

Minimizing Breakdown of Chlorinated Pesticides Using Siltek™-Deactivated GC Accessories

Gas chromatographic (GC) analysis of chlorinated pesticides presents unique challenges to environmental laboratories because these compounds often are at trace levels and susceptible to decomposition. Some analyses, such as the US Environmental Protection Agency (EPA) Methods 8081 and 608, have stringent breakdown and reproducibility criteria. Breakdown occurs when a compound decomposes into related compounds, generally because a thermal or chemical reaction has occurred. Two compounds notorious for exhibiting breakdown are endrin, which breaks down into endrin aldehyde and endrin ketone, and DDT, which breaks down into DDE and DDD. The source of breakdown can be aged samples and standards, the GC column or, most commonly, active sites in the GC injection port. Routine maintenance of the injection port, prevention of sample flashback, and thorough deactivation of the inlet liner and GC columns are essential to minimize compound breakdown.

We chose a pesticide analysis to compare Siltek™-deactivated products against undeactivated products. To evaluate inlet liner effects on endrin and DDT breakdown, an HP 5890 GC equipped with an Rtx®-CLPesticides2 column and an electron capture detector (ECD) was used. A 50pg/μL standard of tetrachloro-*m*-

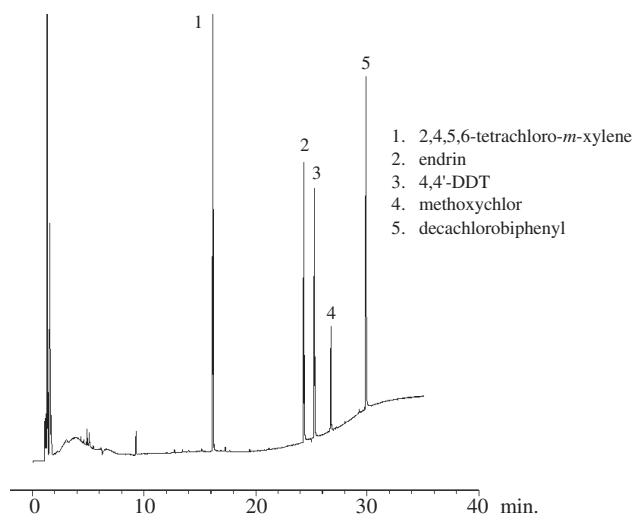
xylene, endrin, 4,4'-DDT, methoxychlor, and decachlorobiphenyl was injected directly onto the column and onto several different liners. The area of the breakdown products was then measured. Helium was used as the carrier gas and the following oven program was used: 120°C (hold 1 min.), to 300°C at 9°C/min. (hold 10 min.). The injector was set at 250°C and the ECD at 300°C.

To measure the column (i.e., "system") breakdown, an on-column injection was made. This eliminated the effects of the injection port. Using this technique, endrin breakdown was 1% and DDT breakdown was below detection limits (Figure 1).

A sample was then introduced by direct injection into an untreated glass Uniliner® sleeve. The advantage of using a Uniliner® sleeve for a direct injection is that the GC column forms a press-tight seal with the liner, allowing the vaporized sample to deposit onto the column with minimal injection port contact. Deactivation is critical for this technique because a direct injection maximizes contact of the sample vapor with the liner. The injection resulted in 62% endrin breakdown with no DDT breakdown (Figure 2).

Figure 1

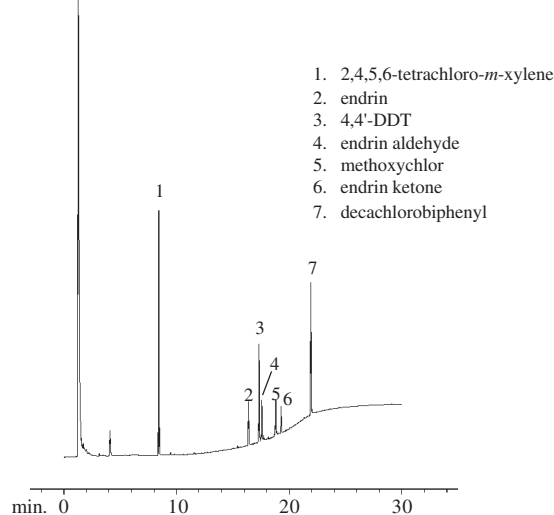
An on-column injection demonstrates system breakdown of less than 1% for endrin and DDT using a Siltek™-deactivated inlet liner.



30m, 0.53mm ID, 0.42μm Rtx®-CLPesticides2 (cat.# 11340).
Inj.: 1μL of 50pg/μL standard. **Oven temp.:** 40°C (hold 1 min.) to 300°C @ 9°C/min. (hold 10 min.); **Inj. temp.:** 250°C;
Det.: ECD, 300°C; **Carrier gas:** helium.

Figure 2

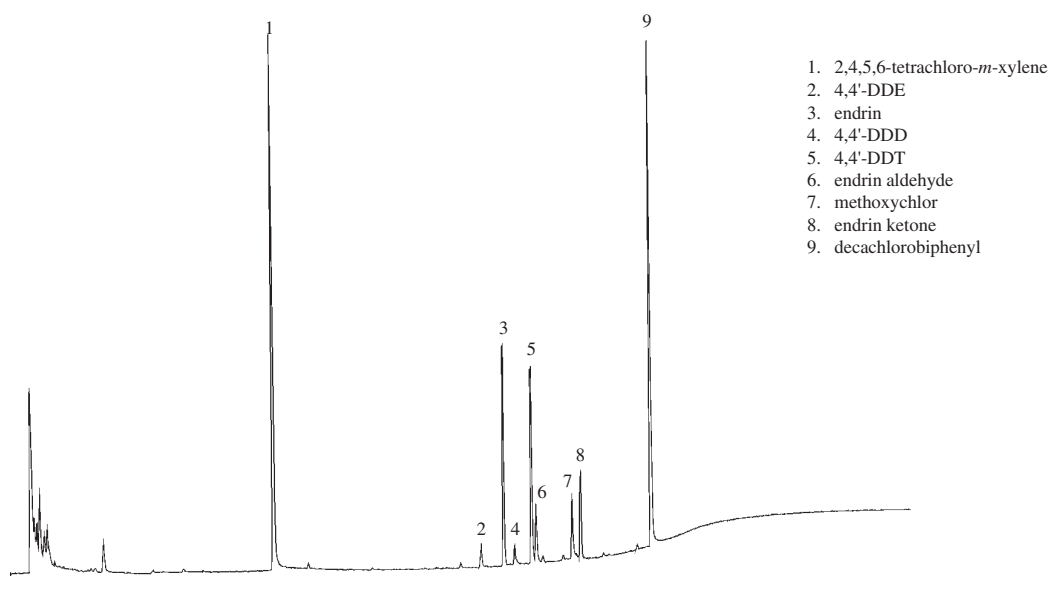
A chromatographic system that is inadequately deactivated will cause poor linearity, poor reproducibility, as demonstrated by 62% endrin breakdown.



30m, 0.53mm ID, 0.42μm Rtx®-CLPesticides2 (cat.# 11340).
Inj.: 1μL of 50pg/μL standard. **Oven temp.:** 120°C (hold 1 min.) to 300°C @ 9°C/min. (hold 10 min.); **Inj. temp.:** 250°C;
Det.: ECD, 300°C; **Carrier gas:** helium.

Figure 3

Untreated metal liners and injection port surfaces will cause high levels of endrin and DDT breakdown.



30m, 0.53mm ID, 0.42µm Rtx®-CLPesticides2 (cat.# 11340). **On-column conc.:** 50ppb; **Oven temp.:** 120°C (hold 1 min.) to 300°C @ 9°C/min. (hold 10 min.); **Inj./det. temp.:** 250°C/300°C; **Purge:** on 0.75 min.; **Carrier gas:** helium, 33cm/sec.

To compare the effects caused by hot metal surfaces, a splitless injection was made into an untreated stainless steel sleeve. Because the splitless sleeve does not form a press-tight seal with the analytical column, the sample vapor is less restricted and is free to contact the metal disk located at the bottom of the HP injection port, as well as the injection port body. Endrin breakdown (40%) was less than in the untreated glass liner, but DDT breakdown (20%) was significant (Figure 3).

As a final comparison, the standard mixture was injected via direct injection into a Uniliner® sleeve that was processed with Siltek™ deactivation (Figure 4). Endrin breakdown measured less than 1% on the Uniliner® sleeve that had been deactivated using the Siltek™ deactivation process. DDT breakdown again was below detection limits and, therefore, insignificant. A completely deactivated system is most effective for analyzing US EPA Method 8081A samples on the same HP system used for the previous injections (Figure 5).

Several conclusions can be drawn from the results of this series of injections. It appears that endrin is more prone to break down on glass surfaces than metal, and DDT is more prone to break down on metal surfaces. In addition, direct injection reduces contact between the sample and the metal injection port. And,

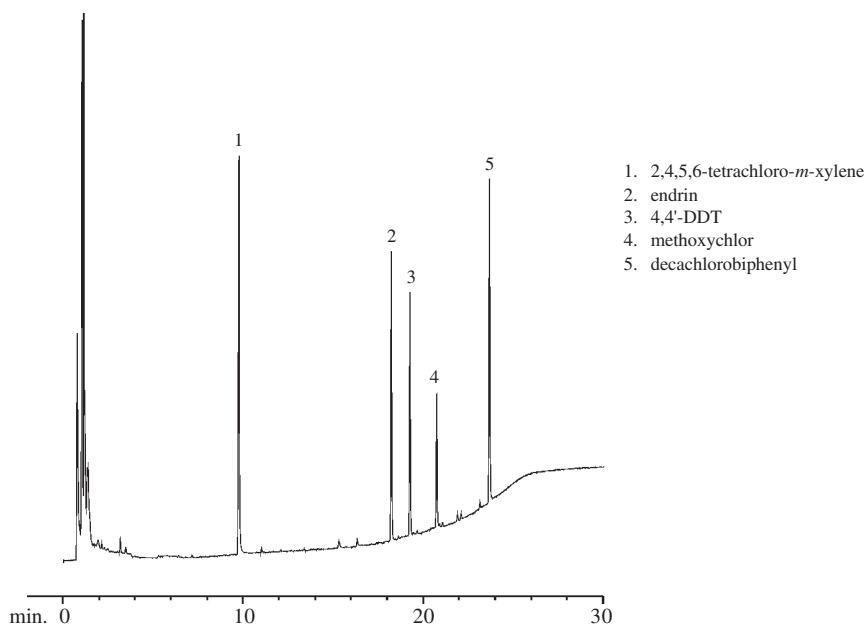
most importantly, careful deactivation of glass sleeves is crucial for minimizing endrin breakdown. Restek's Siltek™ deactivation yields a minimal endrin breakdown of 1%!

Our research demonstrates that a direct injection into a Siltek™-deactivated Uniliner® sleeve provides the best protection against problematic breakdown that occurs in the injection port when analyzing chlorinated pesticides. For a complete, highly inert pathway to analyze these compounds, Restek also offers analytical columns for chlorinated pesticide analysis (Rtx®-CLPesticides and Rtx®-CLPesticides2 columns) and Siltek™-deactivated guard columns.

**For more information on Siltek™—
the next generation of deactivation,
please request our Siltek™ Benefits
brochure (cat.# 59803).**

Figure 4

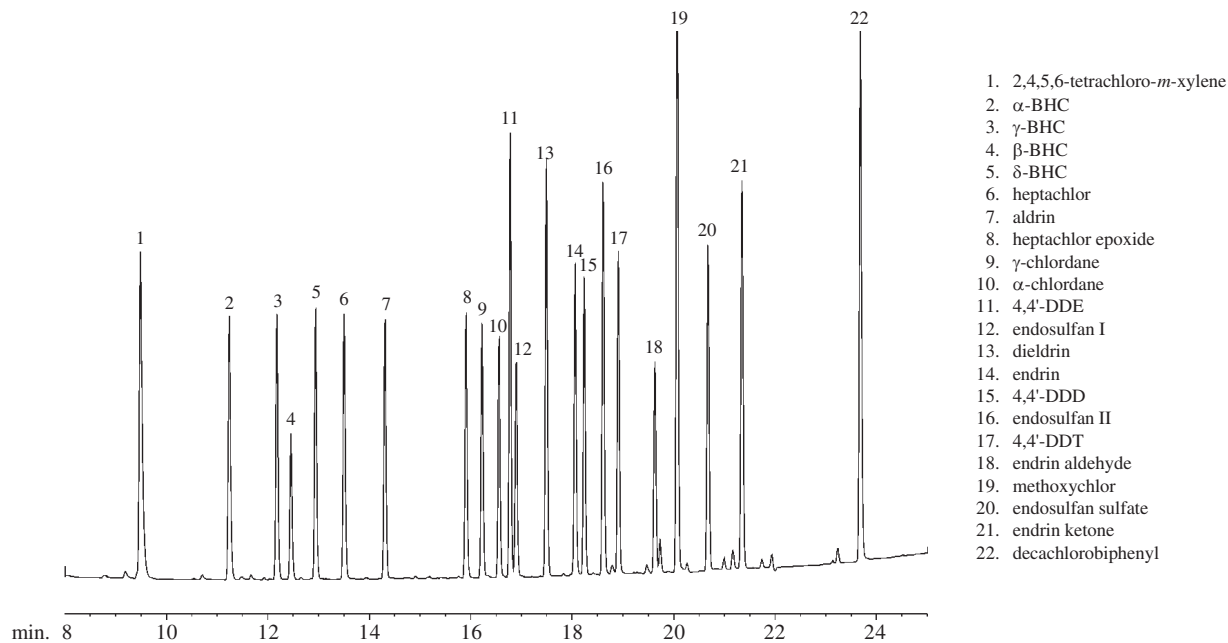
Siltek™-deactivated Uniliner® inlet liners result in less than 1% endrin breakdown and undetectable DDT breakdown.



30m, 0.53mm ID, 0.42µm Rtx®-CLPesticides2 (cat.# 11340). **Inj.:** 1µL of 50pg/µL standard. **Oven temp.:** 120°C (hold 1 min.) to 300°C @ 9°C/min. (hold 10 min.); **Inj. temp.:** 250°C; **ECD temp.:** 300°C; **Carrier gas:** helium.

Figure 5

A completely deactivated GC system shows excellent resolution of the complete list of the chlorinated pesticides in US EPA Method 8081A.



30m, 0.32mm ID, 0.5µm Rtx®-CLPesticides (cat.# 11139) with a 5m, 0.32mm ID Siltek™-deactivated guard column (cat.# 10027) and a Siltek™-deactivated gooseneck liner (cat.# 20798-214.1). **On-column conc.:** 16-160pg; **Oven temp.:** 120°C (hold 1 min.) to 300°C @ 9°C/min. (hold 10 min.); **Inj. temp.:** 250°C, splitless (hold for 0.75 min.); **ECD temp.:** 300°C with anode purge; **Carrier gas:** helium, 31cm/sec.

Product Listing

Rtx®-CLPesticides Columns

ID	df (µm)	Stable to	15m	30m
0.25mm	0.25	340°C	11120	11123
0.32mm	0.50	340°C	11136	11139
0.53mm	0.50	340°C	11137	11140
ID	df (µm)	Stable to	10m	20m
0.18mm	0.18	340°C	42101	42102

Rtx®-CLPesticides Column Kits

These kits include both a CLPesticides and CLPesticides2 column, a Universal Angled 'Y' Press-Tight® Connector, and a 5m guard column. (Note: Columns are not preconnected in these kits.)

Description	cat.#
0.53mm ID Rtx®-CLPesticides Kit	11197
0.32mm ID Rtx®-CLPesticides Kit	11198
0.25mm ID Rtx®-CLPesticides Kit	11199

Organochlorine Pesticide Mix AB #2

aldrin	8µg/mL	dieldrin	16µg/mL
α-BHC	8	endosulfan I	8
β-BHC	8	endosulfan II	16
δ-BHC	8	endosulfan sulfate	16
γ-BHC (lindane)	8	endrin	16
α-chlordane	8	endrin aldehyde	16
γ-chlordane	8	endrin ketone	16
4,4'-DDD	16	heptachlor	8
4,4'-DDE	16	heptachlor epoxide (B)	8
4,4'-DDT	16	methoxychlor	80

in hexane/toluene (1:1), 1mL/ampul.

	each	5-pack	10-pack
	32292	32292-510	
w/data pack	32292-500	32292-520	32392

Organochlorine Pesticide Mix C #2

chlorobenzilate	32µg/mL
diallate (cis & trans)	80
1,2-dibromo-3-chloropropane	8
hexachlorobenzene	8
hexachlorocyclopentadiene	8
isodrin	8

in hexane/toluene (1:1), 1mL/ampul.

	each	5-pack	10-pack
	32295	32295-510	
w/data pack	32295-500	32295-520	32395

Restek Trademarks: Press-Tight, Rtx, Siltek, Uniliner.

Rtx®-CLPesticides2 Columns

ID	df (µm)	Stable to	15m	30m
0.25mm	0.20	340°C	11320	11323
0.32mm	0.25	340°C	11321	11324
0.53mm	0.42	340°C	11337	11340
ID	df (µm)	Stable to	10m	20m
0.18mm	0.14	340°C	42301	42302

Siltek™-Deactivated Guard Columns

nominal ID	nominal OD	5-meter	10-meter
0.25mm	0.37 ± 0.04mm	10026	10036
0.32mm	0.45 ± 0.04mm	10027	10037
0.53mm	0.69 ± 0.04mm	10028	10038

Siltek™-Deactivated Press-Tight® Connectors

type	qty.	cat.#
straight	25-pk.	20449
angled 'Y'	3-pk.	20469

For other Siltek™-deactivated Press-Tight® connectors, add suffix "-266" to the catalog number.

Siltek™-Deactivated Inlet Liners

Siltek™	Siltek™ with Siltek™-deact. wool	Siltek™ with CarboFrit™	qty.
-214.1	-213.1	-216.1	each
-214.5	-213.5	-216.5	5-pk.
-214.25	-213.25	-216.25	25-pk.

For Siltek™-deactivated liners, add the corresponding suffix number to the liner's catalog number.

Siltek™-Deactivated Borosilicate Wool

qty.	cat.#
10 grams	21100

For more information on Siltek™—
the next generation of deactivation,
please request our Siltek™ Benefits
brochure (cat.# 59803).

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Products '08
Australian Distributors **ECH**nology
www.chromtech.net.au E-mail : info@chromtech.net.au Tel : +61 3 9762 2034 Fax : +61 3 9761 1169

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