

Tech-Tips

Improved GC Analysis of Basic Organic Compounds Using Base-Deactivated Columns and Inlet Liners

The gas chromatographic (GC) analysis of basic organic compounds (e.g., amines, basic drugs, and azo dyes) is a challenge to the analyst, especially when the compounds are at low concentrations. The challenge is mainly caused by acidic silanol groups on the pathway surfaces of GC system (i.e., inlet liner, wool, guard and analytical columns) that remain after standard chemical deactivation. Vaporized basic compounds are adsorbed onto the active sites, causing peak tailing and reduced response.

Because of the interaction between basic compounds in the sample with acidic active sites in the sample pathway, a “priming” effect may be noticed. With priming, the first injection of a basic sample results in compounds adsorbing onto the active sites and covering them. Repeated injections cause more and more sites to be covered until an equilibrium is reached. Response subsequently improves, but reproducibility is poor. The priming effect is temporary because the system will revert back to an adsorptive state after column conditioning or periods of inactivity.

Restek has an entire line of base-deactivated products to aid in the analysis of basic compounds—inlet liners, fused silica wool, guard columns, and the Rtx®-5 Amine analytical column. (Restek’s analytical column deactivated specifically for the analysis of organic basic compounds). Base-deactivation is a unique deactivation process that bonds basic functional groups to

the analytical surface. These functional groups on the surface greatly reduce adsorption and priming effects, thereby improving peak shape, response, and reproducibility. Figure 1 compares the effect of a base-deactivated liner/wool system with a standard-deactivated liner/wool system on diethanolamine response. In

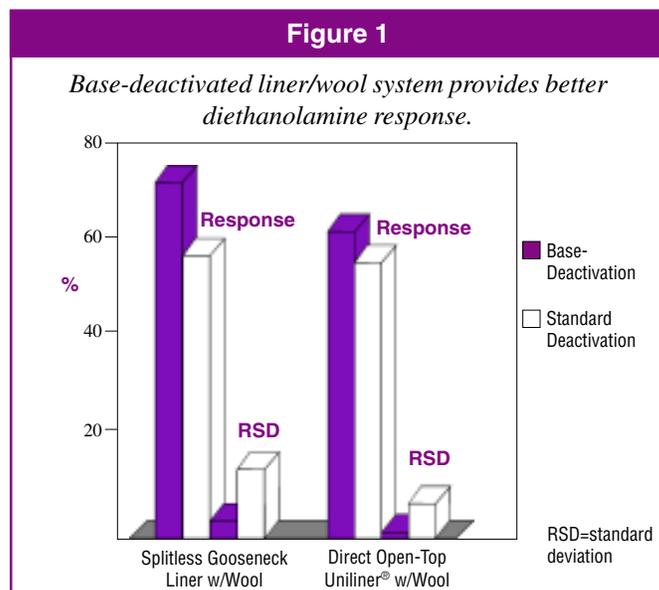
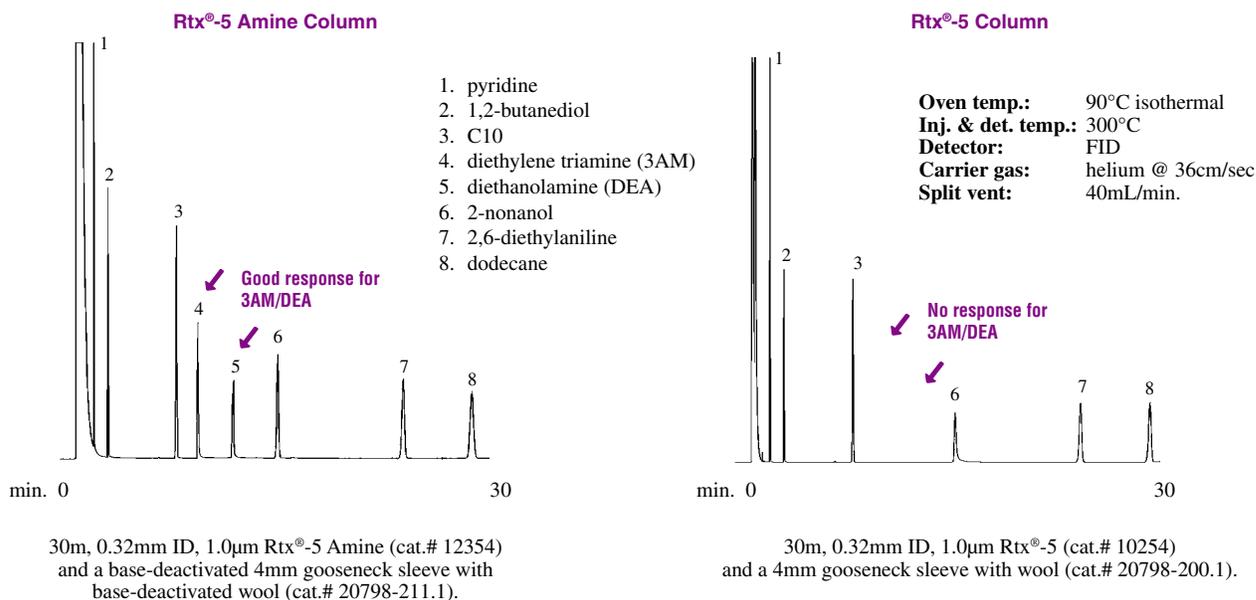


Figure 2

The Rtx®-5 Amine column achieves better response for active basic compounds compared to the standard 5% phenyl column.



each case, response and reproducibility improved using the base-deactivation as opposed to standard-deactivation. Using the base-deactivated wool improves response of basic test probes by preventing the vaporized sample from contacting the highly-adsorptive hot metal inlet disc at the bottom of the injection port, and by providing a high-surface area, non-adsorptive surface on which to vaporize the sample.

The inertness of the Rtx[®]-5 Amine column is obvious when compared to a standard 5% phenyl analytical column (Figure 2). Two of the active basic compounds—diethylene triamine and triethanolamine—are completely adsorbed on the standard 5% phenyl column, yet exhibit good peak shape and response on the

Rtx[®]-5 Amine column. To ensure the integrity of the Rtx[®]-5 Amine columns, Restek uses a demanding test mixture containing amines and alcohol-amines designed to confirm column inertness to basic probes.

Restek's base-deactivation process is effective for all but highly acidic compounds, and is specially-designed to ensure a highly inert GC pathway for basic compounds, from inlet to analytical column. This deactivation provides a surface that is chemically compatible with basic compounds, thereby greatly reducing priming, peak tailing, and poor reproducibility associated with basic compound analysis on standard-deactivated surfaces.

Product Listing

Rtx[®]-5 Amine Columns

ID	df (µm)	Stable to	15m	30m
0.25mm	0.50	340°C	12335	12338
	1.00	340°C	12350	12353
0.32mm	1.00	340°C	12351	12354
	1.50	340°C	12366	12369
0.53mm	1.00	340°C	12352	12355
	3.00	340°C	12382	12385

Base-Deactivated Fused Silica Guard Columns

Nominal ID	Nominal OD	cat.#
0.25mm	0.37 ± 0.04mm	10000
0.32mm	0.45 ± 0.04mm	10001
0.53mm	0.69 ± 0.05mm	10002

Amine Column Test Mix

1,2-butanediol	0.60mg/mL
pyridine	0.60
decane	0.60
diethylenetriamine	1.20
diethanolamine	1.20
2-nonanol	0.60
2,6-dimethylaniline	0.60
dodecane	0.60

In CH₂Cl₂/methanol (50:50), 1mL/ampul. cat.# 35002, each

Base-Deactivated Inlet Liners for HP GCs

Description	each	5-pk.	25-pk.
4mm Split w/wool	20781-211.1	20782-211.5	20783-211.25
Cyclosplitter [®]	20706-210.1	20707-210.5	—
4mm Splitless Straight	20772-210.1	20773-210.5	—
2mm Gooseneck	20795-210.1	20796-210.5	20797-210.25
4mm Gooseneck	20798-210.1	20799-210.5	20800-210.25

Base-Deactivated Inlet Liners for Varian GCs

Description	each	5-pk.	25-pk.
Splitter with wool	20792-211.1	20793-211.5	—
Frit Splitter	20715-210.1	20716-210.5	—

Restek stocks the most requested liners with base-deactivation for immediate delivery. However, if you do not see the liner you need, orders can be placed on a custom basis for base-deactivated liners by adding the appropriate suffix number: each (-210.1), 5-packs (-210.5), and 25-packs (-210.25). For base-deactivated liners packed with base-deactivated wool: each (-211.1), 5-packs (-211.5), and 25-packs (-211.25).

Base-Deactivated Fused Silica Wool

Quantity	cat.#
10 grams	20999

Restek offers additional deactivation procedures—call Technical Service for details.

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