Reduce Downtime and Cost of Materials with Rugged Rxi®-5Sil MS GC Columns

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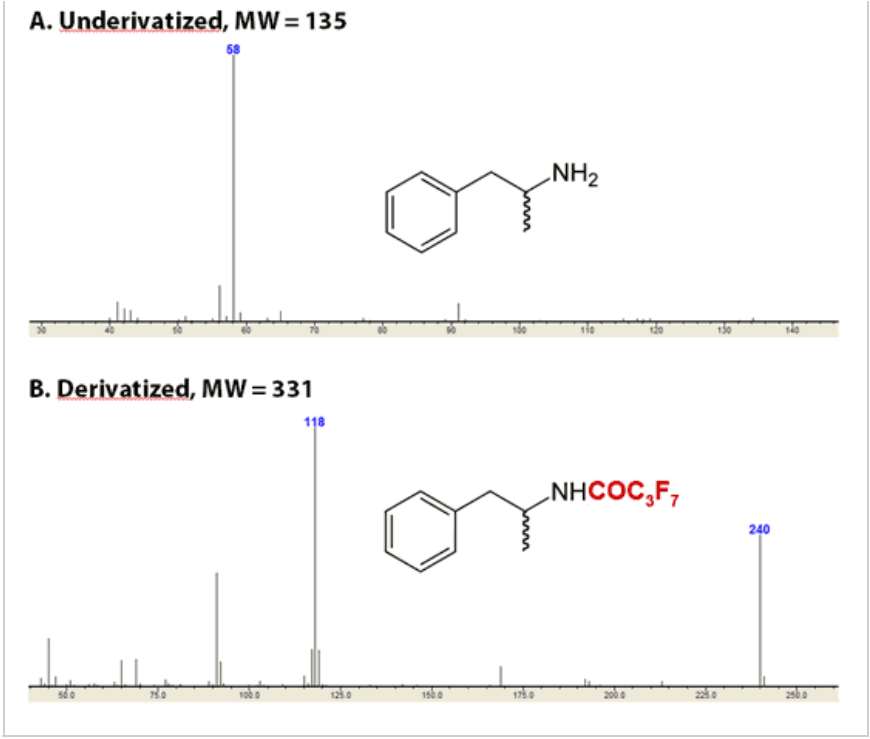
- Save costs with long column lifetime.
- Reduce downtime from column trimming and replacement.
- Improve peak shape for active compounds.

Rugged Rxi®-5Sil MS columns produce consistent results, even under harsh conditions.

When performing GC/MS analysis of drugs, many chemists choose to derivatize samples prior to analysis. Derivatization not only increases the volatility of some drug compounds, but it also reduces activity, resulting in improved peak shape and more accurate quantification. An additional advantage is that derivatized compounds have a higher molecular weight, thus producing more reliable mass spectra than underivatized compounds. Despite these benefits, derivatization reagents are often harsh and can damage analytical columns, leading to high bleed, significant reduction in retention times, and increased tailing for active compounds. Often, this damage is concentrated near the head of the column, so trimming a short length can improve results. However, trimming is a finite solution as repeated clipping ultimately results in decreased efficiency and shorter column lifetimes. Choosing a more rugged column, such as the Rxi®-5Sil MS column, is a better alternative. The Rxi®-5Sil MS column is extremely stable and holds up to harsh treatment, including repeated exposure to derivatization reagents.

The analysis of amphetamine illustrates the ruggedness of the arylene-based Rxi®-5Sil MS polymer. Amphetamine is typically derivatized, because the underivatized form is an active basic compound that produces only a few low molecular weight ions for monitoring. In contrast, upon derivatization, activity decreases, resulting in dramatically improved peak shape and more accurate quantitation. Additionally, several higher molecular weight ions are produced, which can be monitored for definitive identification (Figure 1).

Figure 1 Derivatizing amphetamine results in more definitive identification by creating higher molecular weight ions.



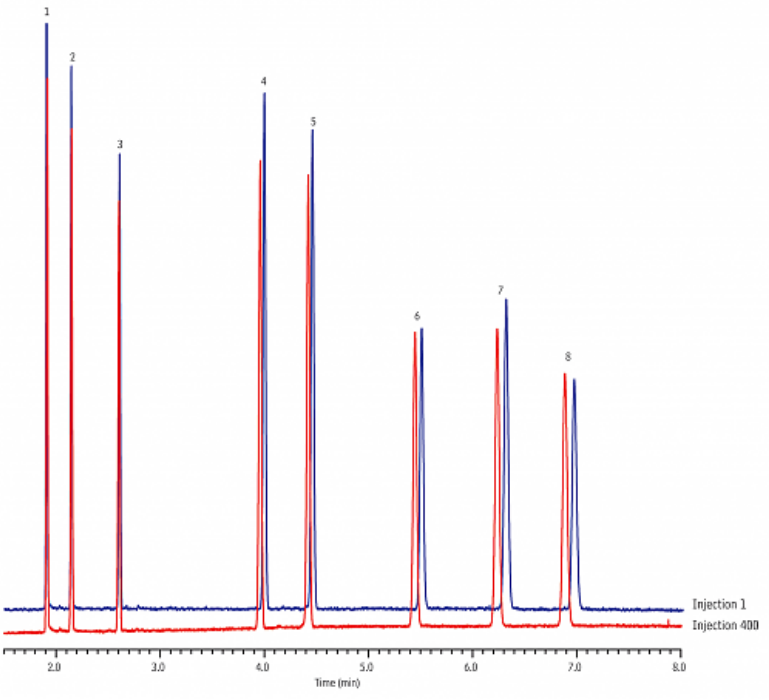
Phase Stability Extends Column Lifetime

In order to demonstrate the ruggedness of the Rxi®-5Sil MS column, 400 injections of heptafluorobutyric acid anhydride (HFBA) in butyl chloride were performed. HFBA is a very harsh derivatization reagent, and the concentration of reagent in the solvent was equivalent to that of a derivatized sample. Throughout the course of 400 injections, bleed, retention, and peak shape for active compounds was monitored by periodically injecting a column test mix containing active compounds (1,6-hexanediol, 4-chlorophenol, and dicyclohexylamine). Chromatographic results were remarkably consistent, even after 400 injections (Figure 2). Column bleed was monitored over the course of the experiment and remained below 5pA (Figure 3). The consistency of retention time data and low bleed levels demonstrate phase stability, which results in longer column lifetimes and reduced maintenance and replacement costs.

Figure 2 Rugged Rxi®-5Sil MS columns produce consistent retention times, even after 400 injections of derivatization reagent.

Peaks

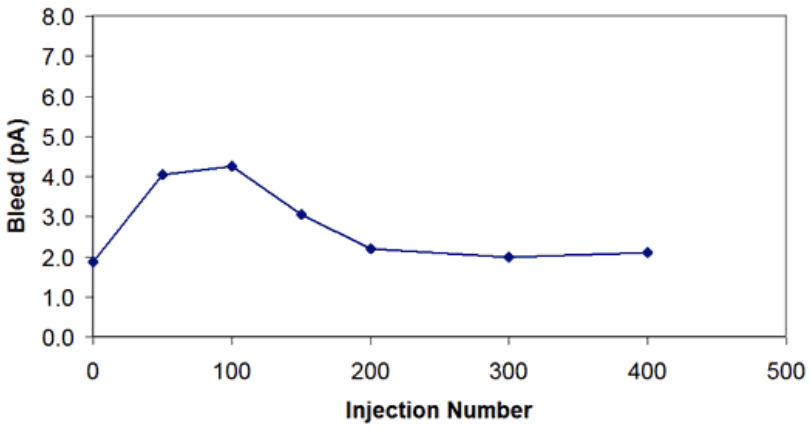
1. 2-Ethylhexanoic acid
2. 1,6-Hexanediol
3. 4-Chlorophenol
4. Tridecane
5. 1-Methylnaphthalene
6. 1-Undecanol
7. Tetradecane
8. Dicyclohexylamine



GC_CF01131

Column	Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623)
Sample	Rxi®-5Sil MS/XLB column test mix (cat.# 35226)
Injection	
Inj. Vol.:	1.0 µL split (split ratio 60:1)
Liner:	4 mm recessed single taper (cat.# 20983)
Inj. Temp.:	250 °C
Oven	
Oven Temp.:	125 °C
Carrier Gas	He, constant pressure
Linear Velocity:	36 cm/sec @ 125 °C
Detector	FID @ 320 °C
Instrument	Agilent/HP6890 GC

Figure 3 Low column bleed results in long column lifetimes, saving labs replacement costs.



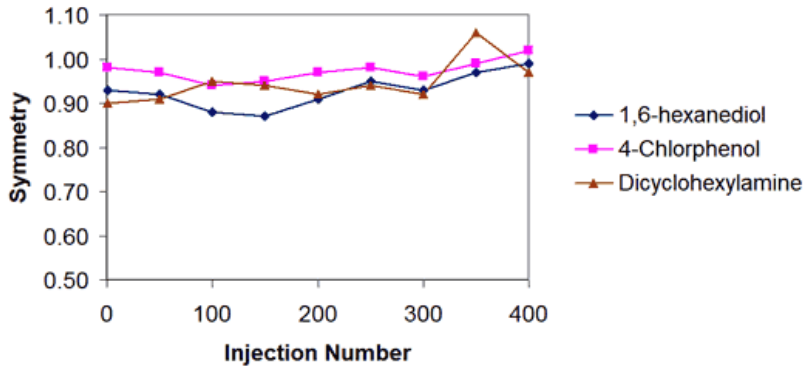
Column bleed over 400 injections of HFBA derivatization reagent.

Symmetric Peaks for More Accurate Results

Peak shape was also monitored to ensure column inertness was stable over time—an important factor in maintaining accuracy. Peaks for the active test probes were symmetric even after 400 injections, allowing easy identification and consistent integration (Figure 4). In a second experiment to complement the test probe results, underivatized amphetamine was injected onto a new Rxi®-5Sil MS column, an Rxi®-5Sil MS column after 400 injections of derivatization reagent, and a new competitor column of equivalent phase chemistry. Even though underivatized amphetamine is highly active, peak symmetry on the Rxi®-5Sil MS column was consiste

Additionally, peak shape on both the exposed and unexposed Rxi®-5Sil MS column was better than that on the new competitor column (Figure 5).

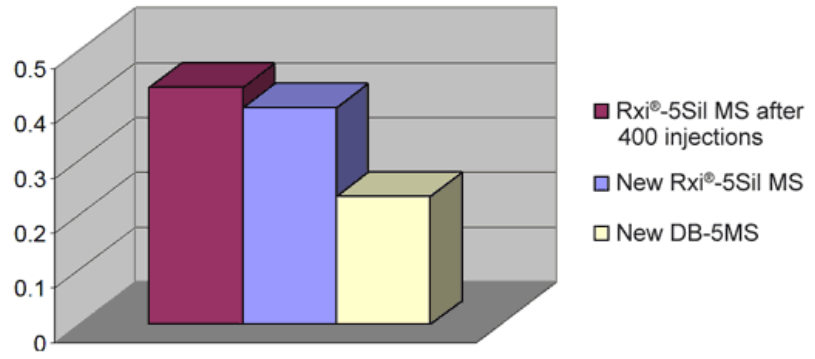
Figure 4 Active probes show consistent, symmetric peak shape, demonstrating the inertness needed for accurate quantification.



Test probe symmetry over 400 injections of HFBA derivatization reagent.

Symmetry values <1 indicate peak tailing and values >1 indicate fronting.

Figure 5 Peak symmetry for underivatized amphetamine is significantly better on an Rxi®-5Sil MS than on a competitor column, even after 400 injections of HFBA derivatization reagent.



Symmetry values <1 indicate peak tailing and values >1 indicate fronting.

The rugged arylene phase of the Rxi®-5Sil MS column results in highly stable performance, even under the most demanding of analytical conditions, and its exceptional inertness ensures good peak shape for reproducible quantitation. The stability of the Rxi®-5Sil MS column results in longer column lifetimes, reducing both downtime and replacement costs.

RELATED SEARCHES

amphetamine, *derivitization*, *derivitize*, *HFBA*, *heptafluorobutyric acid anhydride*, *Rxi-5Sil MS*