

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



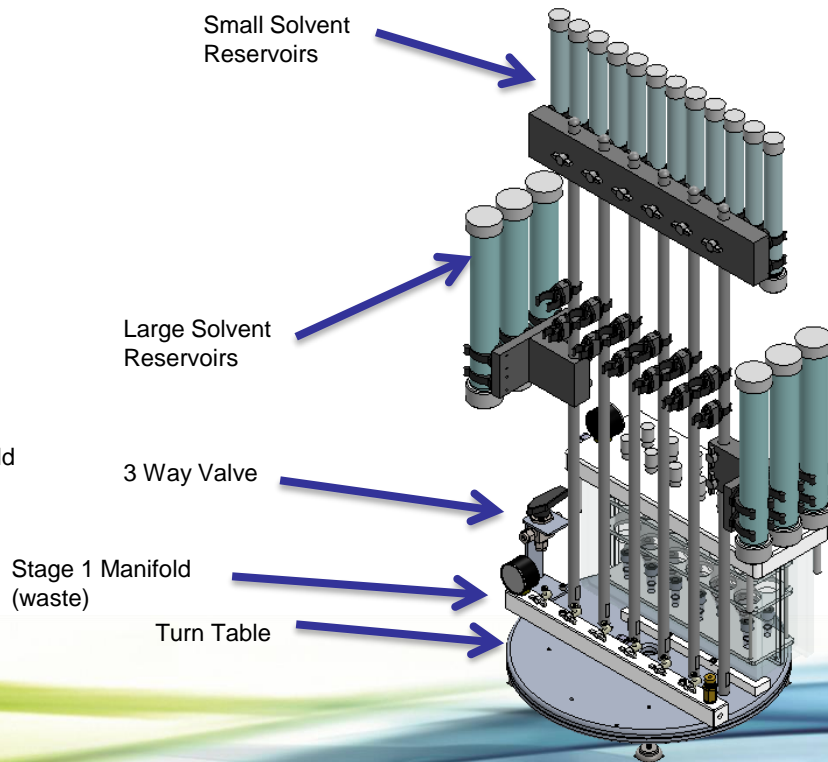
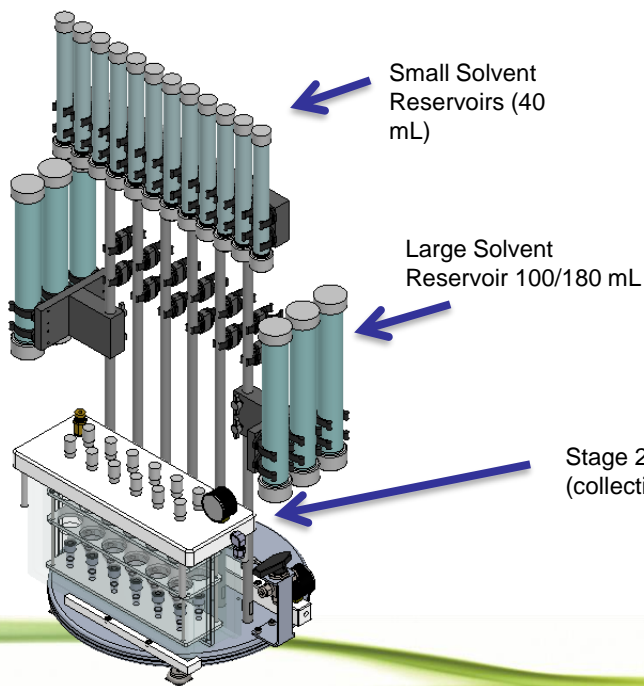
Semi-Automated System

Specification:

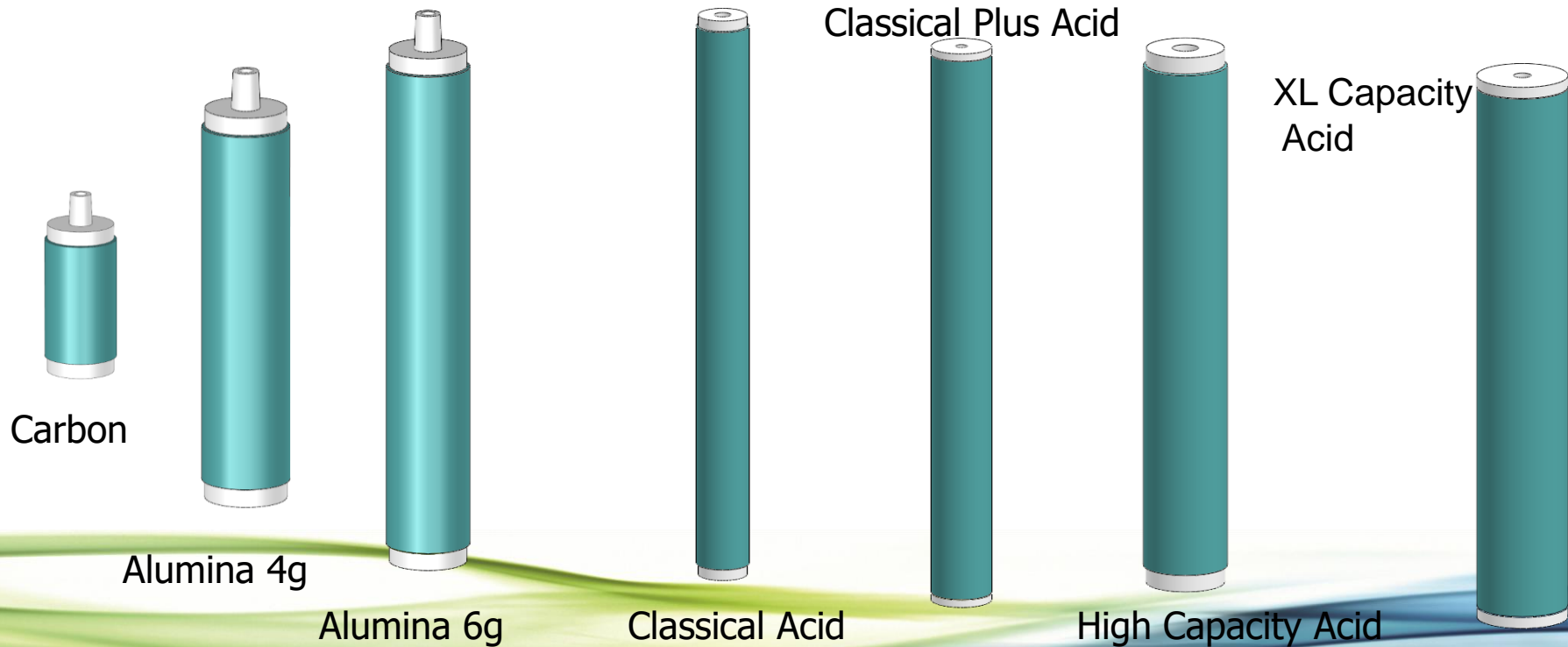
- Simple to run, no computerized instrumentation
- Fast: 30 to 45 min
- Closed loop system to give a clean background, low level detection
- Use certified pre-packaged columns
- Green technology, only vacuum pump uses power
- Low solvents
- Economical column kits, choice of low fat and high fat column kits
- No capital equipment cost
- No electronics or mechanical equipment to fail
- No downtime



Characteristics of Semi-Automated System (EZPrep)

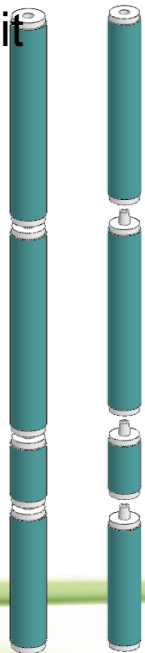


Columns (1)

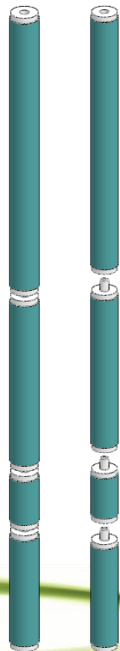


Columns (2)

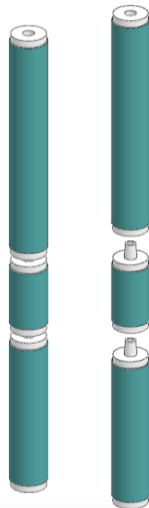
Classical
Kit



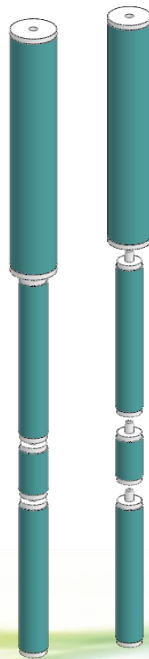
Classical Plus
Kit



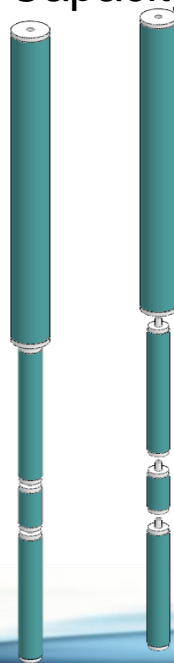
Mini Kit



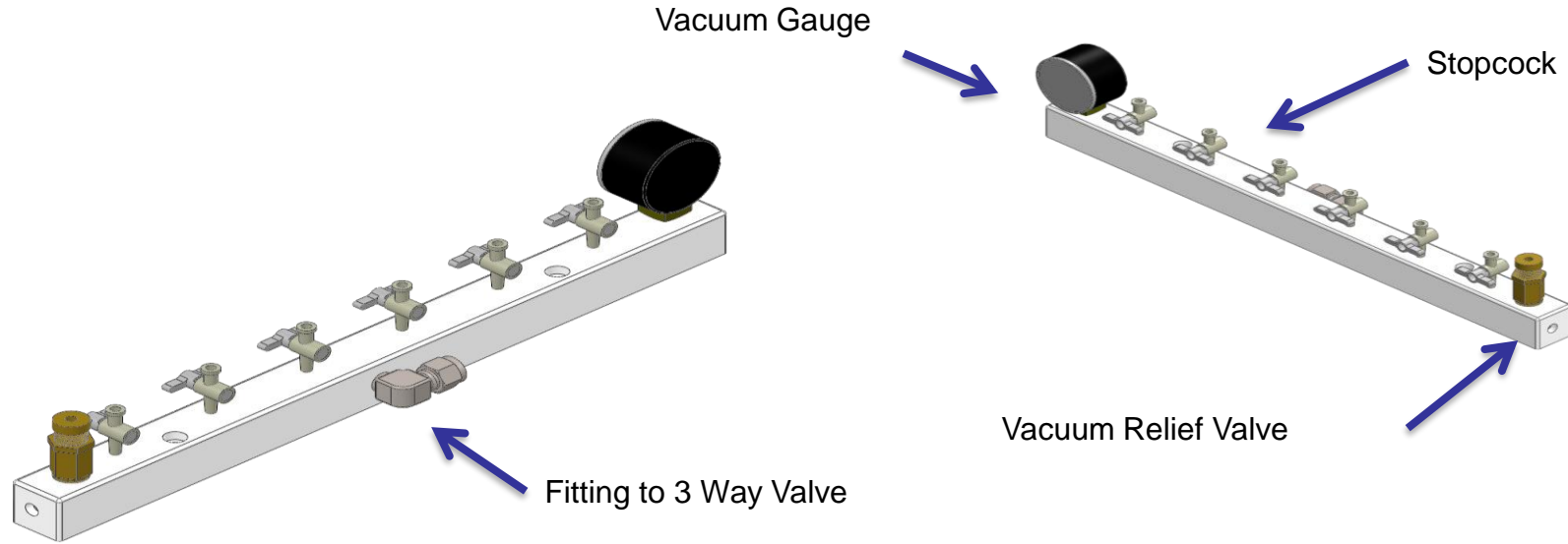
High Capacity
Kit



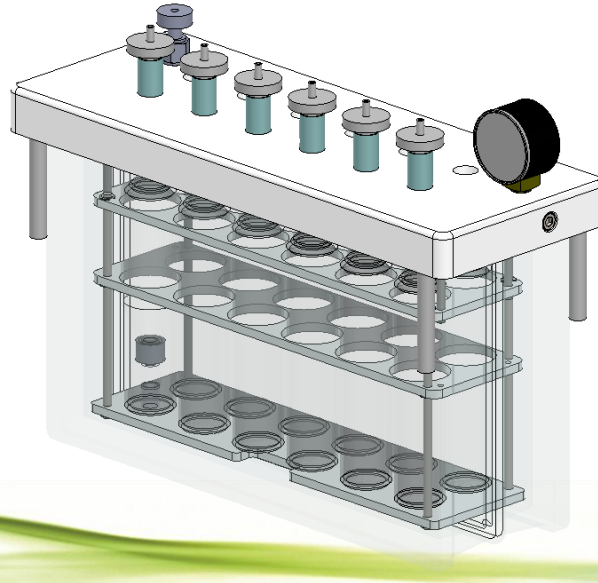
XL Capacity
Kit



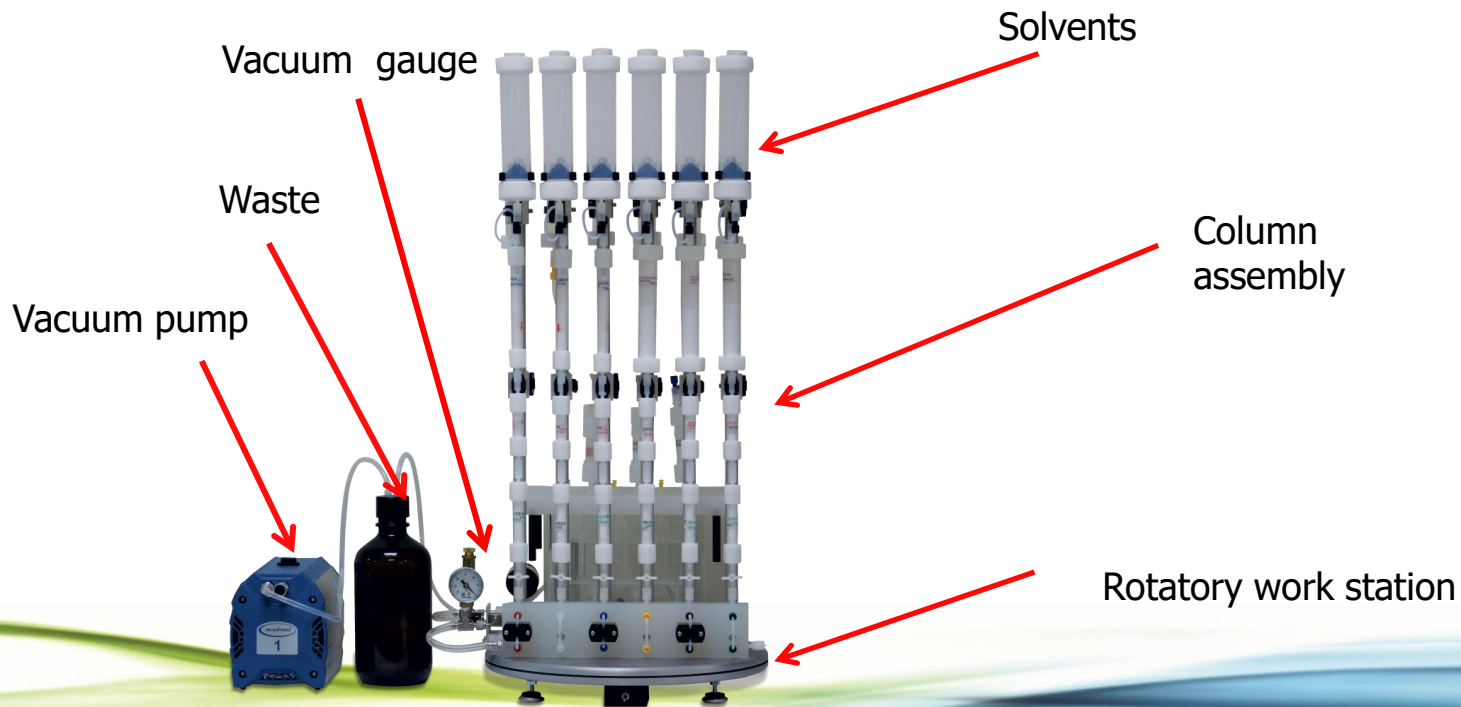
Stage 1 Manifold



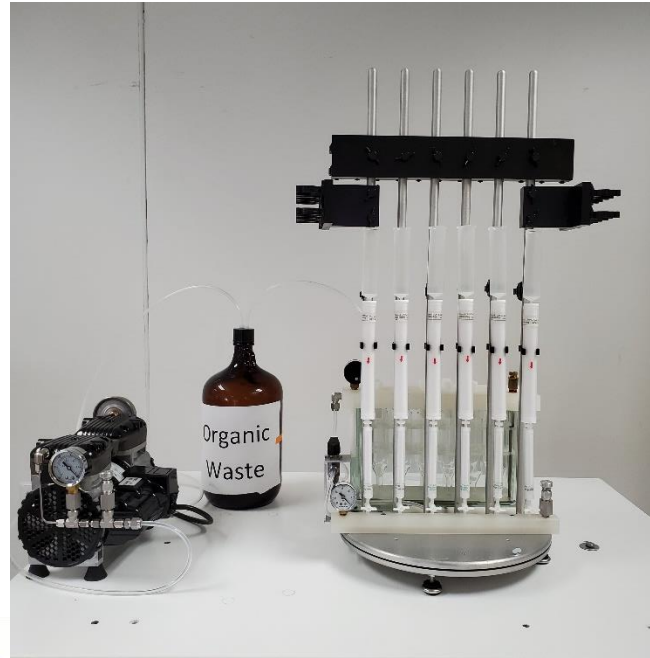
Stage 2 Manifold



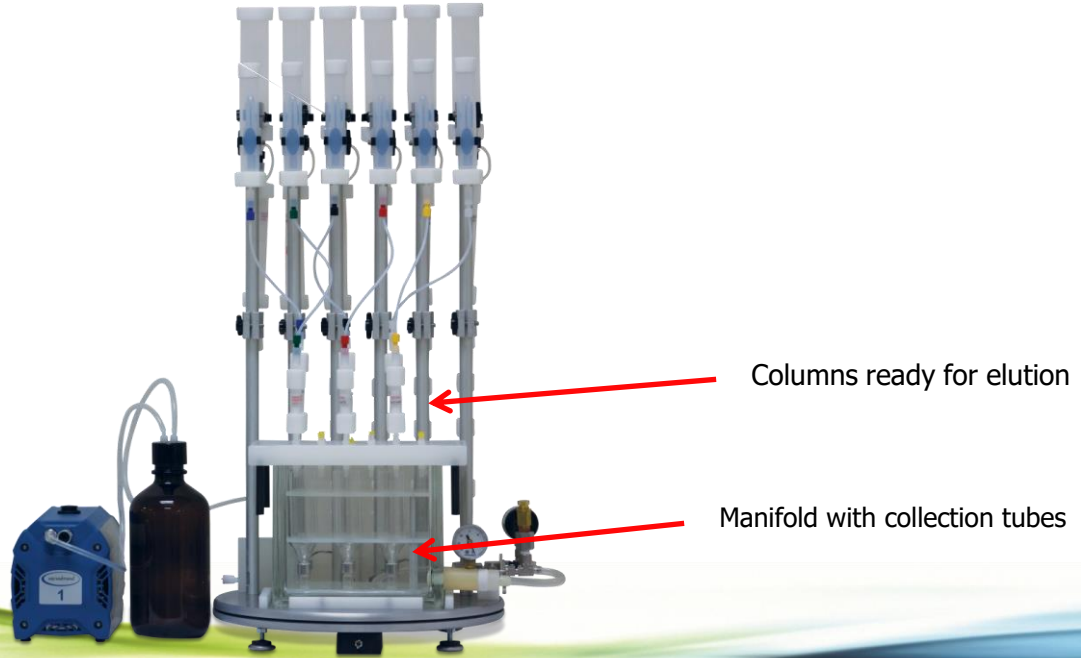
Stage 1: to waste



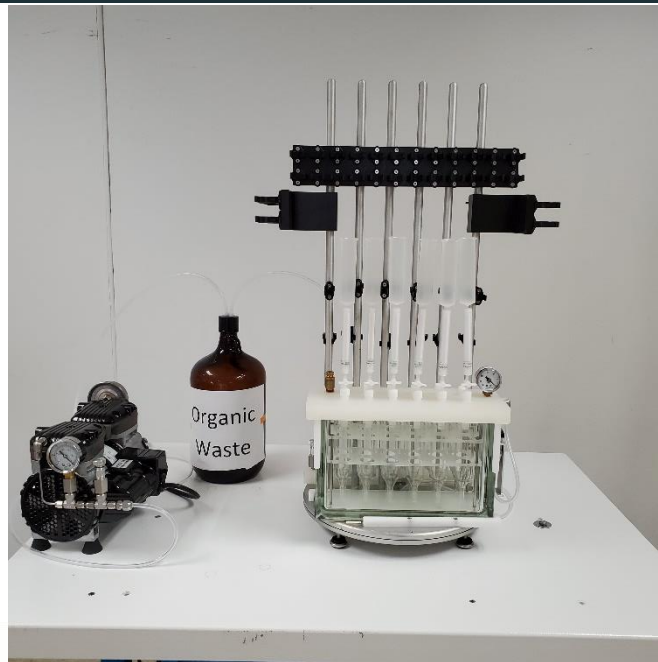
EZPrep Stage 1



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 °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

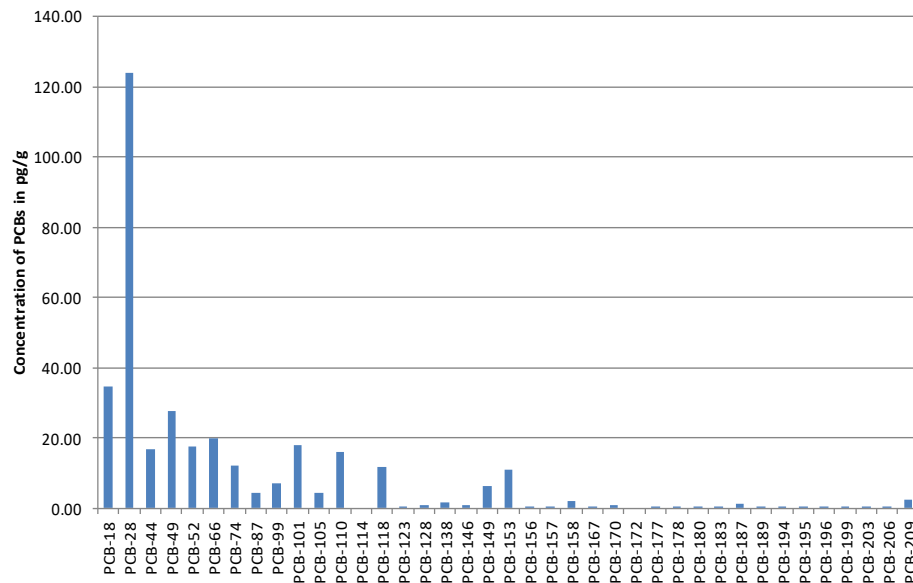


GC vial

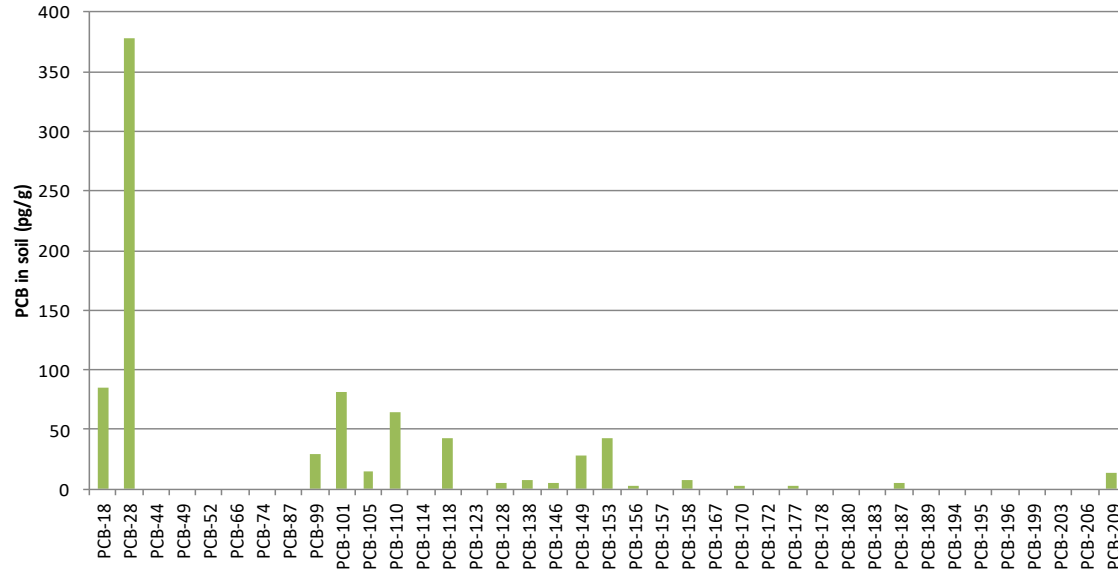
DFS HRGC/HRMS



Native PCBs in Serum extract



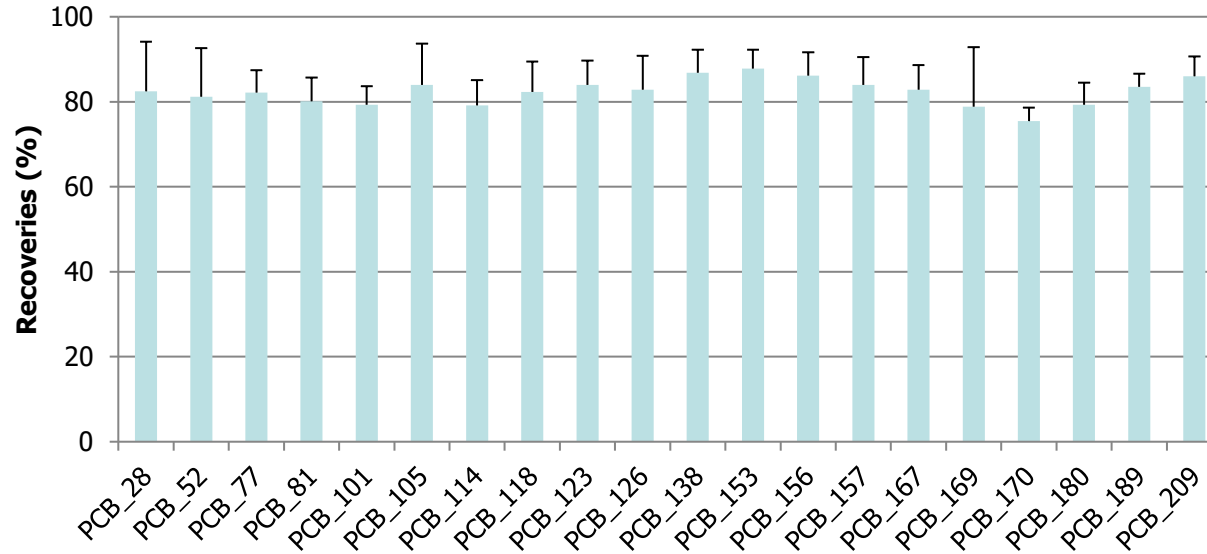
Native PCBs in Soil extract



Extracts in toluene -PCBs

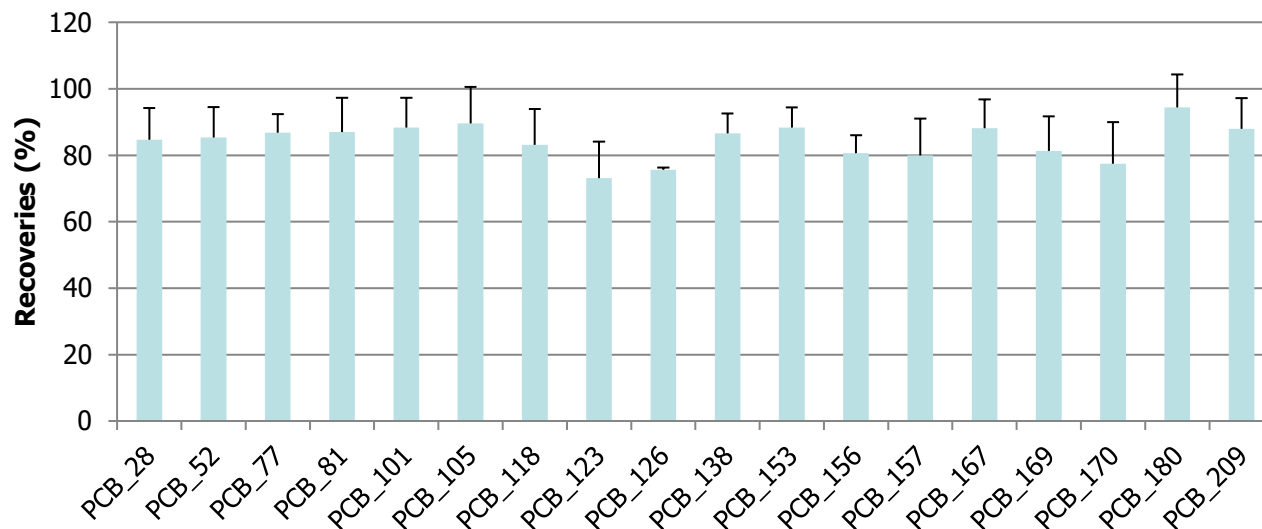
- Stage 1: Connect High Capacity Acid Silica and Alumina (no Carbon) and condition with 60 mL of hexane (vacuum, waste)
- Stage 2: Load sample (in 2-10 mL toluene, collect Fraction # 1), rinse loading vials with hexane, elute with 60 mL hexane (collect Fraction # 1), remove acid silica, elute alumina 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

^{13}C recoveries PCBs salmon



2 g salmon in
toluene, n=6

Column Kits with various fat removal capacities for samples in hexane

Column kits	STAGE 1				STAGE 2
					PCBs
	Fat Removal Capacity	Hexane conditioning (mL)	Hexane sample volume (mL)	Hexane Elute Silica (mL)	DCM 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**

Questions

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