

Automating Solid Phase Extraction and Florisil Clean-up for Organochlorine Pesticides and PCB Aroclors



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- **EPA 508.1**
- **EPA 608**
- **EPA 8080/8081/8082**



Objective

- Develop a completely automated procedure combining the extraction and clean-up for chlorinated pesticides and PCB Aroclors utilizing Solid Phase Extraction
- Minimize manual steps to lessen error and maximize limited man hours



Reasons for SPE

- Reduced solvent
- Reduced glassware
- Simplified procedures
- Automation versus manual protocols =
Reproducibility



Determining Factors

- Extraction sorbent (C18, DVB, HLB, etc)
- Clean-up Sorbent (Florisil, Silica Gel, Alumina, NH₂)
- Extract drying agent (NaSO₄, MgSO₄, Diatomaceous Earth)
- Solvent compatibilities (EtAC, DCM, Hexane, Acetone, etc)





FMS TurboTrace ABN





FMS, Inc. SuperVap w/ direct to GC vial

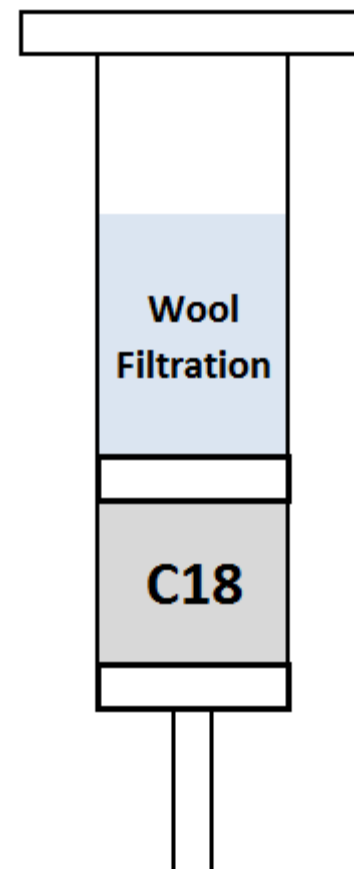
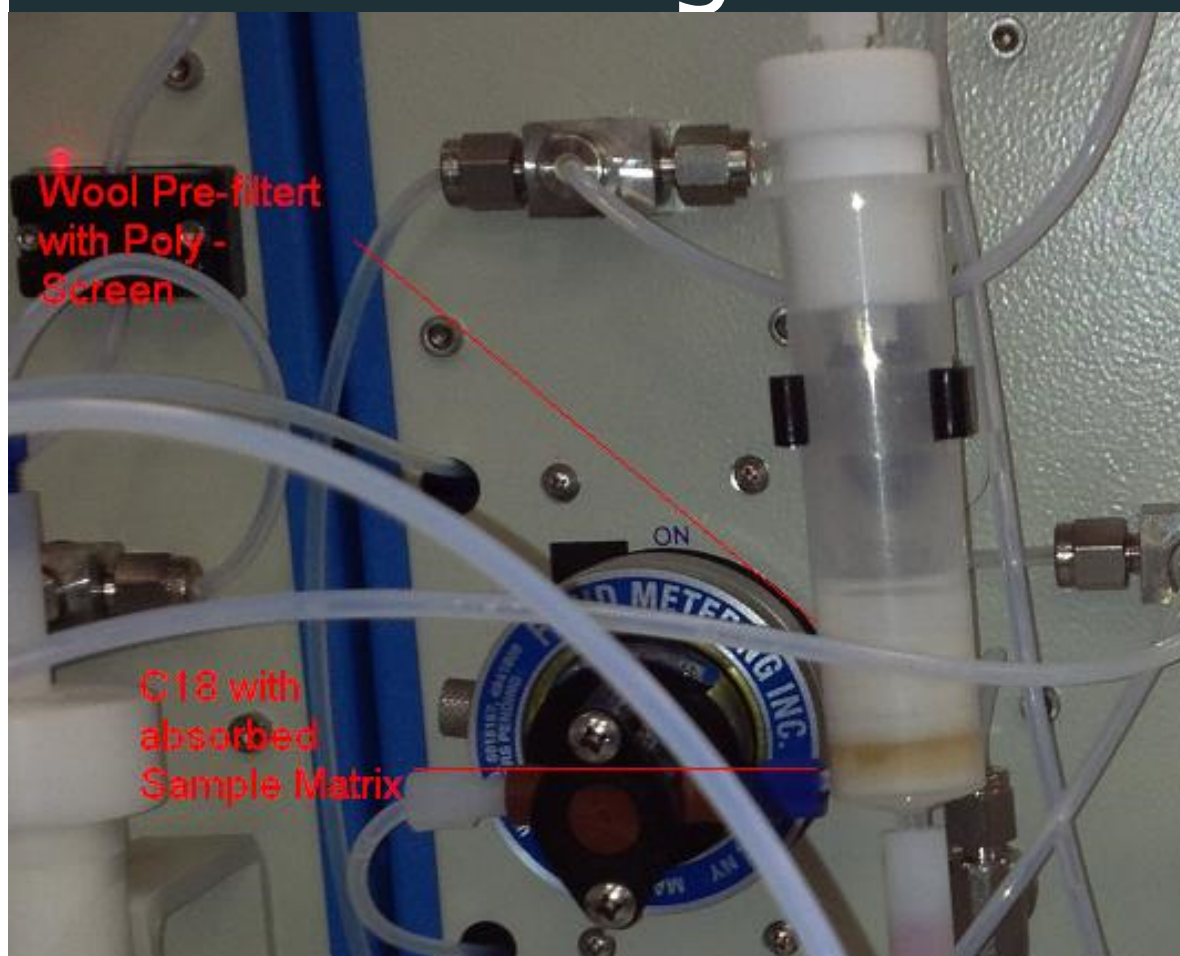


Extraction w/C18

- Advantages
 - Widespread current usage
 - Lower cost compared to DVB/HLB
 - Easier/shorted drying times
- Format
 - 1 gram sorbent amount to 1 liter sample
 - Ensure adequate matrix retention per unit volume
 - 25 ml cartridge
 - Sufficient surface area to reduce clogging
 - High enough flow rate to maintain smooth sample loading.



C18 Cartridge: Loading of a Dirty Matrix



Clean-up with Floirisil

- Offers suitability for all OCPs and PCBs
 - Dieldrin & Endrin
- Approved in all existing EPA methods
- Great application flexibility (amount, level of deactivation, elution gradient, etc)
- Can be utilized without the usage of Methylene Chloride*



Drying Agent

- NaSO_4
 - Widely used in manual procedures
 - Can be H_2O soluble
 - Prone to clumping
- MgSO_4
 - Wide spread usage of OPP/OPN application
 - Ultra fine particle size
 - Greater difficulty than NaSO_4 to in lab purify.

Drying Agent

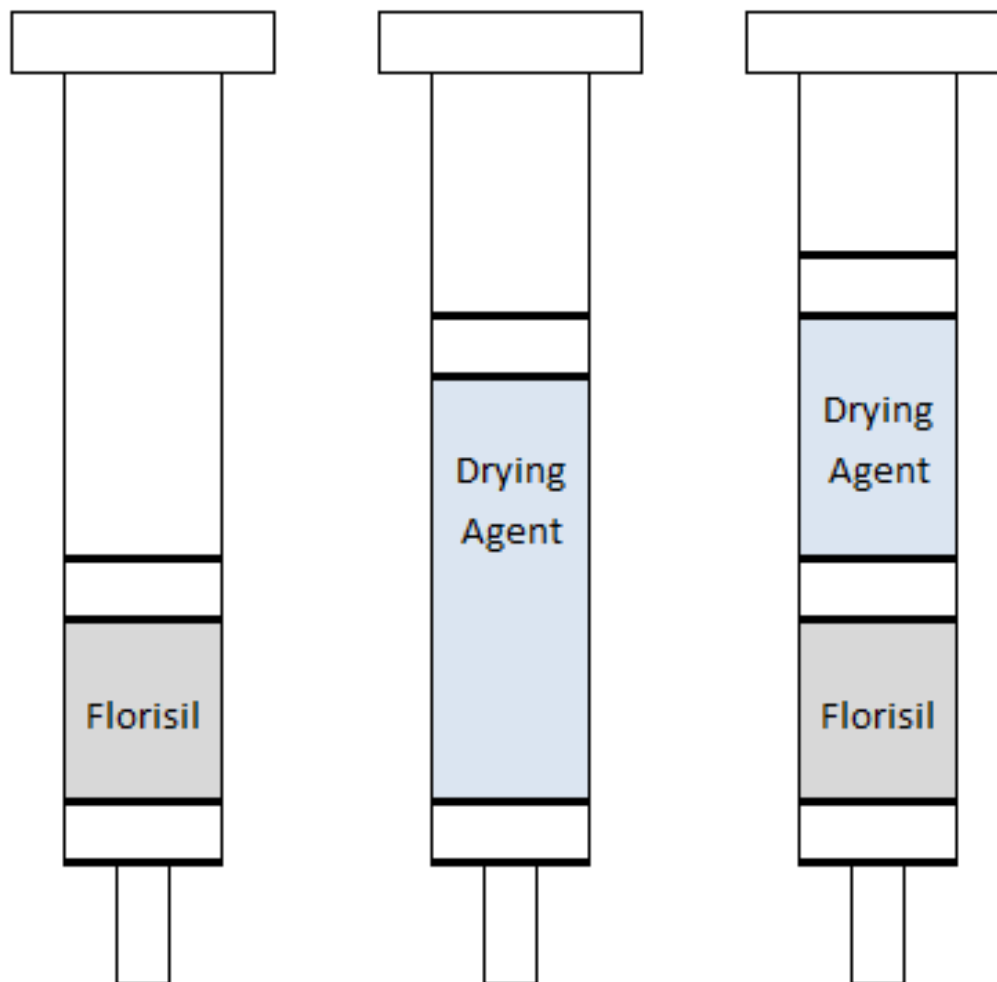
- Diatomaceous Earth
 - Silica based
 - Generally used for extraction drying for PFE
 - Good H₂O removal
 - Does not clump
 - Can be easily baked at 500C for easy purification
 - Large pellet size allows for smooth inline SPE flow.

Clean-up Cartridge(s)

- Packaged into a 6ml SPE format
- 1 gram of activated Florisil
- Drying agent can either be packaged in an independent 6 mls cartridge stacked, or layered in the same cartridge



Clean-up Cartridge(s)



Extract Evaporation

SuperVap 12 position concentrator with
direct to GC Vial vessels

Operating Temperature = 35 degrees C

Nitrogen Pressure = 5 PSI

Final Volume = 1ml



Extraction Procedure

- Condition clean-up Cartridge with elution solvent (10% Acetone in Hexane)
- Condition C18 Cartridge with elution solvent.
- Nitrogen purge off elution solvent
- Condition C18 cartridge with methanol
- Condition C18 cartridge with H₂O
- Load sample across C18 via vacuum
- Nitrogen dry C18 Cartridge



Elution Protocol

- Sample bottle rinsed with elution solvent
- Bottle rinse loaded and eluted across C18 and Clean-up cartridges, collected into FMS SuperVap concentrator
- Cartridges eluted with additional 20mls elution solvents.
- Cartridges nitrogen purged into SuperVap

Total elution solvent = 45mls of 90:10 Hexane/Acetone

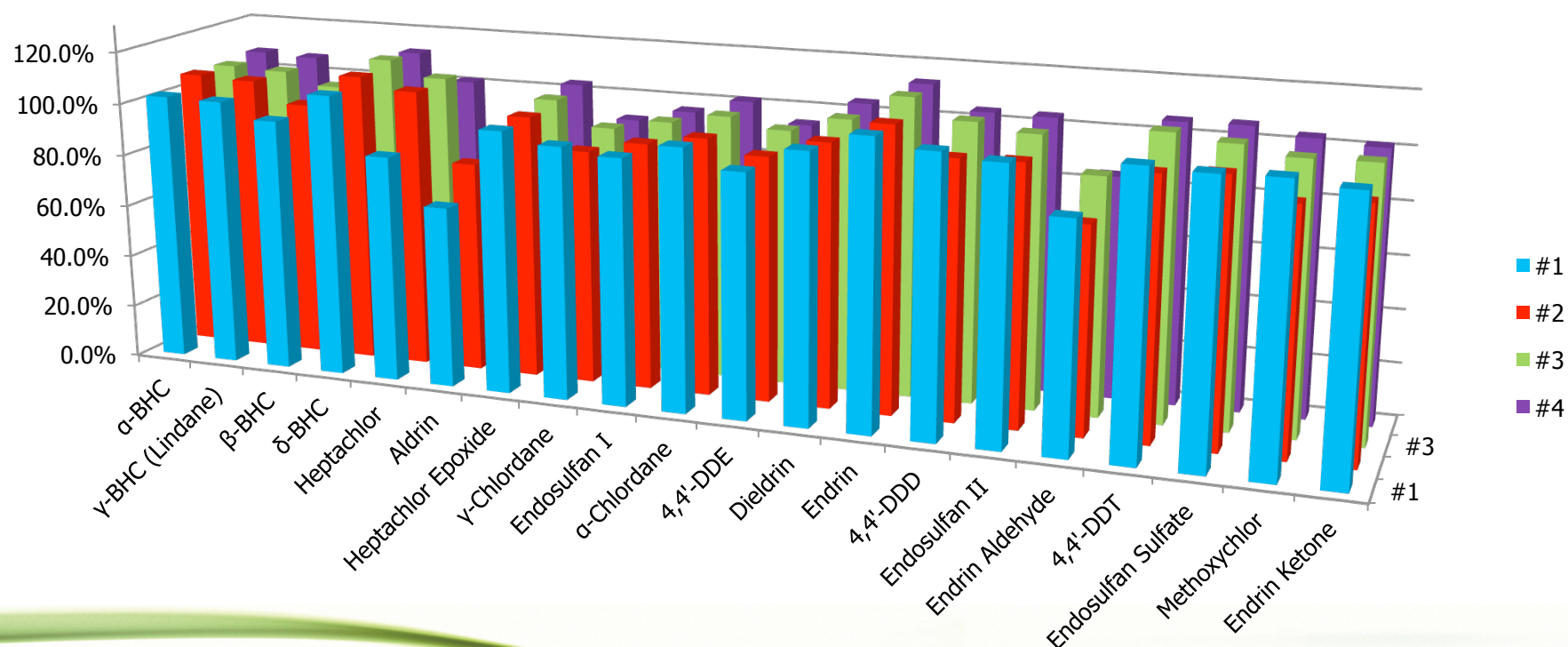


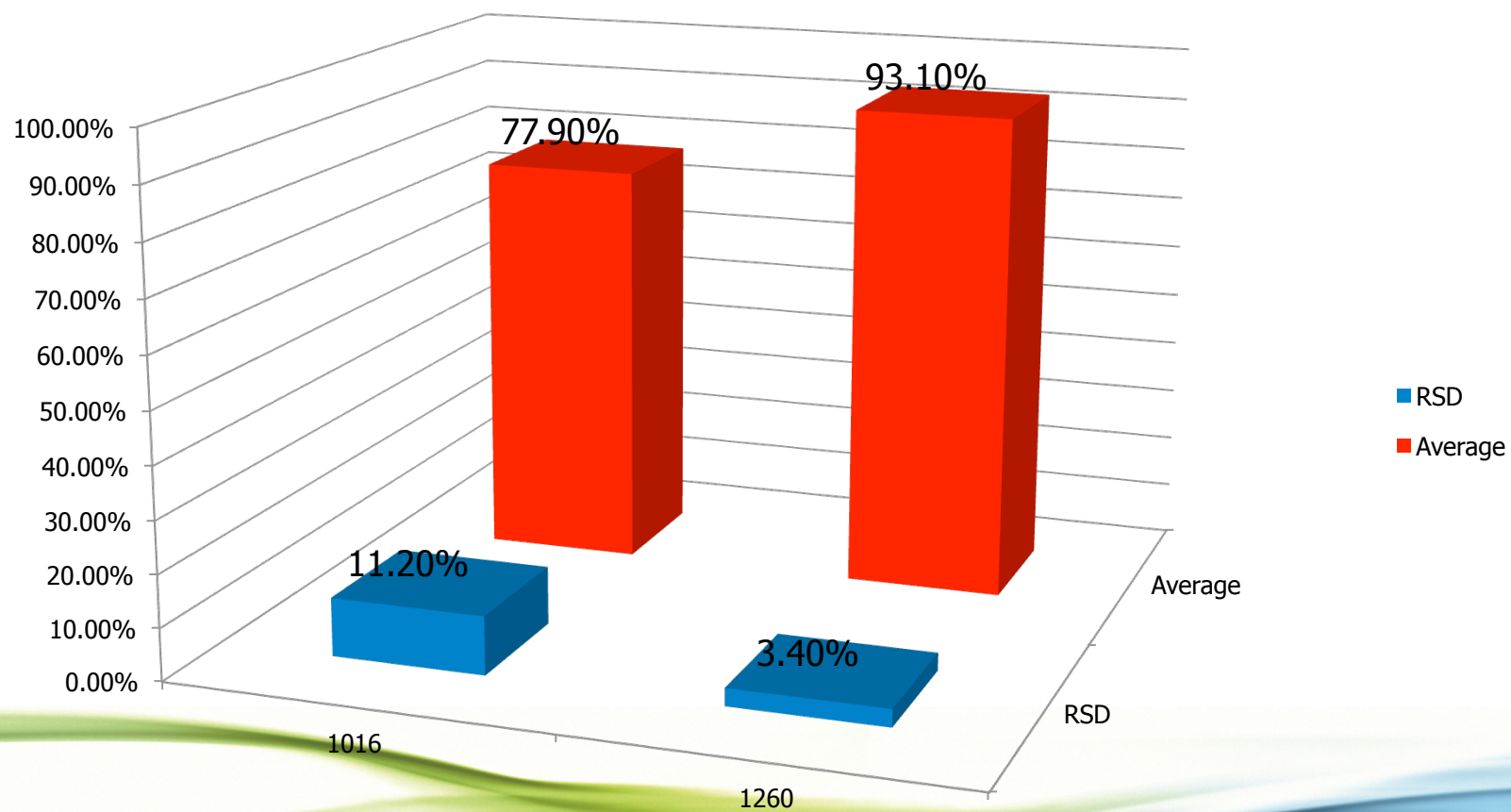
Analytical Platforms

- Agilent 7890 w/ μ ECD
- Thermo Trace GC, DSQ (MS Confirmation in live matrices)

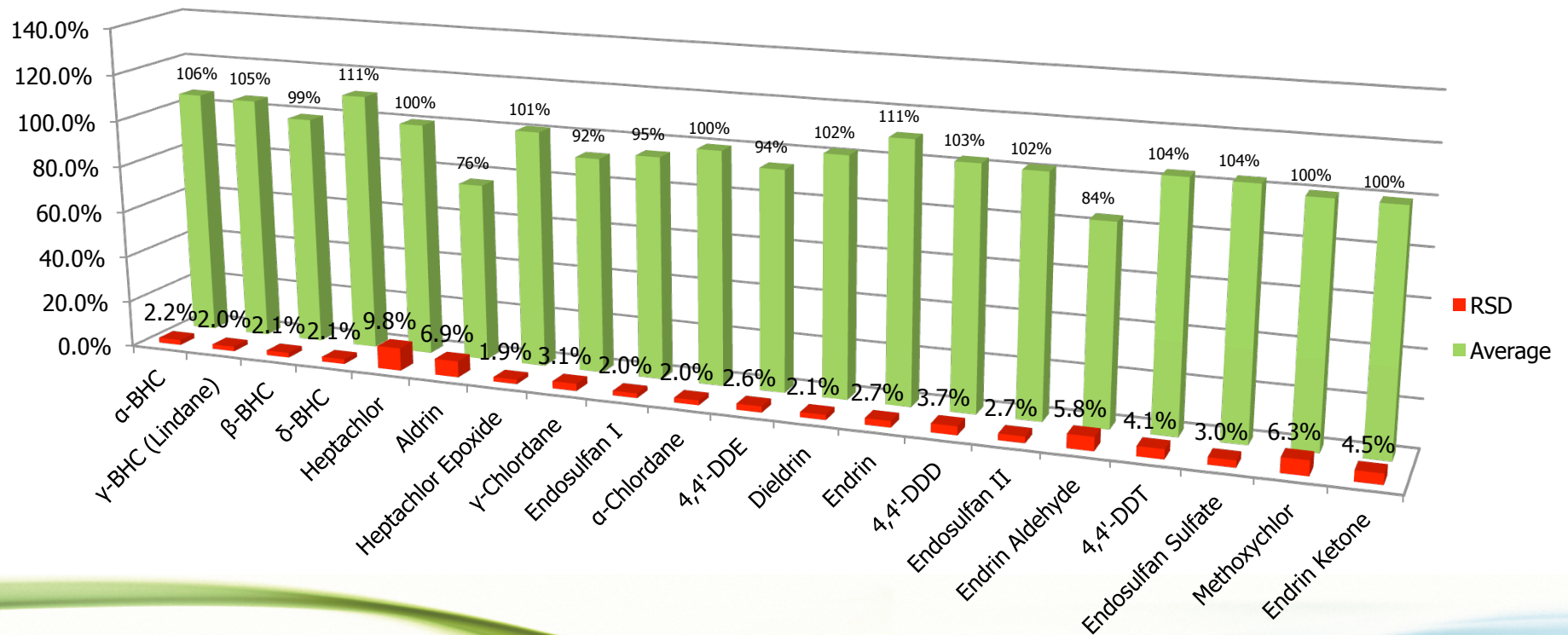


Data: IPR

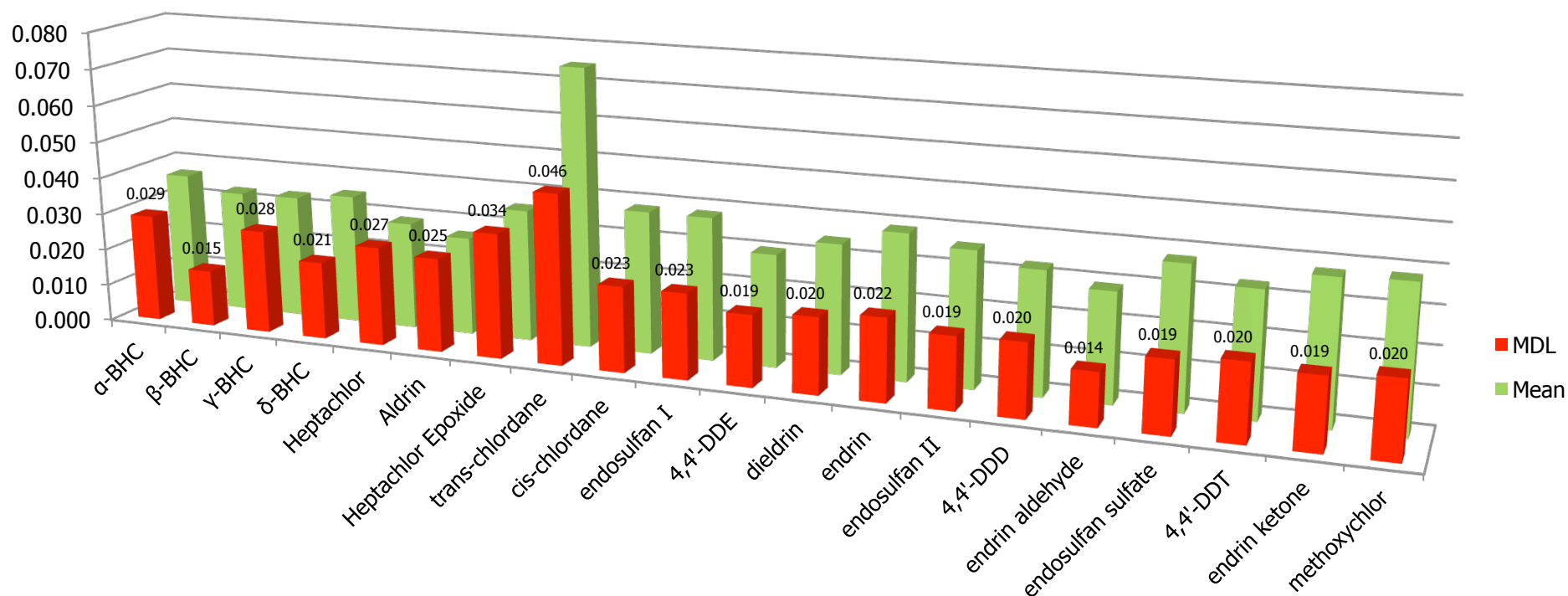




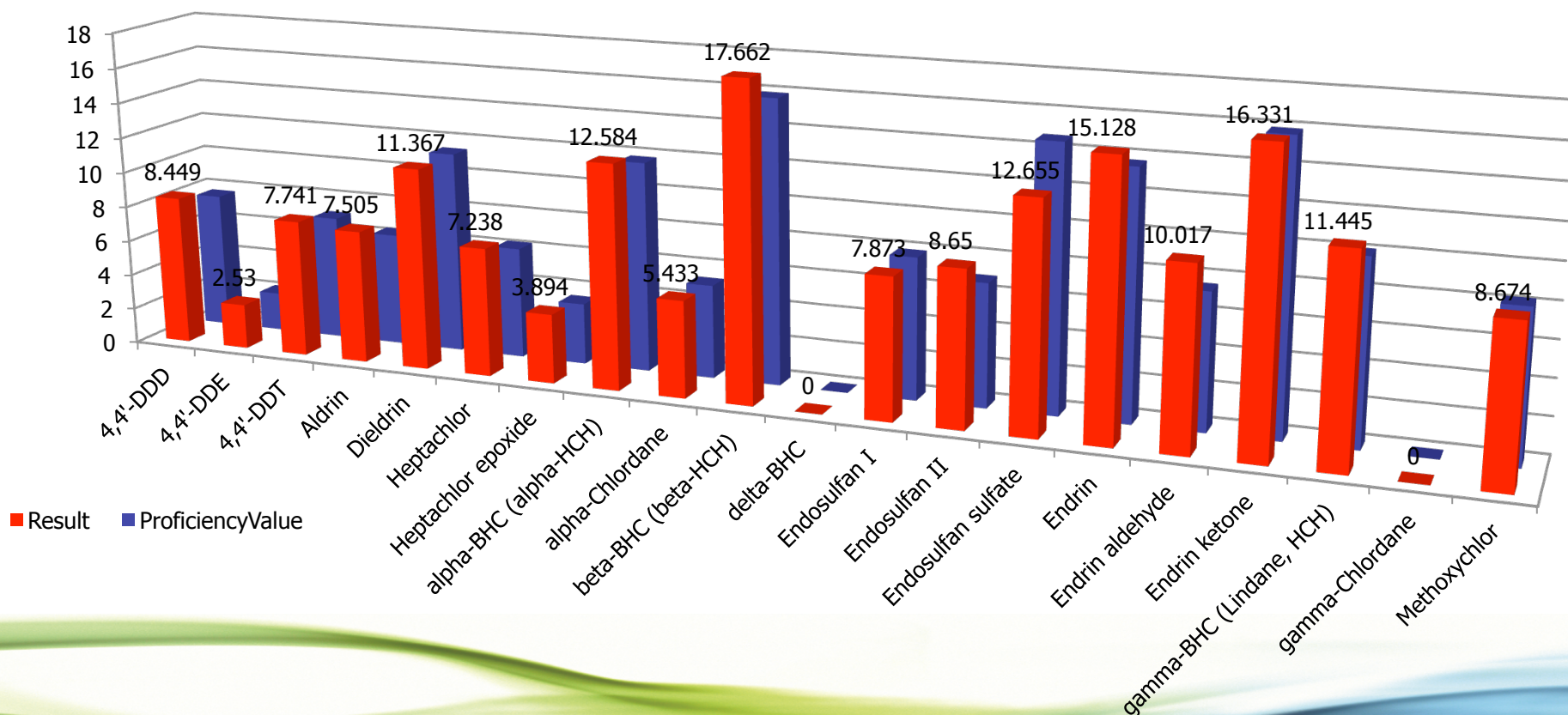
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Data MDL



Commercial PT Performance



Commercial PT Rank

<u>Analyte</u>	<u>Rank</u>	<u>Analyte</u>	<u>Rank</u>
4,4'-DDD	11/23	delta-BHC	-----
4,4'-DDE	13/23	Endosulfan I	1/23
4,4'-DDT	10/23	Endosulfan II	19/23
Aldrin	11/23	Endosulfan sulfate	4/23
Dieldrin	1/23	Endrin	13/23
Heptachlor	11/23	Endrin aldehyde	21/23
Heptachlor epoxide	13/23	Endrin ketone	7/22
alpha-BHC (alpha-HCH)	10/23	gamma-BHC (Lindane, HCH)	16/23
alpha-Chlordane	7/26	gamma-Chlordane	-----
beta-BHC (beta-HCH)	11/23	Methoxychlor	3/23

Conclusions

- All IPR recoveries within 70%-130%
- Capable of delivering clean blanks and acceptable MDLs
- Excellent performance in live sample matrices and blind PT studies
- Complete automation of the entire process from bottle to GC ready extract.



Questions?

