

#### A SIMPLE, QUICK, POWERFUL DIOXIN & PCB SAMPLE CLEANUP SYSTEMS

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## Introduction

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





#### 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:
- Cleaner Background Interferences:
- Quality Results:
- Green Technology:
- QA/QC & Accreditation Requirements:
- Computerized Method:

30 to 45 Minutes for 6 Samples Closed Loop System Certified Pre-packaged Columns Low solvent and power use Easy to manage 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





## Combine best features (EZPrep Family)

Combine both features:

- Fast: 30-40 min
- Simple to run, no computerized instrumentation
- Closed loop system to give a clean background, low level detection
- Use certified pre-packaged columns
- Green technology, only uses vacuum or pressurization to do cleanup
- Low solvent volumes
- Economical column kits, five choices of low fat to high fat column kits
- Low capital equipment cost
- Little electronics or mechanical equipment to fail
- Little cleaning and no cross-contamination
- Minimal downtime
- Can be vacuum or pressurized



## Combine best features (EZPrep Family)

| Features  | EzPrep                 | EzPrep/+               |  |
|---|------------------------|------------------------|--|
| System run time for 6 samples                               | 30 ~70 min             | 30 ~ 40 min            |  |
| Fat Removal Capacity  | .1 ~ 5g                | .1 ~10g                |  |
| 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<br>Cross contamination | 30~60 min<br>No Tubing | 10~20 min<br>No Tubing |  |



#### EZprep with vacuum Pump





#### EZprep with vacuum Pump





#### Design of Semi-automatic EZprep 123

#### Stage 2 Step 3: Collect Dioxins Collect PCBs









#### Alternative system: EZPrep123





Stage 2 / Back









#### Automated EZprep/+





# EzPrep /+ Control







•Programmable Flow rate and Volume

•Pressure indicator and over pressure alarm

•Real time read-out for dispensed volume

•Ability to select from 1 to six samples

•Can accommodate up to 4 solvents

•Allows for less expensive automation/pressurization



#### Attributes EZPrep Plus

- Closed loop system, eliminates background contaminants
- Optimized for solvent reduction while obtaining highest possible recoveries
- Easy automated sample loading on top of silica column via sample vial and use of nitrogen to load sample
- Columns connect easy with SNAP connections
- Multi pump brings convenient automated solvent dispense with controllable flow & volume
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- All advantages of more expensive automated systems without the disadvantages





## Columns (1)





## Columns (2)





#### Comparison Systems

| Task                       | Manual      | EZPrep    | EZPrep+   |
|----------------------------|-------------|-----------|-----------|
|                            |             | vacuum    |           |
| Labor time                 | > 1 day     | 30-70 min | 10-20 min |
| Accreditation/validation   | Slow        | Fast      | Fast      |
| Pre-packaged Columns       | No          | Yes       | Yes       |
| Native background          | Can be high | Low       | Low       |
| Electro-Mechanical Valves  | No          | No        | minimal   |
| Failure & Downtime         | No          | No        | minimal   |
| Training Requirements      | Hours       | Hours     | Hours     |
| Capital Equipment Cost     | Minimal     | Low       | Low       |
| Service & Maintenance Cost | Minimal     | Minimal   | Minimal   |





## Program (1)

Stage 1:

- Assemble the columns in the order: acidic silica-carbon-alumina (step 1)
- Condition column assembly with hexane from multi-pump (step 2)
- Load sample in hexane onto top of acidic silica column from sample vial and apply nitrogen to push sample onto acidic silica columns (step 3; nitrogen optional)
- Elute silica column with hexane from multi-pump, analytes onto carbon and alumina (step 4)
- Note all Stage 1 steps go to waste





## Program (2)

#### Stage 2

- Disassemble the column set, discard acidic silica and keep the carbon and alumina columns (step 1)
- Rotate turn table, place collection vessels inside glass manifold, install carbon and alumina columns on top of manifold, note that carbon and alumina are eluted individually (step 2)
- Elute columns each in reverse direction with 40 mLs toluene from multi-pump, six fractions # 1 (PCDD/F/co-planary PCBs) and six fractions # 2 (mono- and di-ortho PCBs)





#### SuperVap 12 Concentrator 50 mLs





## SuperVap Concentration/Evaporation

- 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 temperature.





## **Direct-to-Vial**





#### SuperVap 24 position GC vial Concentrator







## 7010B TripleQuad





## 13 PCDD/F recoveries



3 g olive oil, high-capacity acid silicacarbon-alumina, n=12



## 13 PCB recoveries



3 g olive oil, high-capacity acid silicacarbon-alumina, n=12



# FILLIE MARAGEMENT Systeme PCBs - Automated vs Semi-Automated

2 g Fish Oil results in pg/g

|         |           | Semi-     |  |
|---------|-----------|-----------|--|
|         | Automated | Automated |  |
| 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        |  |
|         |           |           |  |

Fluid Management Systems Column Kits with various fat removal capacities

|                |          | STAGE 1           |             |              | STAGE 2               |         |              |
|----------------|----------|-------------------|-------------|--------------|-----------------------|---------|--------------|
|                |          |                   |             |              | PCBs                  | Dioxins |              |
|                | Fat      |                   |             |              |                       |         |              |
|                | Removal  | Hexane            | Hexane      | Hexane       | Toluene               | Toluene |              |
| Column kits    | Canacity | conditioning (ml) | sample      | Elute Silica | Poverse Alumina (ml.) | (ml)    | Time (min)   |
| Column Kits    | capacity |                   | volume (mr) | (1112)       | Reverse Alumina (mL)  | (1112)  | rine (iiiii) |
|                |          |                   |             |              |                       |         |              |
| Mini           | 0.15     | 20                | 10          | 80           | 40                    | 40      | 30           |
| Classical      | 0.5      | 20                | 10          | 90           | 40                    | 40      | 30           |
| Classical Plus | 1.0 g    | 20                | 10          | 100          | 40                    | 40      | 30           |
| High Capacity  | 4.0 g    | 40                | 30          | 160          | 40                    | 40      | 40           |
| Extra high     |          |                   |             |              |                       |         |              |
| Capacity       | 7.0 g    | 60                | 30          | 180          | 40                    | 40      | 40           |





## Conclusions (1)

- Fast cleanup possible with EZPrep Plus system 30-70 min
- Multi-pump allows for easy solvent elution and nitrogen application is used for sample loading
- Column kits tailored to specific lipid capacity needs (0.15 to 7 g of fat) allow for optimum use of consumables
- High sample throughput  $\rightarrow$  12 samples/hour
- 6 samples in parallel per station
- 2 stations fit in one hood



#### Conclusions (2)

- System gives excellent recoveries for PCDD/Fs and PCBs comparable to more expensive fully automated systems
- PBDEs also option are always in PCBs fraction
- Use of certified pre-packaged columns guarantees low native background
- Little breakdown or downtime
- Little washing needed
- No cross-contamination
- Less expensive than other automated systems



# Come see us at booth G-2 Questions?

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