 2: collect



EZPrep Stage 2 Fractions



Attributes

- **Closed loop system:**
  - **Eliminates background contaminants**
  - **No washing needed.**
  - **Capped solvent reservoirs**
- **Optimized for solvent reduction while obtaining highest possible recoveries**
- **Easy sample loading on top of silica column via syringe vial**
- **Columns connect easy with SNAP connections**

Extracts in hexane - PCBs

- **Stage 1: Connect High Capacity Acid Silica and Alumina (no Carbon) and condition with 60 mL of hexane (vacuum, waste)**

- **Stage 2: Load sample (in hexane, collect Fraction # 1), rinse loading vials with hexane, elute with 160 mL hexane (collect Fraction # 1), remove acid silica, elute alumina with 50 mL dichloromethane (collect Fraction # 1)**

- **All 209 PCBs are now in Fraction # 1**

SuperVap 6 Concentrator 250 mLs

SuperVap Concentration/Evaporation

- **System pre-heated to 55-60 ^{o}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.**

SuperVap 24 position GC vial Concentrator

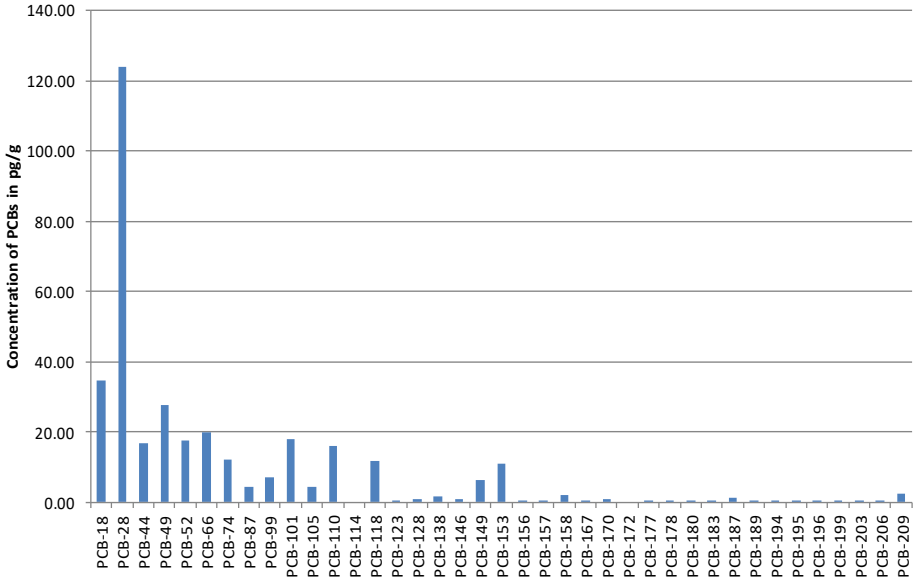
Direct-to-Vial

GC vial

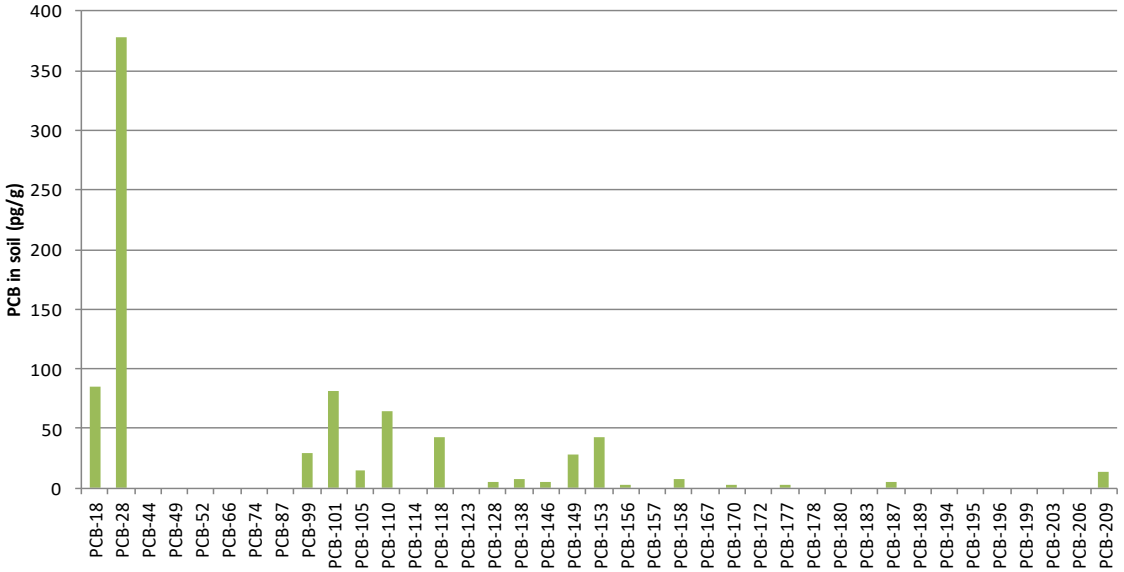
FMS
Fluid Management Systems

DFS HRGC/HRMS

Native PCBs in Serum extract



Native PCBs in Soil extract



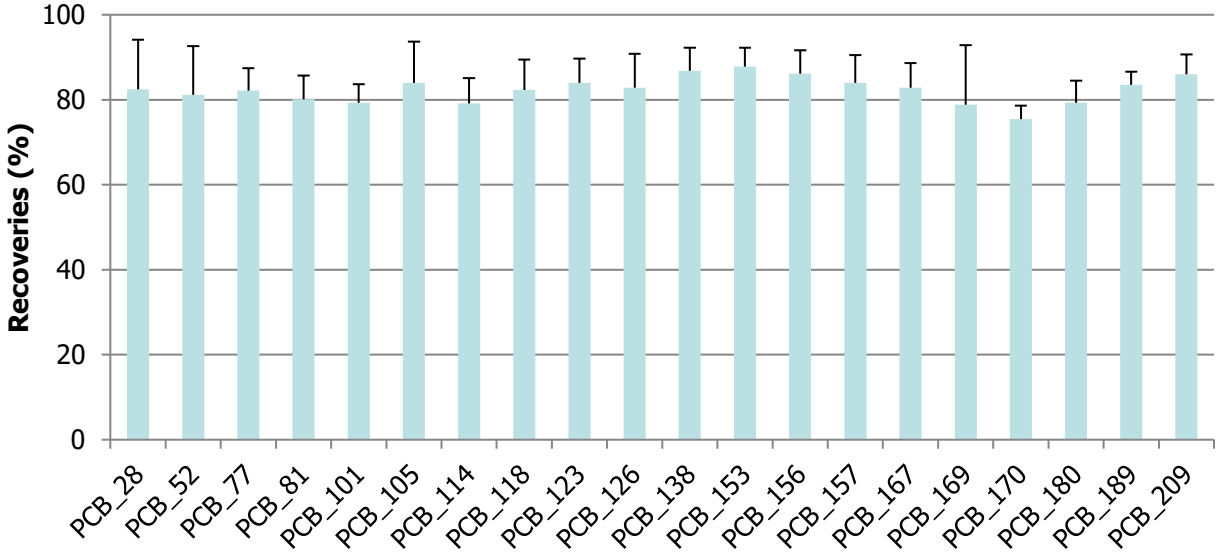
Extracts in toluene -PCBs

- Stage 1: Connect High Capacity Acid Silica and Alumina (no Carbon) and condition with 60 mL of hexane (vacuum, waste)

- Stage 2: Load sample (in 2-10 mL toluene, collect Fraction # 1), rinse loading vials with hexane, elute with 60 mL hexane (collect Fraction # 1), remove acid silica, elute alumina with 50 mL dichloromethane (collect Fraction # 1)

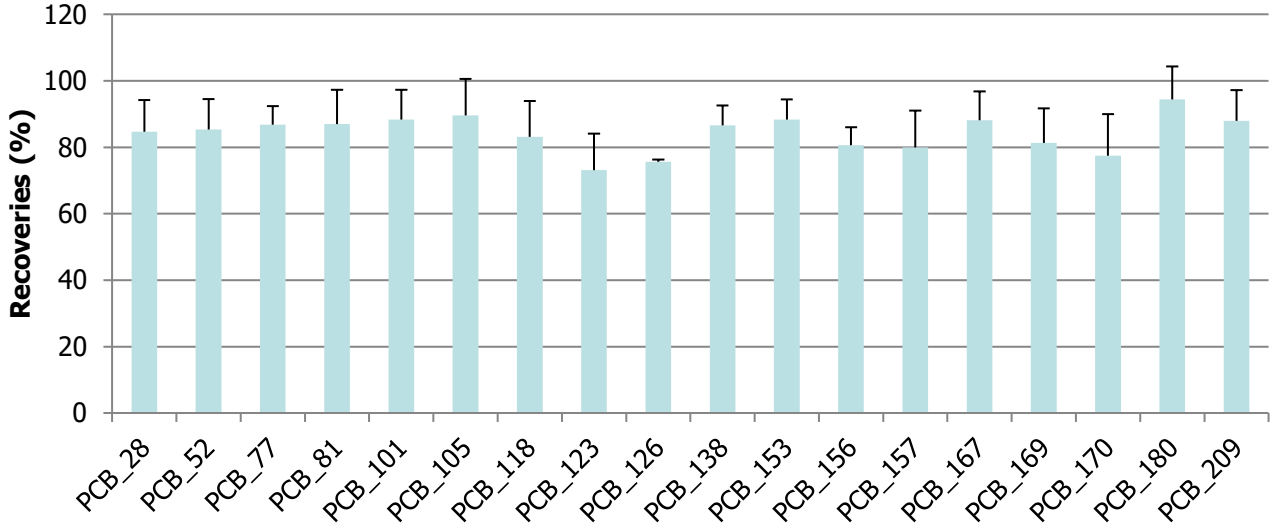
- All 209 PCBs are now in Fraction # 1

13C recoveries PCBs soil



10 g soil in toluene, n=6

13C recoveries PCBs salmon



2 g salmon in toluene, n=6

Column Kits with various fat removal capacities for samples in hexane

		STAGE 1			STAGE 2
					PCBs
	Fat Removal	Hexane	Hexane	Hexane	DCM
Column kits	Capacity	conditioning (mL)	sample volume (mL)	Elute Silica (mL)	Alumina (mL)
Classical Plus	1.0 g	20	30	100	50
High Capacity	2.5 g	40	30	160	50
Extra high Capacity	5.0 g	60	30	180	50

Conclusions (1)

- **Samples in toluene (environmental, food): 2-10 mL toluene, collect all 209 PCBs in one fraction using hexane followed by DCM**
- **Reduced hexane volume needed for silica column because of presence toluene**
- **Works also for samples in hexane but more hexane needed in that case for silica elution ("toluene effect" not present)**
- **EZPrep suitable for environmental and food analyses in toluene as solvent. Also suitable for samples in hexane**
- **High sample throughput → 18 samples/hour**
- **6 samples in parallel per station**
- **3 stations fit in one hood**

Conclusions (2)

- **System gives excellent recoveries for PCBs comparable to automated systems**
- **Use of certified pre-packaged columns guarantees low native background**
- **No worries about breakdown or downtime**
- **No washing needed**
- **No cross-contamination**
- **Low cost**