

# **Modified Sample Clean-up for Combined POPs Using Automated Multi-Column Fractionation and Analytical Optimization.**

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# Objective

- Utilize Florisil columns in place of alumina for combined POPs extract clean-up to maximize PCB recovery efficiency in environmental samples
  - Single fraction, single solvent.
- Establish elution protocols for a greater array of compound classes in addition to PCBs



# Analyte Classes

## **Primary**

- **Tetra- through Octa-Chlorinated Dioxins and Difurans**
- **Polychlorinated Biphenyl Congeners**

## **Additional**

- **Polybrominated Diphenyl Ethers**
- **Polychlorinated Naphthalenes**
- **Select Organochlorine Pesticides**





# PowerPrep Sample Clean-up System







# FMS, Inc. SuperVap w/ direct to GC vial



# Comparison

- Multi-Layer Silica Gel (acid/base/ $\text{AgNO}_3$ )
- Activated Alumina
- Carbon

- Multi-Layer Silica Gel (acid/base/ $\text{AgNO}_3$ )
- Activated Florisil
- Carbon



# Clean-up Functionality

- AgNO<sub>3</sub> impregnated Silica Gel
  - Remove sulfur from environmental samples (chlorinated compounds)
- H<sub>2</sub>SO<sub>4</sub> impregnated Silica Gel
  - Oxidation: removal of organic interferences
- NaOH impregnated Silica Gel
  - Reduction: removal of organic interferences



# Clean-up Functionality

- Activated Florisil
  - Absorption: fractionation, removal of interferences
- Carbon
  - Isolation of PCDD/PCDF/PCNs





# Experimental

- Florisil Optimization
  - Specific ration of deactivation to sorbent amount required for accurate fractionation of PCBs (including co-planars)
  - Full retention of PCDD/PCDFs for subsequent carbon fractionation



### Power-Prep System Plumbing Diagram

The diagram illustrates the plumbing for a Power-Prep System. It shows the flow of solvents and samples through various components:

- Solvent Sources:** Hexane (1), Methylene Chloride (2), Toluene (3), and Sample (4) are introduced into the system.
- Flow Control:** The system uses 2-Way Valves (2WV) and 3-Way Valves (3WV) to direct the flow of solvents and samples.
- Components:** The system includes a Pump, a Filter, a Cartridge, and a Fraction Collector.
- Flow Path:** The flow starts from the solvent sources, passes through the Pump and Filter, then through the Cartridge, and finally into the Fraction Collector.
- Legend:**
  - 1. Hexane
  - 2. Methylene Chloride
  - 3. Toluene
  - 4. Sample
  - 5. Fraction 1
  - 6. Fraction 2
  - 7. Fraction 3
  - 8. Waste

**Legend:**  
 2W - 2 Way Valves  
 3W - 3 Way Valves

# Elution Order

- F1: Hexane through Silica and Florisil
  - PCBs
  - PBDEs (BDE-209, partial others)
  - Some OCPs



# Elution Order

- F2: DCM through Florisil and Carbon
  - Remaining PBDEs
  - Remaining non-acid labile OCPs





# Elution Order

- F3: Toluene reverse eluted through Carbon
  - PCDDs
  - PCDFs
  - PCNs (tetra-Octa)



# Analytical

## Configuration #1

- Thermo DFS HRMS
- Dual 1310 GC's with dissimilar columns
- Dual TriPlus RHS autosamplers

## Configuration #2

- Thermo Quantum ULTRA TSQ
- Trace Ultra GC with dissimilar injection ports (PTV & Split/Splitless)
- TriPlus RHS autosamplers

# Analytical

## Configuration #1

PCDD/DFs

PCB Congeners

## Configuration #2

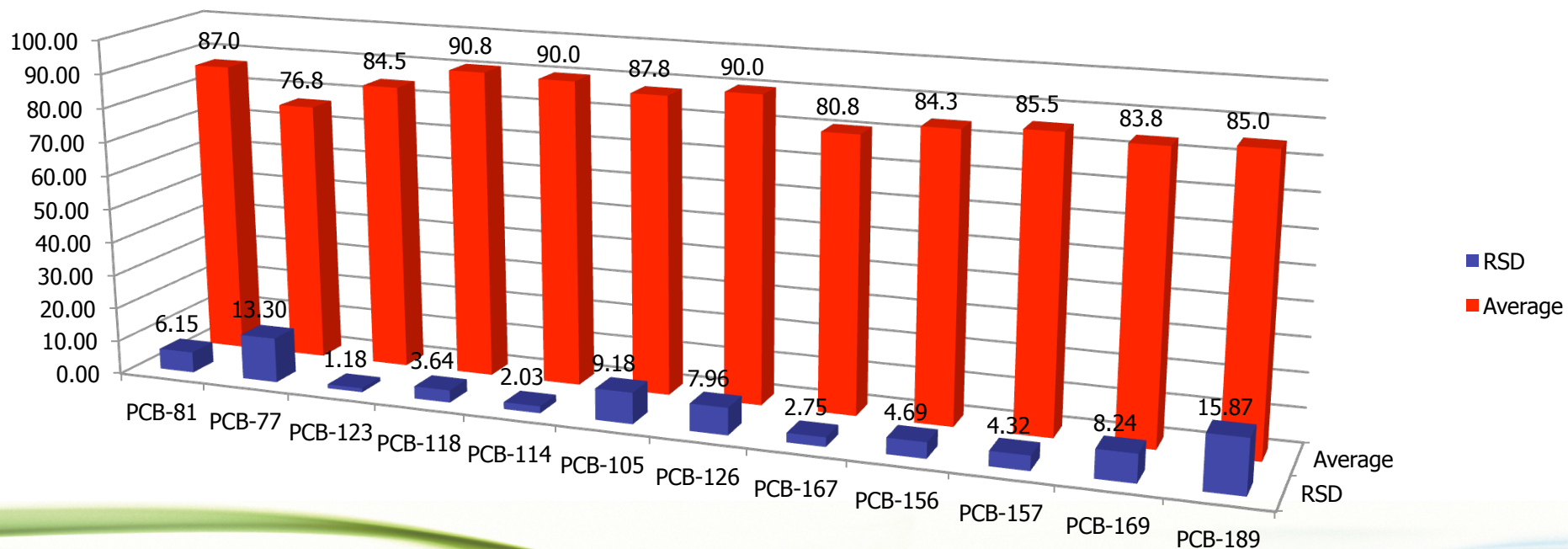
PBDE Congeners  
(PTV)

PCN Congeners (S/  
SS)



# PCB Labeled Recovery in a Soil Matrix

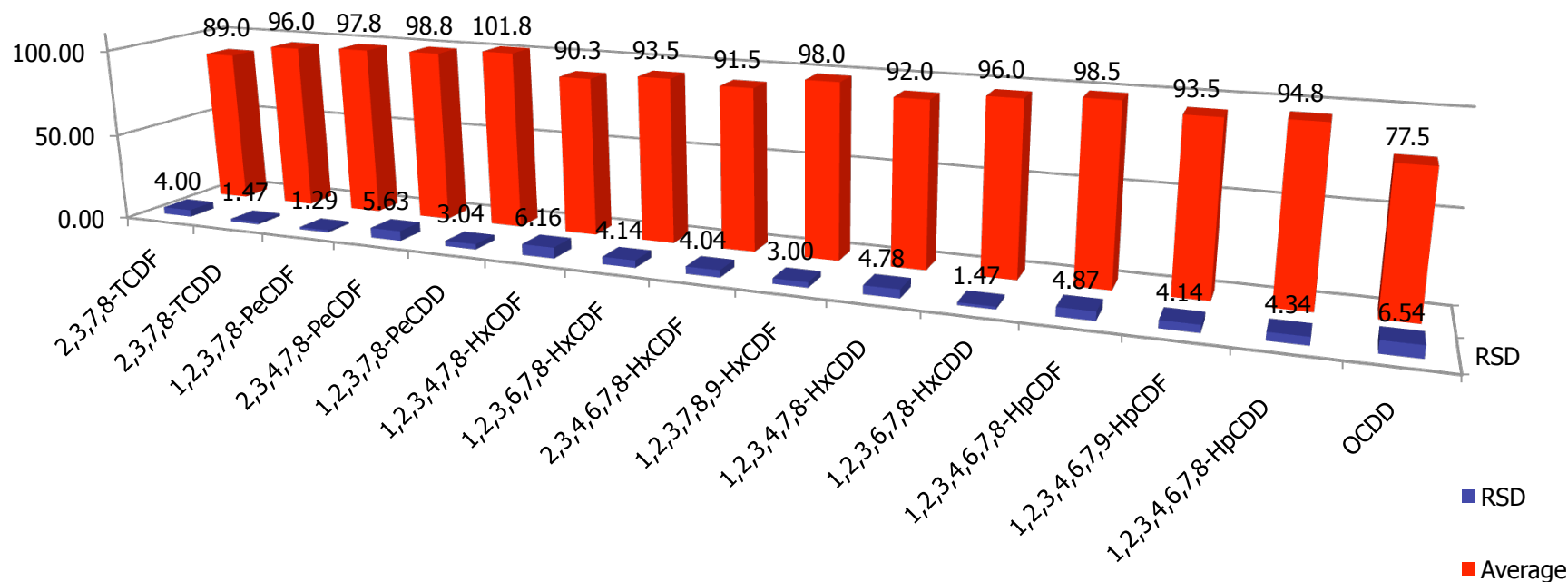
## Average Recovery



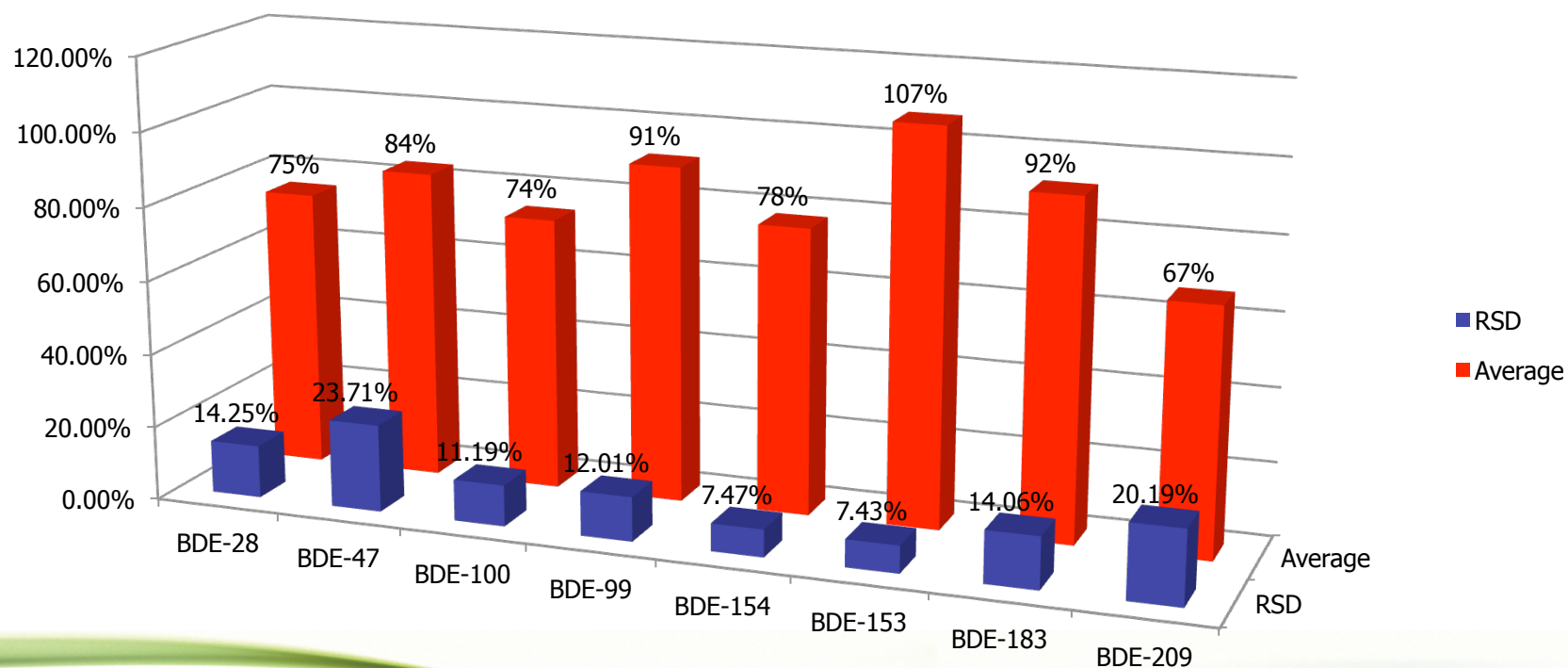


# PCDD/DF Labeled Recovery in a Soil Matrix

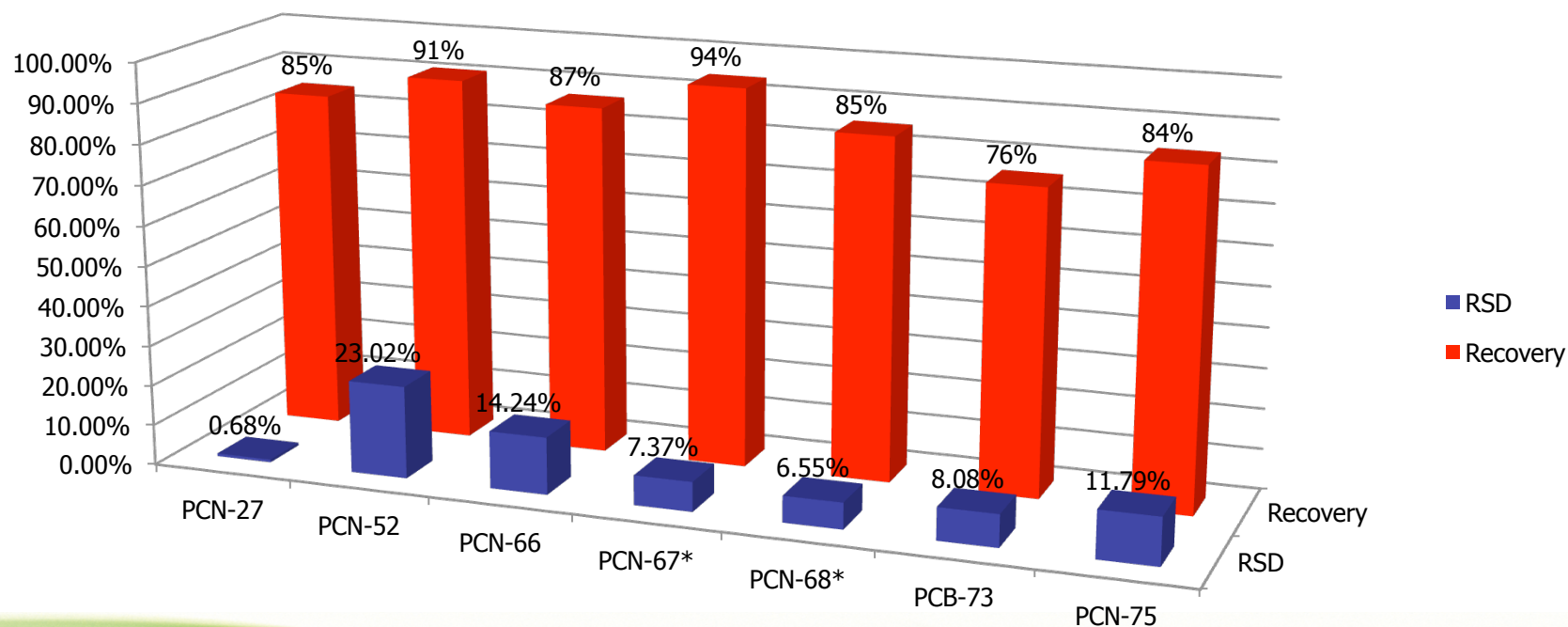
**Average Recovery**



# PBDE Labeled Recovery in a Soil Matrix



# PCN Labeled Recovery in a Soil Matrix



# Conclusions

- Florisil Effective for optimizing PCBs in an environmental matrix
- PCN data limited due to commercially available labeled congeners
- PBDE's not applicable for  $\text{AgNO}_3$  sulfur removal (Activated copper required)
- OCPs show greater analyte loss with increasing amounts of acidified silica gel (not suitable for 10 gram samples)



# Questions?

