

# Reducing Instrument Downtime for Organochlorine Pesticide Sample Analysis Using an Optimized Graphitized Carbon Black Cartridge for Extract Cleanup

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Pure Chromatography

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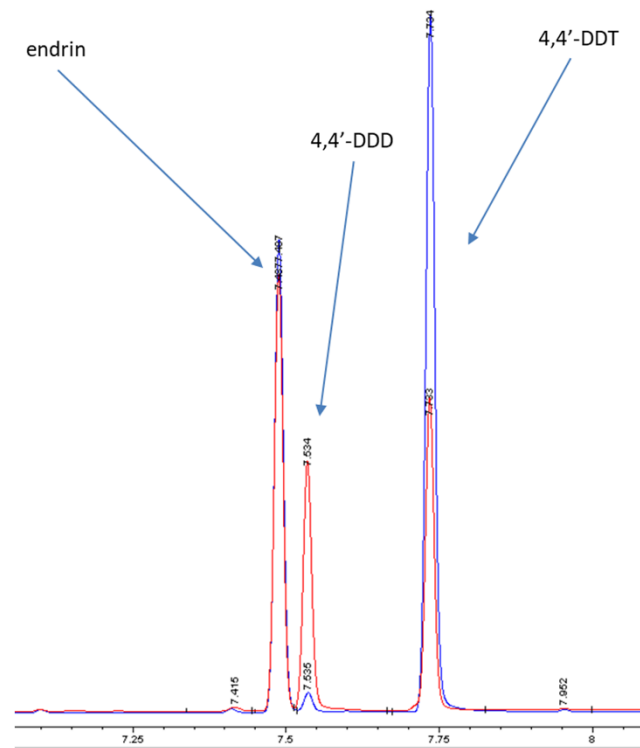
# EPA Method 8081 Organochlorine Pesticides in Waters, Soil, & Sludges

- Tests for legacy pesticides/degradation products
- Heavily employed method in environmental labs
- Analyzed using a selective detector (electron capture detection)
- General extraction procedure

# EPA 8081 Analyst Problems (pain points)

Complexity of matrix contributes to:

- Loss of instrument inertness
- Loss of instrument calibration
- Extraneous peaks in the chromatogram
- Column degradation
- Need a cleanup solution



## Two Solutions Already Exist in the Catalog

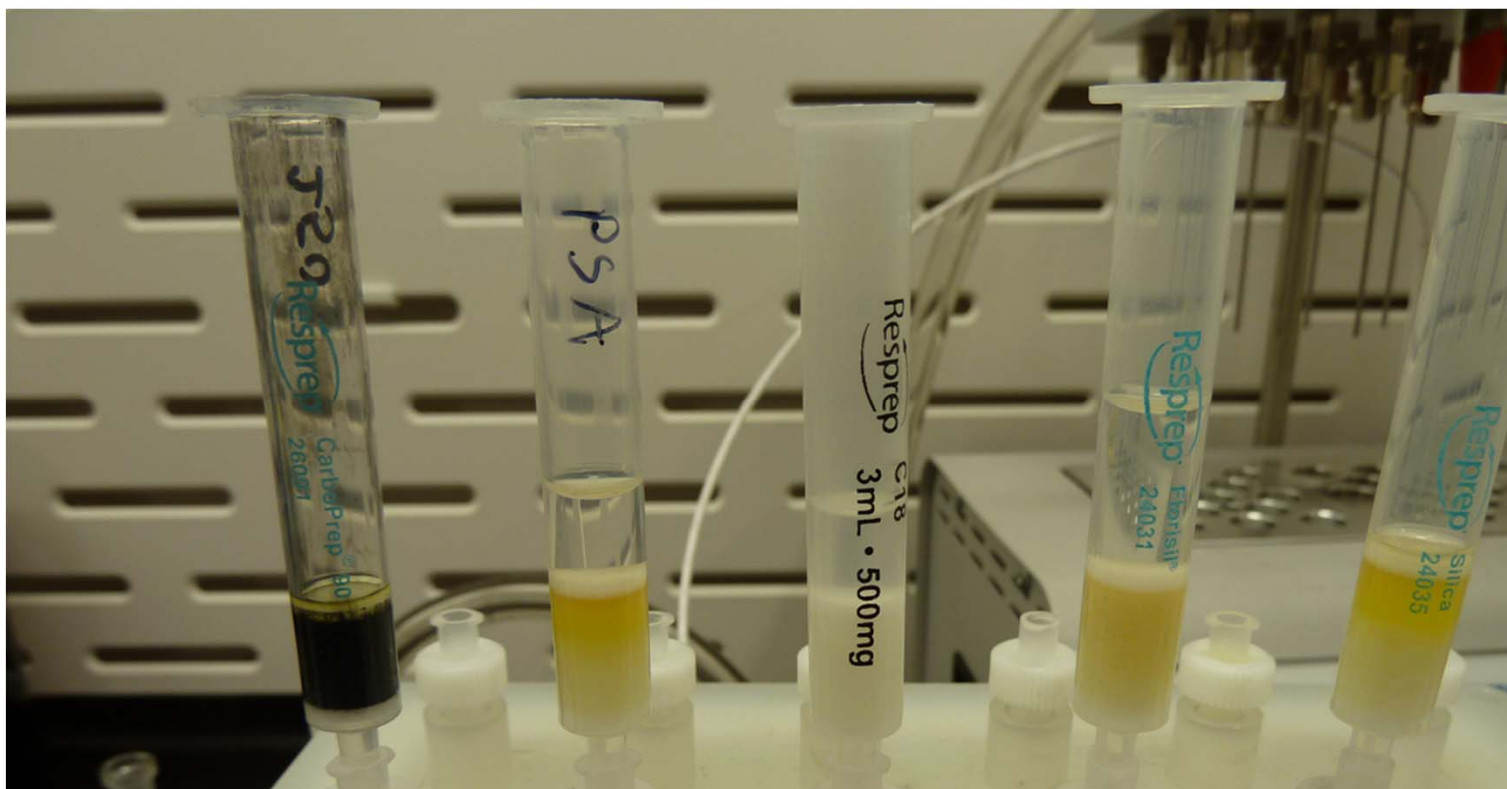
Florisil – recommended by EPA as Method 3620

- Good at small polar interferences (trichlorophenol)
- Does not handle nonpolar/larger molecular weight contaminants

GCB – people have adopted this format, but others are reluctant to

- Interferences from the cartridges a problem
- Retention for certain analytes highly variable
- Requires too much solvent volume/tube to elute retentive analytes
- Requires dichloromethane to elute retentive analytes

## Candidate Sorbents for Cleanup



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## Legacy Product Problems Solved

### Interferences contributed by the cartridge

- Carbon treatment – key attribute of new product
- Tube selection – evaluations conducted to ensure low background source was chosen
- Frit selection – ensures low extractable background from this cartridge component



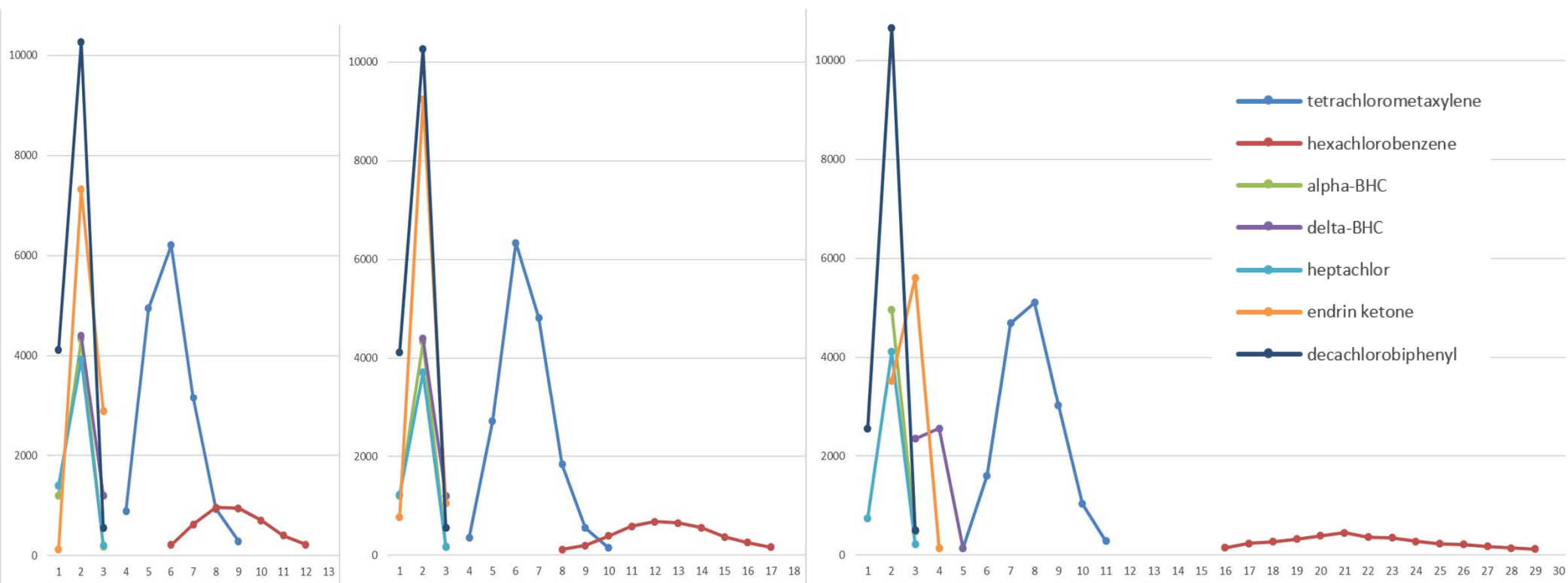
# Legacy Product Problems Solved

High variability and excess retention of certain analytes

- Treatment for cleanliness tempers retention
- Elution volume reduced for HCB to a consistent level, thereby virtually eliminating variability in selectivity for this analyte



# Lot to Lot Variation in Analyte Retention

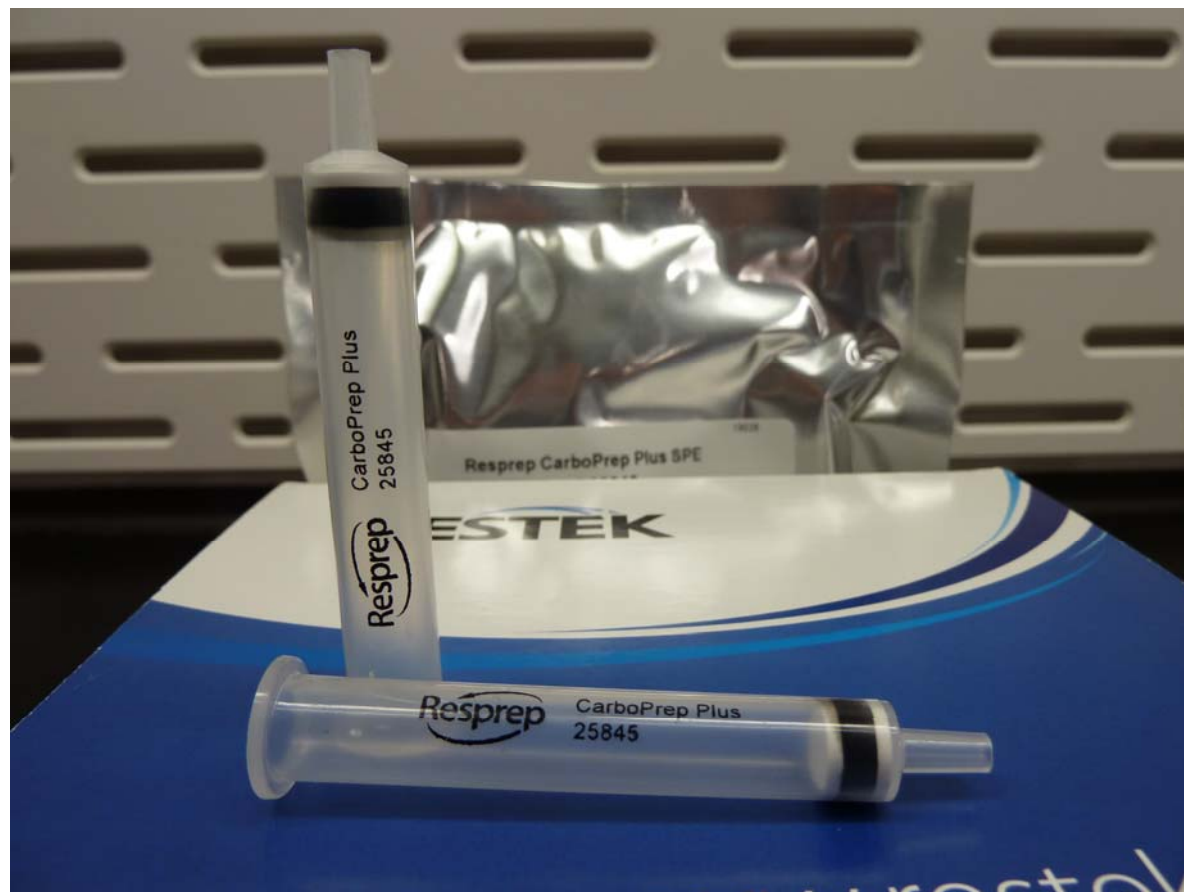


# Legacy Product Problems Solved

Large elution volume/dichloromethane required for some analytes

- What if we could get away w/Florisil method
- Reduce the mass of sorbent (95 mg)
- Loss of cleaning ability
- Drop in Florisil replacement

# The Resulting New Product



Keep Your Instruments Up and Running with  
**CarboPrep Plus SPE Cartridges**

- Maximum Contaminant Removal
- Less Instrument Downtime
- Higher Sample Throughput

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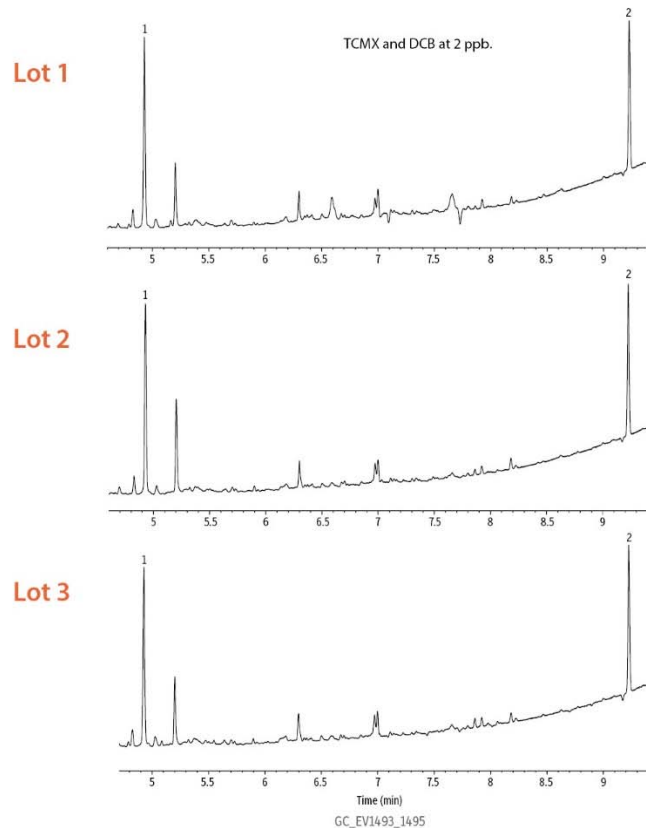


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# Cartridge Background



## Peaks

1. 2,4,5,6-Tetrachloro-*m*-xylene (TCMX)
2. Decachlorobiphenyl (DCB)

**Column** Rtx-CLPesticides, 30 m, 0.32 mm ID, 0.32  $\mu$ m (cat.# 11141)

**Sample** 2,4,5,6-Tetrachloro-*m*-xylene (cat.# 32027)  
Decachlorobiphenyl (BZ #209) (cat.# 32029)  
Hexane

## Diluent:

## Injection

Inj. Vol.: 4  $\mu$ L splitless (hold 0.75 min)  
Liner: Topaz 4.0 mm ID single taper inlet liner w/wool (cat.# 23303)  
Inj. Temp.: 250 °C  
Purge Flow: 50 mL/min

## Oven

Oven Temp.: 90 °C (hold 0.5 min) to 330 °C at 25 °C/min (hold 2 min)

**Carrier Gas** He, constant flow

**Flow Rate:** 3.5 mL/min

**Detector** Micro-ECD @ 340 °C

**Make-up Gas** Flow Rate: 50 mL/min

## Make-up

Gas Type: N<sub>2</sub>

Data Rate: 50 Hz

**Instrument** Agilent/HP6890 GC

## Notes

Conditioned a Resprep CarboPrep Plus SPE cartridge (cat.# 25845) by adding one cartridge volume of hexane:acetone (90:10) and letting it stand for five minutes before drawing the solvent down to frit level. Then, 1 mL of sample extract was loaded onto the cartridge and eluted with 9 mL of hexane:acetone (90:10).

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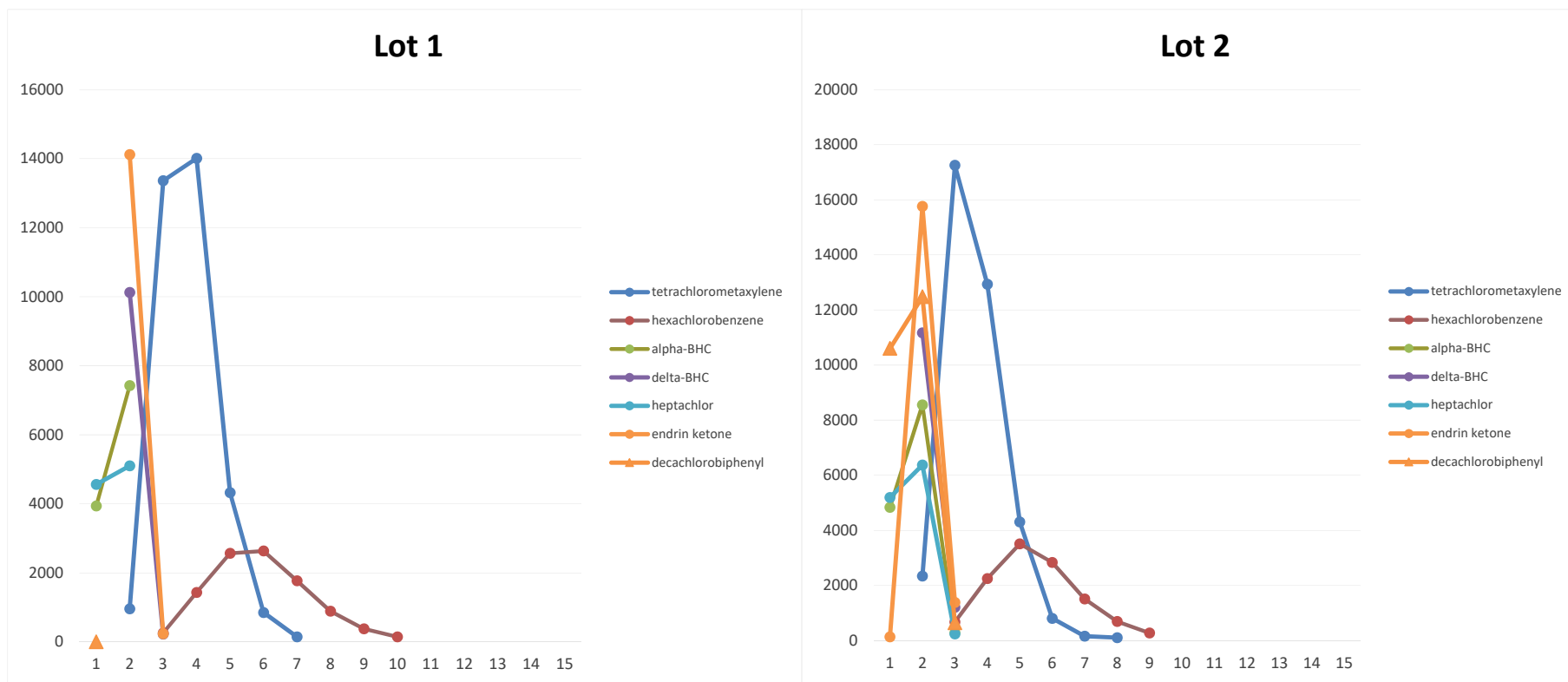
## Recovery of Key EPA 8081 Analytes

- Method requirement of 80 – 110% recovery of analytes
- Excellent recoveries with high reproducibility

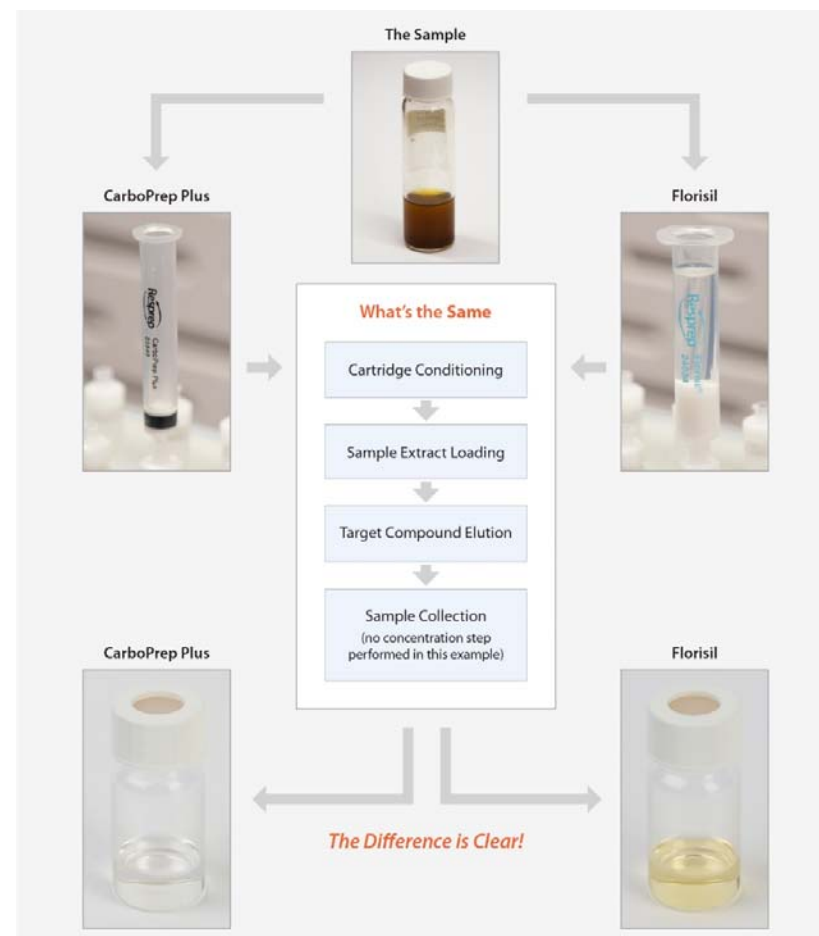
Compound	Average % Recovery*	% RSD
TCMX	99.8	1.3
hexachlorobenzene	97.6	2.3
alpha-BHC	99.2	1.3
gamma-BHC	98.1	1.4
beta-BHC	98.7	1.4
delta-BHC	98.7	1.7
heptachlor	101.2	1.7
aldrin	97.1	1.7
heptachlor epoxide	98.6	1.6
gamma-chlordane	98.2	1.8
alpha-chlordane	96.9	2.2
4,4'-DDE	96.8	1.6
endosulfan I	98.0	1.4
dieldrin	97.8	1.4
endrin	98.4	1.6
4,4'-DDD	97.4	1.4
endosulfan II	94.6	5.5
4,4'-DDT	98.2	1.2
endrin aldehyde	96.1	1.2
methoxychlor	100.2	2.1
endosulfan sulfate	97.1	2.2
endrin ketone	98.1	0.8
DCB	97.8	2.2

\* n = the analysis of 6 cartridges each from 3 different lots for a total of 18 data points

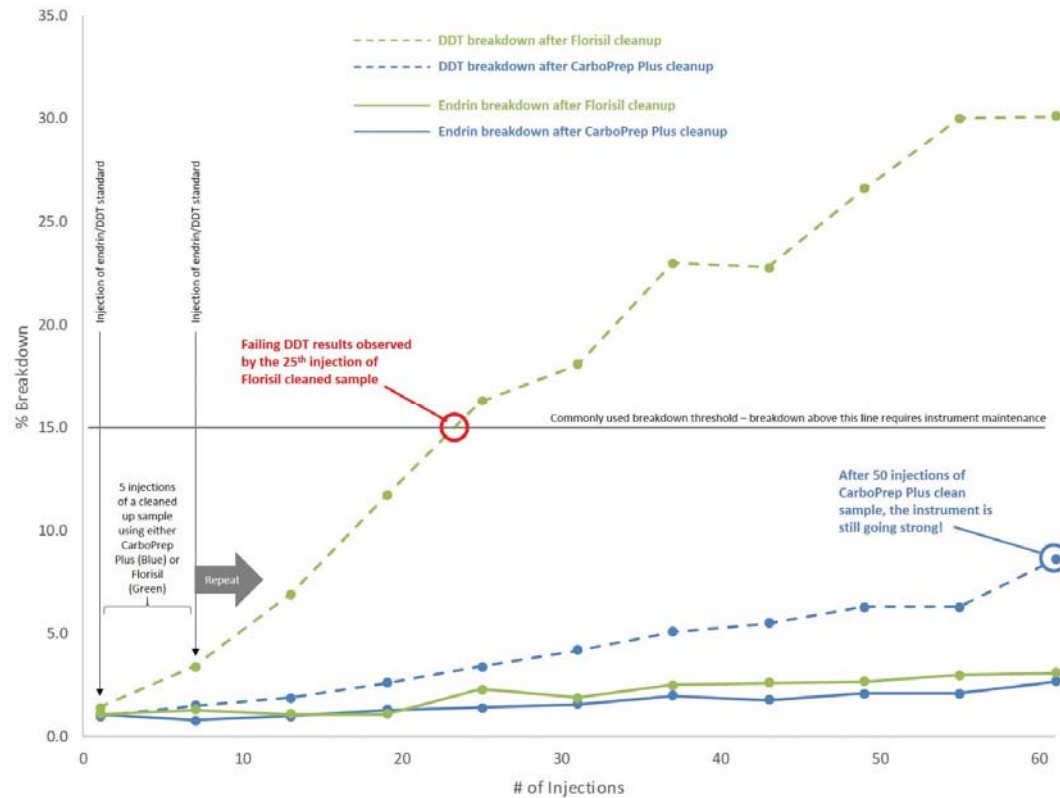
# Retention of Key EPA 8081 Analytes



# Visual comparison of cleanup capabilities of CarboPrep Plus versus Florisil



# Endrin/DDT Breakdown Comparison





Comments/Questions?



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