# 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

**Pimary**

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

**Additional**

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

# FMS PowerPrep Sample Clean-up System

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

# Comparison

- Multi-Layer Silica Gel (acid/base/ $AgNO_3$)
- Activated Alumina
- Carbon

- Multi-Layer Silica Gel (acid/base/ $AgNO_3$)
- Activated Florisil
- Carbon

# Clean-up Functionality

- AgNO3 impregnated Silica Gel
  - Remove sulfur from environmental samples (chlorinated compounds)
- $H_2SO_4$ impregnated Silica Gel
  - Oxidation: removal of organic interferences
- NaOH impregnated Silica Gel
  - Reduction: removal of organic interferences

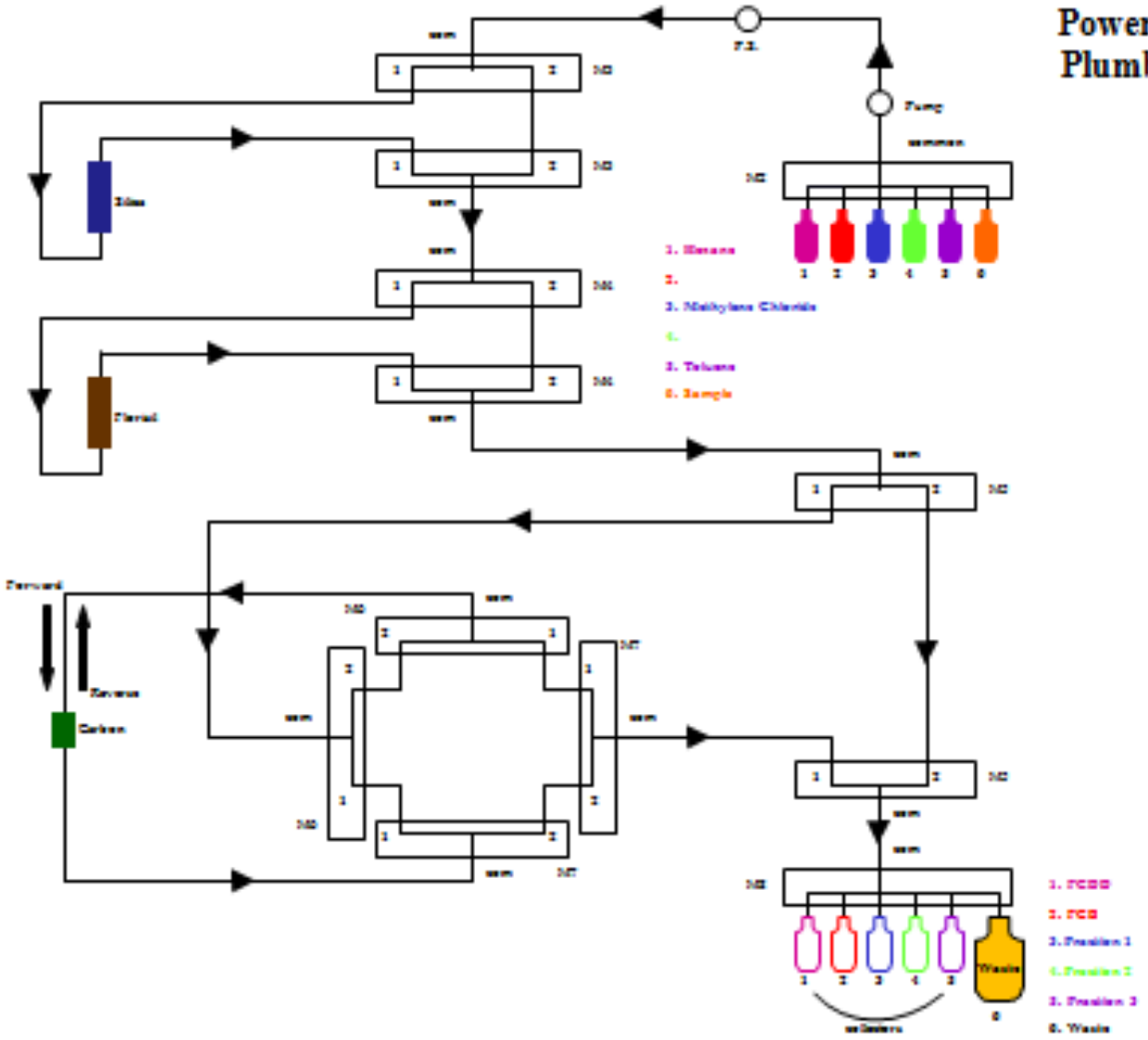
# Clean-up Functionality

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

# Expirmental

- 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

# Fluidic Pathway



# 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

<u>Configuration #1</u>

PCDD/DFs

PCB Congeners

<u>Configuration #2</u>

PBDE Congeners (PTV)

PCN Congeners (S/SS)

# PCB Labeled Recovery in a Soil Matrix

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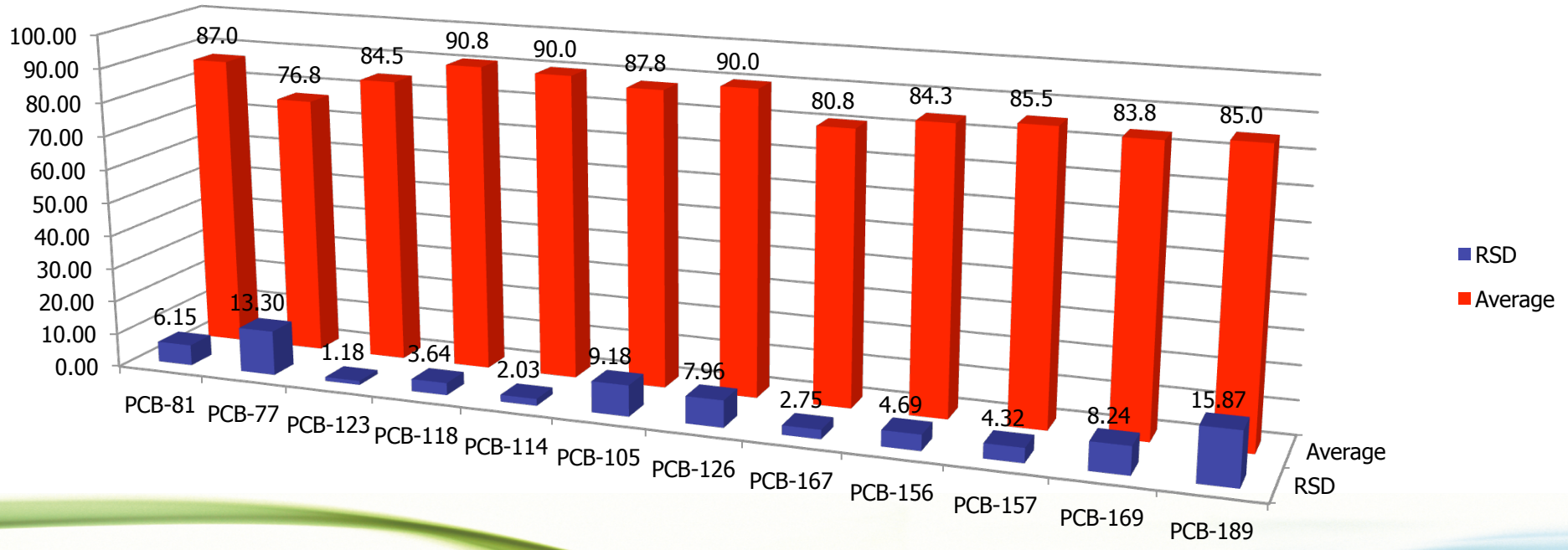
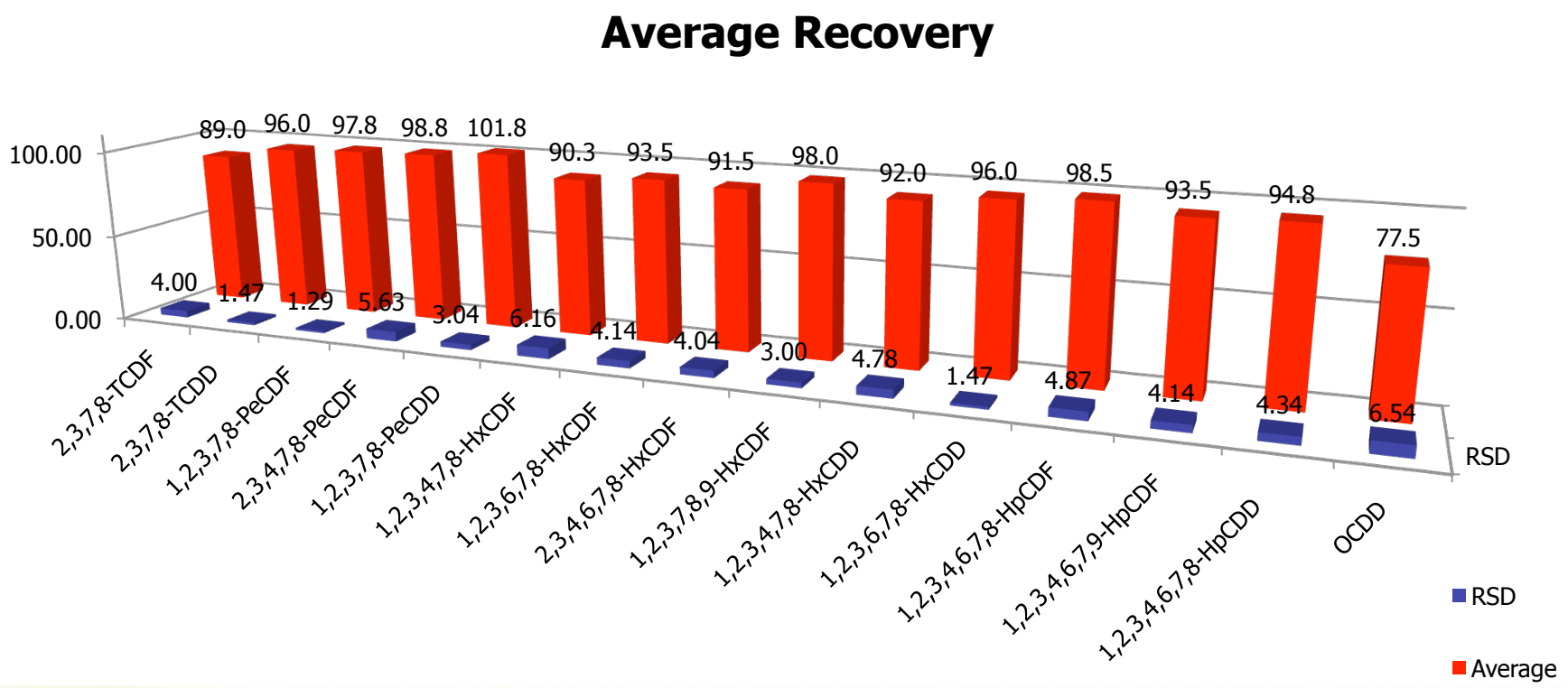
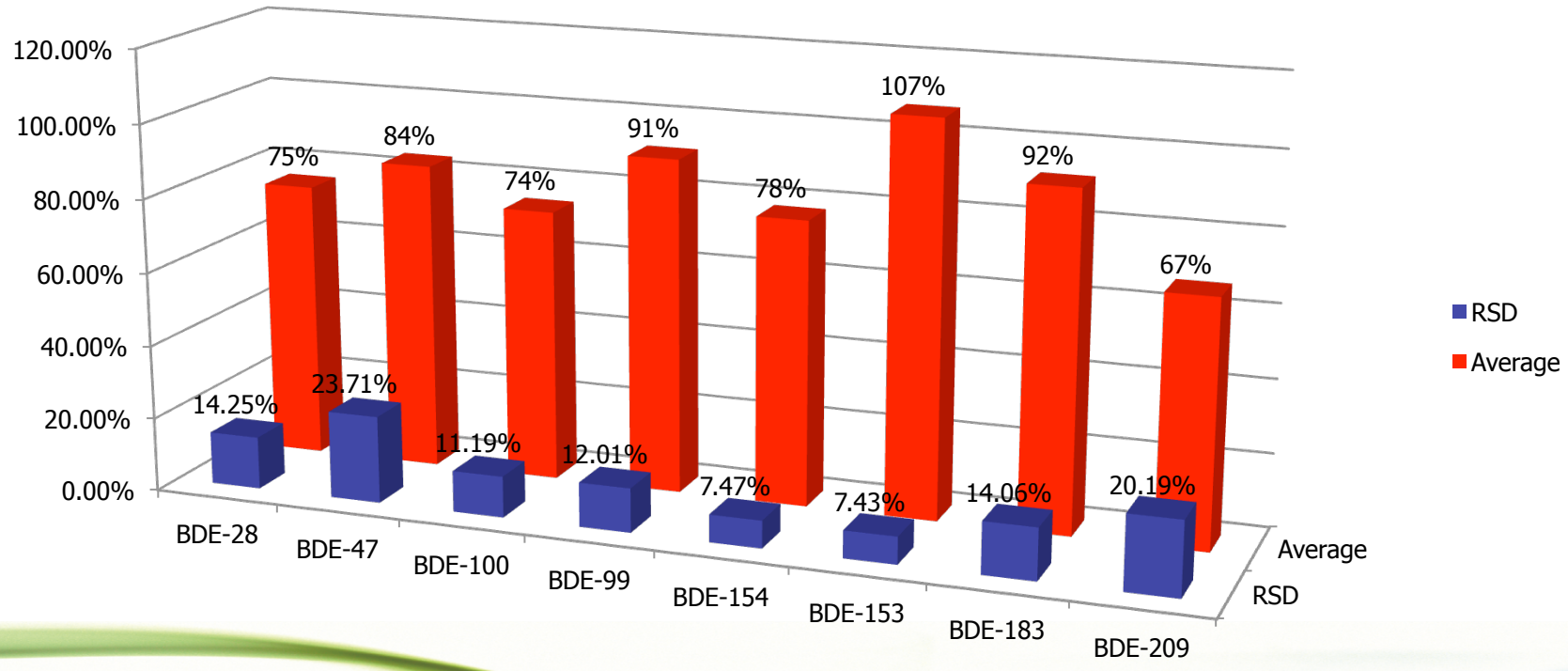
## Average Recovery



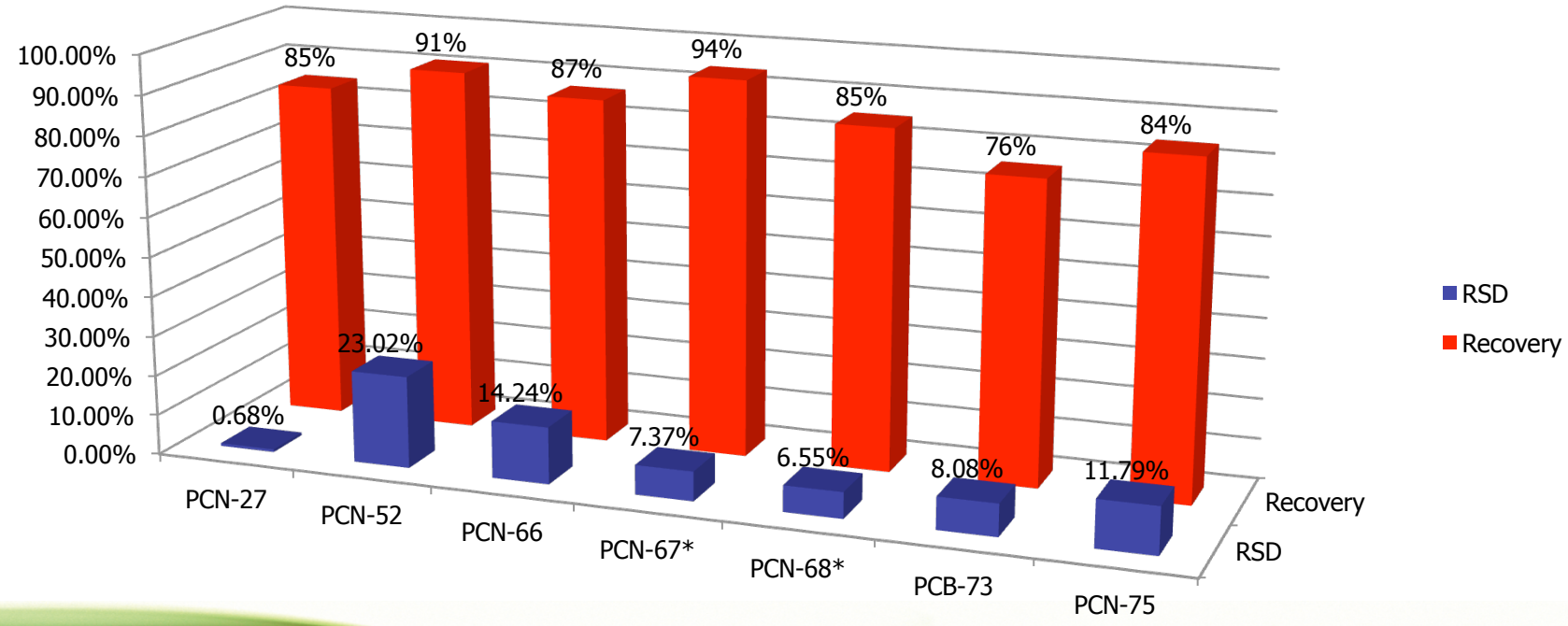
# PCDD/DF Labeled Recovery in a Soil Matrix



# 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 $AgNO_3$ sulfur removal (Activated copper required)
- OCPs show greater analyte loss with increasing amounts of acidified silica gel (not suitable for 10 gram samples)

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# Questions?