RESTEK Pure Chromatography

Reduced Instrument Downtime for Organochlorine Pesticide Analysis by Using an Optimized SPE Cartridge for Sample Extract Cleanup

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Abstract

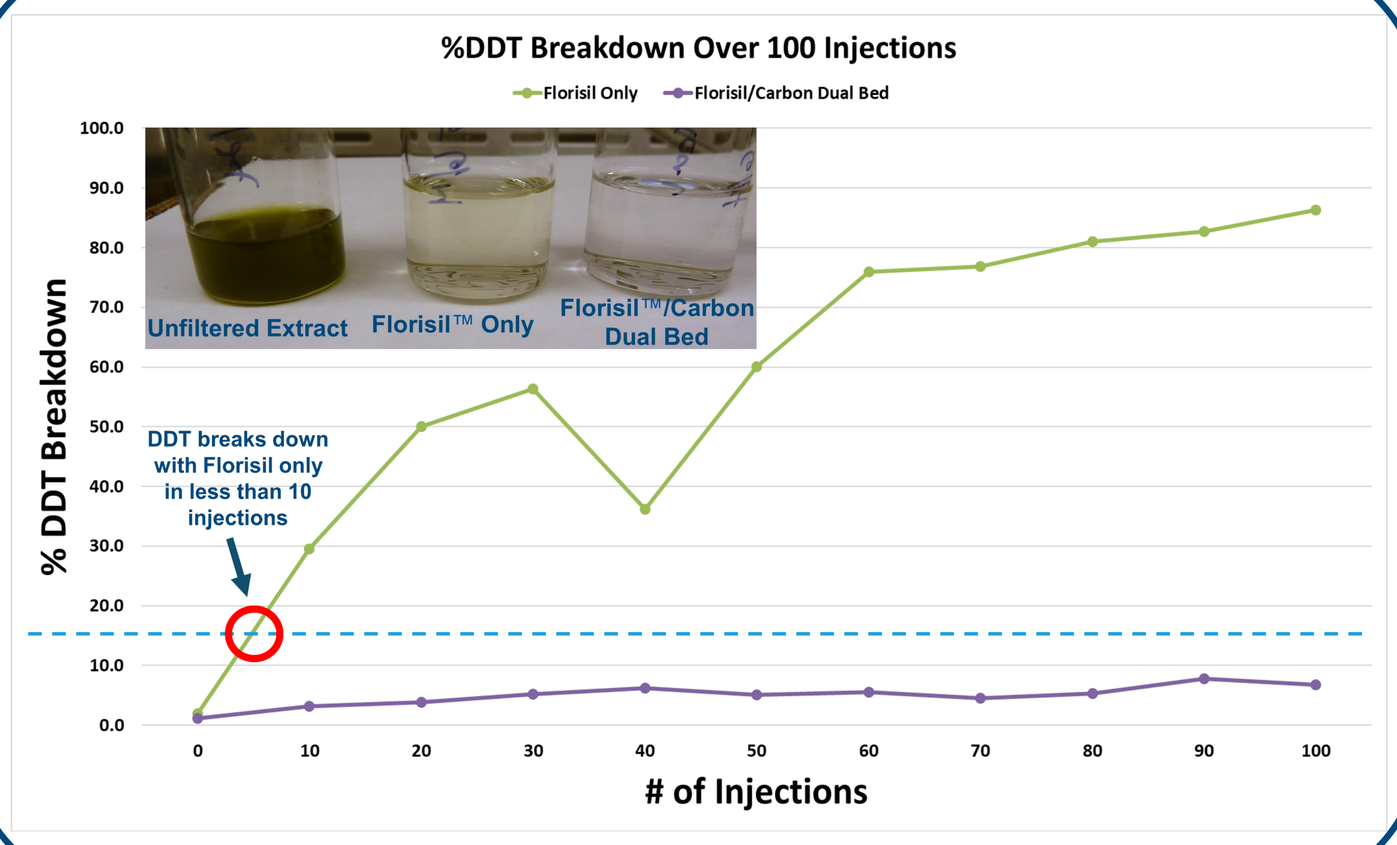
Some of the more commonly encountered problems experienced by those analysing environmental samples for organochlorine pesticides are instrument downtime and shortened calibration periods both due to the deleterious effects of coextracted matrix components that are introduced into the analytical instrument during sample injection. In addition to this, chromatographic interferences complicate identification and quantification and make life difficult for environmental analysts. Although there are cleanup options provided such as Florisil™, silica gel, and alumina, these normal phase solutions often do not adequately remove the less polar and high molecular weight compounds that are responsible for diminishing instrumental performance and sample path inertness.

In this presentation, a cartridge is introduced that is designed specifically to be utilised exactly like the frequently employed Florisil™ cartridge, but with a drastic improvement in results when used with highly pigmented and inlet degrading samples. What this means for the analyst is the ability to consistently generate calibration curves that can be maintained longer and reduced instrument maintenance, ultimately leading to higher sample throughput.

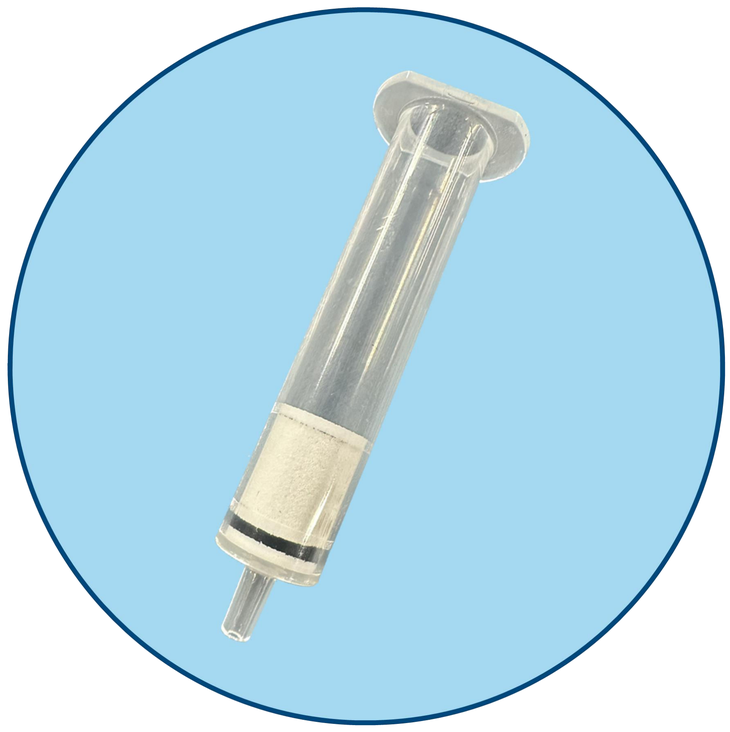
Current Workflow

Laboratories following EPA SW-826 test method 3620C use Florisil™ SPE cartridges for the cleanup of sample extracts. Method 3620C can be used for EPA method 8081B, for the analysis of organochlorine pesticides by gas chromatography. Alternatively, for extremely dirty samples, laboratories will use a carbon SPE cleanup in place of the Florisil™ SPE cartridge. Whilst Florisil™ is an effective cleanup, it does not remove humic acid, which contributes to the breakdown of endrin and DDT. Presented is a solution combining both Florisil™ and Carbon into a single SPE cartridge to enhance the cleanup efficiency of problematic samples, thus reducing instrument downtime.

1. Florisil™/Carbon Dual Bed vs Florisil™ Only

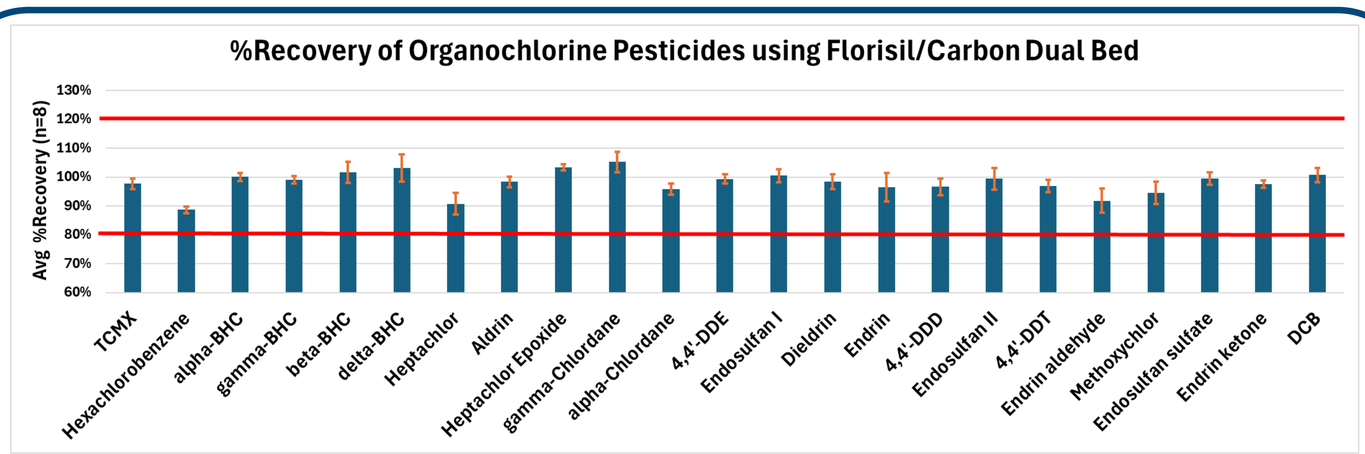


A Single Step Solution



Florisil™/Carbon Dual Bed Performance

2. Pesticide Recoveries



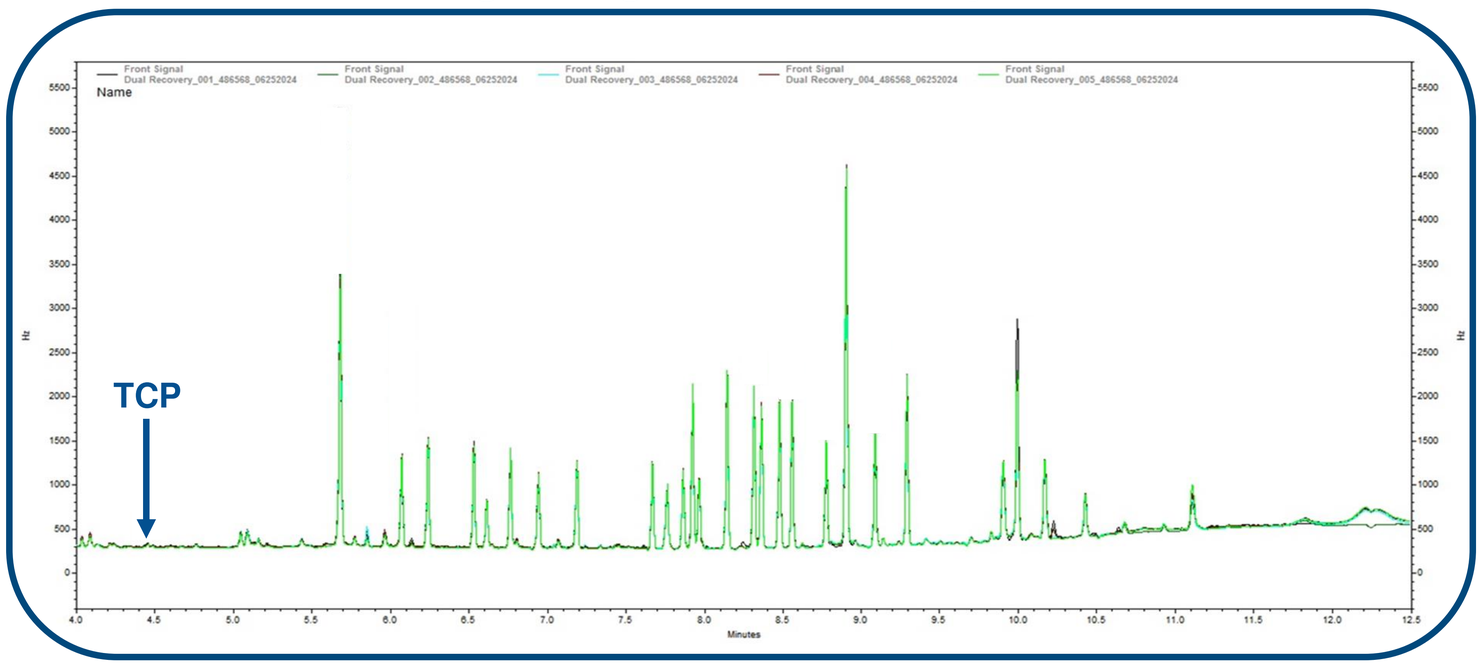
The performance of the Florisil™/Carbon dual bed SPE cartridge can be seen in the three figures to the left.

1. A sample containing soil, tea, and spiked with humic acid was prepared and used to compare Florisil™ only and Florisil™/Carbon cartridges. 100 injections were made after collecting extracts from both cartridges to understand when each cartridge would fail against the 15% DDT breakdown limit. The Florisil™ only cartridge failed in <10 injections, while the Florisil™ /carbon cartridge was able to complete all 100 injections without surpassing 15% DDT breakdown.

2. To evaluate pesticide recoveries, 8 samples were processed using the Florisil™/carbon dual bed. All 24 pesticides fell within the acceptable recovery range of 80-120% while achieving %RSDs of ≤5%.

3. Per EPA 8081B, acceptable SPE cartridges containing Florisil™ will result in <5% recovery of trichlorophenol (TCP). Over the same 8 samples evaluating pesticide recoveries, TCP was showed <1% recovery, exceeding method requirements.

3. TCP Breakthrough (Avg. <1% Recovery, n=8)



Conclusions

Florisil™ only cartridges have proven to be problematic for the cleanup prescribed in EPA 3620C for method 8081B. While this cleanup can be successful for some samples, samples classified as very dirty or containing interferences require carbon in addition to the Florisil™. The presented solution, Florisil™/carbon dual bed SPE, has proven itself as an effective cleanup for these difficult sample types by removing problematic compounds, like humic acid. In addition, the Florisil™/carbon dual bed meets method requirements with all pesticides of interest being recovered between 80-120% and having <5% TCP recovered. The Florisil™/carbon dual bed shows its great advantage in sample cleanup allowing laboratories to reach at least 100 injections without DDT breakdown exceeding 15%, leading to longer instrument uptime and less maintenance.

References

1. **U.S. Environmental Protection Agency.** Method 3620C: Florisil Cleanup. *EPA.* [Online] July 2014.
2. **U.S. Environmental Protection Agency.** Method 8081B: ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY. *EPA.* [Online] February 2007.

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