#59351B

RESTEK environmental

Applicationsnote

Stx™-CLPesticides and Stx™-CLPesticides2 Columns Provide Improved System Inertness for Chlorinated Pesticides Analysis

Many laboratories performing gas chromatography (GC) analysis of chlorinated pesticides struggle with endrin breakdown caused by the compound interacting with or adsorbing to active sites throughout the analytical system, most notably in the injection port and the analytical column. Restek Siltek™ technology—used successfully to passivate injection port liners and guard columns—is now available for column technology and is used in Stx™-CLPesticides and Stx™-CLPesticides2 capillary columns. The combination of a properly deactivated injection system and inert analytical columns provides the lowest possible level of endrin breakdown.

Method Requirements

Chlorinated pesticide analysis following US Environmental Protection Agency (EPA) Methods 8081, 608, 505 and 508 recommend dual-column confirmation using electron capture detection (ECD). The compounds in Figure 1 represent some of the more common analytes. As in all analytical methods, the instrument used for quantitative analysis must be calibrated to ensure accurate results are reported. For chlorinated pesticides this usually entails a calibration curve of three to five points and check standards injected at specified time intervals during sample analysis. In addition, performance standards containing endrin are analyzed periodically to ensure system inertness. Typically, endrin breaks down to endrin aldehyde and endrin ketone when there are active sites in the sample pathway.

Endrin Breakdown

Maintaining a low breakdown level for endrin is necessary for laboratories analyzing chlorinated pesticides. Reduction of endrin breakdown generally focuses on improving the inertness of the injection port. Traditionally, deactivated injection port liners have been used for this analysis but, more recently, liners treated with Siltek™ passivation have been proven to further reduce endrin breakdown. This innovative passivation technology also was incorporated into capillary guard tubing so that the entire sample introduction pathway is inert for pesticide analysis.

Endrin Response on Capillary Columns

Through experimentation we have found that the columns typically used for pesticide analysis exhibit low response for endrin compared to other pesticides eluting in the same region even though the endrin breakdown products are not present. This indicates that endrin is being adsorbed by active sites in the sample pathway, thus effectively reducing endrin response. To minimize the on-column adsorption of endrin, we incorporated Siltek™ passivation technology into the analytical columns.

Combining Siltek™ passivation with the unique selectivity of Restek CLPesticides phases results in the Stx™-CLPesticides and Stx™-CLPesticides2 columns, and a significant improvement in endrin response (Figures 1 & 2). Using the Stx™-CLPesticides and Stx™-CLPesticides2 columns, the endrin peak response is notably higher than the analytes eluting in the same region—something not always seen in columns using traditional deactivations.

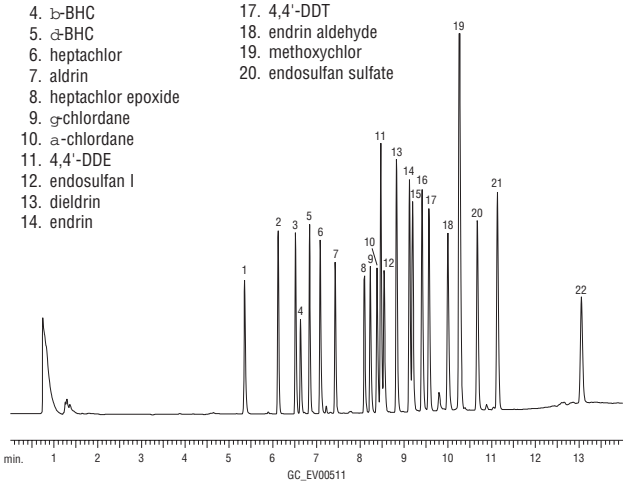
Column Installation and Optimizing Resolution

The Stx™-CLPesticides2 column is the ideal confirmational column to the Stx™-CLPesticides column. They were designed to achieve resolution of the chlorinated pesticides using the same backpressure and oven temperature program. The columns can be installed in parallel using a glass universal Press-Tight® "Y" connector or a metal MXT® "Y" connector (Figure 3). This parallel set-up reduces downtime caused by maintenance of multiple injection ports. Of course, these columns can also be installed in separate injection ports and mounted in the same GC oven.

Figure 1

The Stx®-CLPesticides column provides the ultimate in inertness for active pesticides such as endrin, DDT, and methoxychlor.

1. 2,4,5,6 -tetrachloro-*m*-xylene (ss)
2. a-BHC
3. g-BHC
4. b-BHC
5. d-BHC
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. g-chlordane
10. a-chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl (ss)



30m, 0.32mm ID, 0.50µm Stx-CLPesticides (cat# 11544)
Oven temp.: 120°C (hold 1 min.) to 300°C @ 9°c/min.; **Det.:** 310°C, Agilent ECD; **Inj.:** 1µL, pesticide standard mix A and B (cat.#32003,32004, 20/40/200ng/mL); **Inj. temp.:** 220°C (splitless, 1 min. purge off hold); **Inlet liner:** 4mm Siltek™ single gooseneck (cat.# 20798-214.1); **Dead time:** 0.9 min. @ 120°C.

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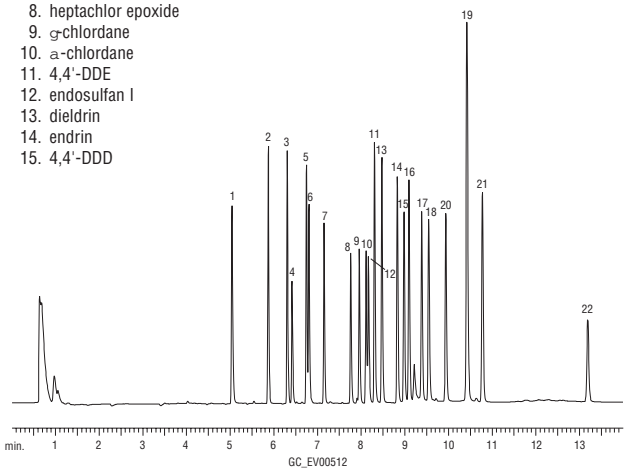
Figure 2

The Stx™-CLPesticides2 column is the ideal confirmation column to the Stx™-CLPesticides column for chlorinated pesticides analysis.

1. 2,4,5,6-tetrachloro-m-xylene (ss)
2. a-BHC
3. g-BHC
4. b-BHC
5. d-BHC
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. g-chlordane
10. a-chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl (ss)



30m, 0.32mm ID, 0.25µm Stx-CLPesticides2 (cat# 11444)
Oven temp.: 120°C (hold 1 min.) to 300°C @ 9°C/min.; **Detector:** 310°C, Agilent ECD;
Inj.: 1µL, pesticide standard mix A and B (cat.#32003,32004, 20/40/200ng/mL);
Inj. temp.: 220°C (splitless, 1 min. purge off hold); **Inlet liner:** 4mm Siltek™ single gooseneck (cat.# 20798-214.1); **Dead time:** 0.8 min. @ 120°C.

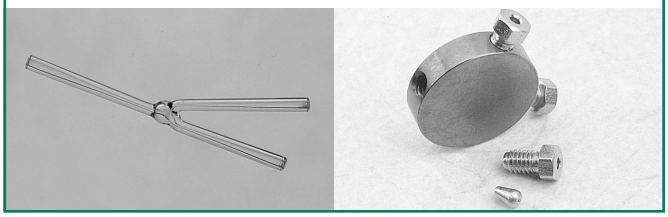
The key to maximizing resolution with these columns is setting the proper flow rate. The elution order of endosulfan I and 4,4'-DDE on the Stx™-CLPesticides column are used to indicate optimized flow rate; 4,4'-DDE should elute between a-chlordane and endosulfan I. If 4,4'-DDE and endosulfan I are not adequately resolved, decreasing the column flow will improve separation. Once resolution is acceptable for these two compounds, then the remaining compounds will be resolved in a dual-column single injection port configuration.

Conclusion

Endrin breakdown and adsorption are caused by active sites throughout the analytical system, especially the injection port and column. Using Siltek™-treated liners, guard columns, and Stx™-CLPesticides and Stx™-CLPesticides2 analytical columns for chlorinated pesticides analyses results in a chromatographic system with unsurpassed inertness, allowing for longer calibration periods.

Figure 3

Universal Press-tight® 'Y' connector and metal MXT® 'Y' connector.



Stx™-CLPesticides Column

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.25	-60 to 310/330°C	11540	11543
0.32mm	0.50	-60 to 310/330°C	11541	11544
0.53mm	0.50	-60 to 310/330°C	11542	11545

Siltek™ Guard Tubing

ID	5-Meter	10-Meter
0.25mm	10026	10036
0.32mm	10027	10037
0.53mm	10028	10038

Connectors

Description	Each	3-pack
Universal 'Y' Press-tight	20405	20406
MXT low dead-volume 'Y' (0.28mm)	20396	—
MXT low dead-volume 'Y' (0.53mm)	20395	—

Stx™-CLPesticides2 Column

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.20	-60 to 310/330°C	11440	11443
0.32mm	0.25	-60 to 310/330°C	11441	11444
0.53mm	0.42	-60 to 310/330°C	11442	11445

Stx™-CLPesticides Capillary Column Kits

Includes an Stx™-CLP column, an Stx™-CLP2 column, Siltek™ guard tubing, and a Universal 'Y' Press-Tight® Connector

0.25mm (cat.# 11190); **0.32mm** (cat.# 11191); **0.53mm** (cat.# 11192)

Siltek™ Inlet Liners

Qty.	Siltek™	Siltek™ w/Siltek™ Wool	Siltek™ w/Carbofrit™
each	-214.1	-213.1	-216.1
5-pack	-214.5	-213.5	-216.5
25-pack	-214.25	-213.25	-216.25

For Siltek™-deactivation, include the suffix number shown above to the inlet liner catalog number.