

Siltek™ Deactivation Delivers Inertness to Analyte Breakdown and Reactivity, and Durability to Physical and Chemical Challenges

A common concern in gas chromatographic (GC) analyses is the interaction of analytes with active surfaces in the GC pathway. The injection port is the first source of active sites, often leading to adsorption and breakdown of analytes. However, not all analyses are affected by reactivity within the injection port. Hydrocarbons, typically, are not susceptible to adsorption or breakdown. In contrast, active compounds such as pesticides, drugs, phenols, amines, and alcohols, which are often injected via splitless mode, are more prone to these problems. With a splitless injection, carrier gas flow rate through the liner is very slow, increasing the sample residence time in the injector and the chance for reactivity. Complete and effective liner deactivation is crucial to minimize available active sites and ensure repeatable results.

Restek has designed Siltek® deactivation to deliver both enhanced inertness and durability. Gas chromatography accessories coated with Siltek® deactivation provide durability for matrices of extreme pH or high-temperature applications.

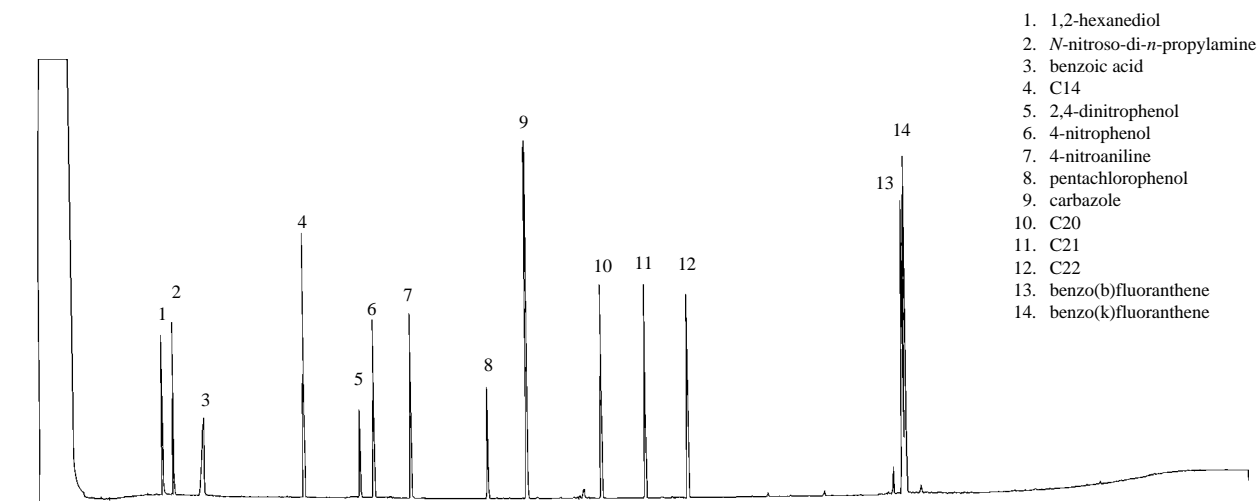
Inertness

Semivolatile analysis places extreme demands on the GC system. One key to successfully analyzing semivolatiles is having the capability to handle basic and acidic compounds in the GC system. The analytical column must provide selectivity for both classes without resulting in poor peak shape. Additionally, liner deactivation is critical to analytical success because the vaporized sample comes in contact with the inlet liner first.

The Restek XTI test mix was chosen to evaluate the inertness of a Siltek™-deactivated liner. This mix contains both acidic and basic probes, some of which are pollutants monitored in US Environmental Protection Agency (EPA) Method 8270 (4-nitroaniline, *N*-nitroso-di-*n*-propylamine, 2,4-dinitrophenol, pentachlorophenol, benzoic acid, benzo(b)- and benzo(k)fluoranthene). A splitless injection of the XTI mix with an on-column concentration of 4-10ng shows an excellent response for all of the probes, including the active compounds dinitrophenol, 1,2-hexanediol, and benzoic acid (Figure 1).

Figure 1

Siltek™-deactivated liner shows excellent inertness for acidic and basic probes.



1. 1,2-hexanediol
2. *N*-nitroso-di-*n*-propylamine
3. benzoic acid
4. C14
5. 2,4-dinitrophenol
6. 4-nitrophenol
7. 4-nitroaniline
8. pentachlorophenol
9. carbazole
10. C20
11. C21
12. C22
13. benzo(b)fluoranthene
14. benzo(k)fluoranthene

30m, 0.25mm ID, 0.25µm XTI®-5 (cat.# 12223) with a Siltek™-deactivated 4mm splitless single gooseneck sleeve (cat.# 20798-214.1). **Oven temp.:** 40°C (hold 2 min.) to 100°C @ 30°C/min., to 180°C @ 9°C/min., to 330°C @ 30°C/min. (hold 10 min.); **Inj. temp.:** 250°C; **Det.:** 330°C; **Carrier gas:** He.

Thermal Stability

To test the durability of the Siltek® liner deactivation, two sources of stress were investigated—high inlet temperature over a period of 10 days and repeated exposure to aqueous injections of low, then high pH. High inlet temperatures can promote degradation of the deactivation layer by causing it to bake or bleed off of the liner. In the first study, a baseline splitless XTI injection was performed, and response factors (relative to C14) were calculated. The injection port was then set at 330°C overnight and another XTI injection was made. This process was continued for 10 days. After 10 days at 330°C, the Siltek® deactivation retained its integrity, achieving essentially unchanged response factors, even for the critical probes (Figure 2).

Resistance to Chemical Attack

For the next durability study, a Siltek®-deactivated liner was repeatedly exposed to aqueous HCl injections, pH 1.4. The ability to withstand low pH aqueous samples is important with environmental applications that require acidification of the matrix. Very low or very high pH samples can cause pinpoint holes in the deactivation layer that will eventually undercut the layer and strip it away. For this study, a baseline XTI injection was made via direct injection and relative response factors were calculated. In the direct injection mode, a leak-free connection is formed in the liner, minimizing sample exposure within the injection port. Ten microliters of the pH 1.4 sample were injected, followed by a direct injection of the XTI test mixture.

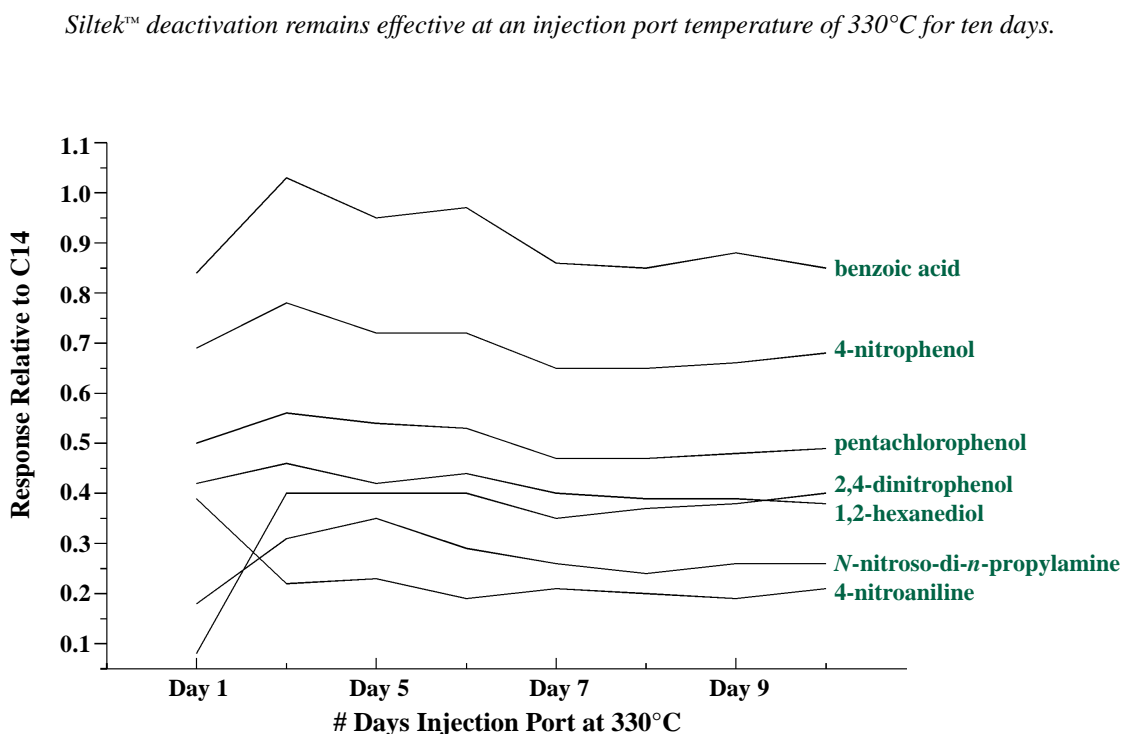
This cycle continued until a total of 180µL were injected (Figure 3). Key probes, such as 2,4-dinitrophenol (DNP), pentachlorophenol (PCP), *N*-nitroso-di-*n*-propylamine (*n*-propylamine), and 1,2-hexanediol (diol) retained their responses up to at least 120µL injected.

The experiment was repeated with an identical set-up using aqueous NH₄OH injections, pH 10.1 (Figure 4). Under these demanding conditions, the response for the XTI compounds was consistent for 70 injections. As expected, the response of the acidic compounds began to decrease with repeated injections but many compounds continued to have excellent response for more than 120µL injections.

Siltek® deactivation offers both inertness and resistance to temperature and pH extremes within a GC system. It is available as a deactivation for fused silica guard columns and inlet liners.

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

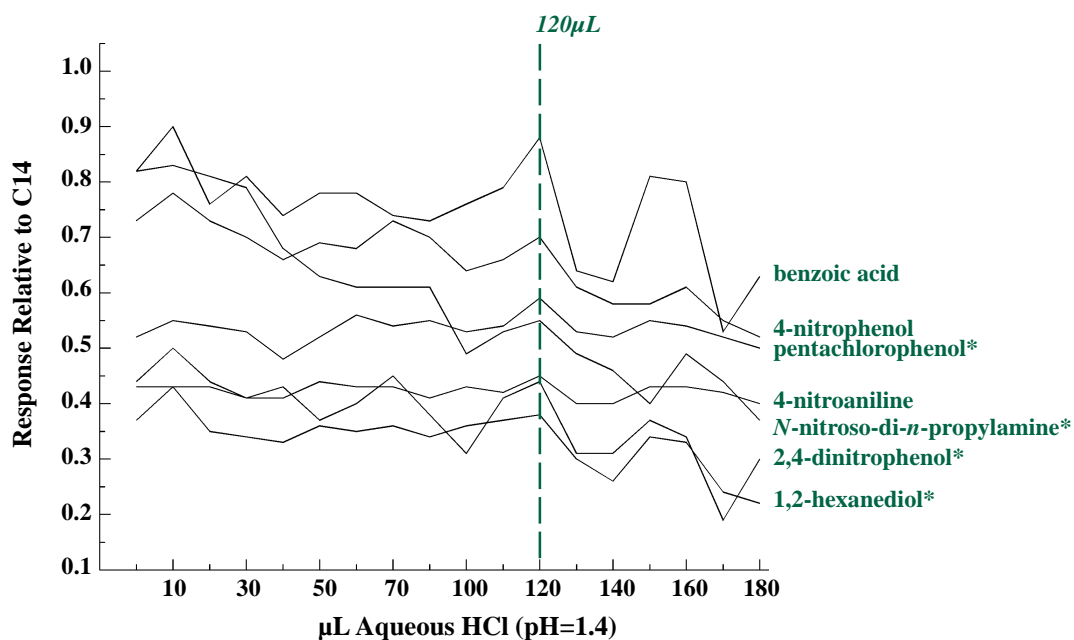
Figure 2



30m, 0.25mm ID, 0.25µm XTI®-5 (cat.# 12223) with a Siltek™-deactivated splitless sleeve (cat.# 20798-214.1). **Oven temp.:** 40°C (hold 2 min.) to 190°C @ 6°C/min., to 330°C @ 30°C/min. (hold 10 min.); **Inj. temp.:** 330°C; **Det.:** 330°C; **Carrier gas:** He.

Figure 3

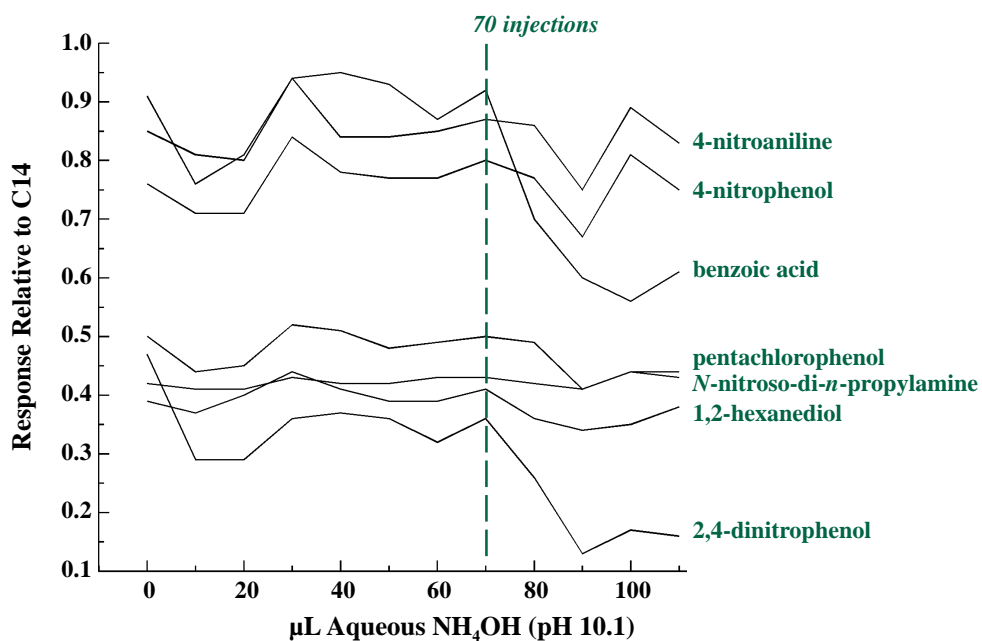
Difficult probes* retain their response on Siltek™ deactivation up to 120µL of an aqueous solution of pH 1.4.



30m, 0.25mm ID, 0.25µm XTI®-5 (cat.# 12223) with a Siltek™-deactivated 4mm open-top Uniliner® w/o wool (cat.# 20843-214.1).
Oven temp.: 40°C (hold 2 min.) to 190°C @ 6°C/min., to 330°C @ 30°C/min. (hold 10 min.); Inj. temp.: 250°C; Det.: 330°C; Carrier gas: He.

Figure 4

Response for XTI compounds on Siltek™ deactivation was consistent for 70 injections of an aqueous solution at pH 10.1.



30m, 0.25mm ID, 0.25µm XTI®-5 (cat.# 12223) with a Siltek™-deactivated 4mm open-top Uniliner® w/o wool (cat.# 20843.214.1).
Oven temp.: 40°C (hold 2 min.) to 190°C @ 6°C/min., to 330°C @ 30°C/min. (hold 10 min.); Inj. temp.: 250°C; Det.: 330°C; Carrier gas: He.

Product Listing

XTI®-5 Columns

ID	df (µm)	Temp. limits	15m	30m
0.25mm	0.25	-60 to 360°C	12220	12223
	0.50	-60 to 330/350°C	12235	12238
	1.00	-60 to 325/350°C	12250	12253
0.32mm	0.25	-60 to 360°C	12221	12224
	0.50	-60 to 330/350°C	12236	12239
	1.00	-60 to 325/350°C	12251	12254
0.53mm	0.50	-60 to 330/360°C	12237	12240
	1.00	-60 to 325/350°C	12252	12255
	1.50	-60 to 310/330°C	12267	12270

Inlet Liners for HP GCs

Liner type	ID/OD/Length (mm)	ea.	5-pk.	25-pk.
4mm split w/wool	4.0/6.3/78.5	20781	20782	20783
2mm splitless	2.0/6.5/78.5	20712	20713	20714
4mm splitless	4.0/6.5/78.5	20772	20773	20774
4mm gooseneck	4.0/6.5/78.5	20798	20799	20800
4mm double gooseneck	4.0/6.5/78.5	20784	20785	20786
Cyclo splitter®	4.0/6.3/78.5	20706	20707	20708

Inlet Liners for Varian GCs

Liner type	ID/OD/Length (mm)	ea.	5-pk.	25-pk.
2mm splitless	2.0/6.3/74	20721	20722	20723
4mm splitless	4.0/6.3/74	20904	20905	20906
0.5mm SPI	0.53/4.6/54	20775	20776	20777
0.8mm SPI	0.80/4.6/54	20778	20779	20780
SPI with buffer	2.4/4.6/54	20850	20851	20852

Siltek™-Deactivated Guard Columns

Nominal ID	Nominal OD	5m	10m
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 Glass 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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