Rxi GCColumns

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Highest Inertness • Lowest Bleed • Exceptional Reproducibility

Rxi®-5Sil MS

Assured Performance
for Forensic Applications

- Exceptional column inertness means greater certainty and lower detection limits.
- Versatile selectivity lets you keep analyzing samples instead of changing columns between methods.
- Robust, low-bleed phase results in better sensitivity and longer column lifetime.

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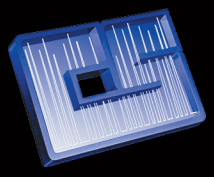

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Rxi®-5Sil MS Columns…

Whether analyzing postmortem samples or supporting athletic or workplace drug testing, toxicology labs are challenged with producing critical evidence that stands up under scrutiny. Increased pressure for fast, definitive results is driving labs to investigate standardized procedures and certifications aimed at reducing variability. GC column choice plays a vital role in data quality and using rugged, versatile Rxi®-5Sil MS capillary columns is an easy way to improve chromatography performance and simplify lab operations.

For years, "5" type (5% diphenyl/95% dimethyl polysiloxane) columns have been recognized as the column of choice for analyzing drugs of abuse, because they offer higher selectivity and retention for functionalized compounds than "1" type columns (100% dimethyl polysiloxane). While the selectivity of 5 type columns has many forensic applications, column performance can vary significantly among these columns. Some 5 type columns have inadequate deactivations, causing tailing peaks, or are poorly stabilized, resulting in high bleed levels, reduced sensitivity, and shorter column lifetimes. Rxi®-5Sil MS columns are based on a silarylene phase (Figure 1) that offers improved inertness and stability compared to typical 5 type columns.

Toxicology labs interested in improved data quality can increase confidence in results and reduce downtime by using Rxi®-5Sil MS columns. Exceptional inertness increases accuracy and precision at trace levels, while ruggedness assures low bleed and long column lifetime. As shown on the following pages, these versatile columns can improve lab efficiency and data quality for many different drugs of abuse, including cannabinoids, benzodiazepines, cocaine, opiates, and amphetamines.

Figure 1: Rxi®-5Sil MS columns: phase structure results in a more inert, low-bleed column with broad selectivity for a wide range of compounds.

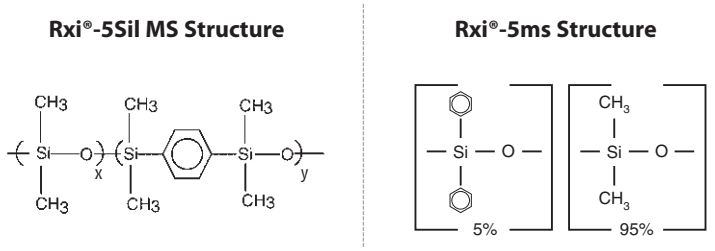
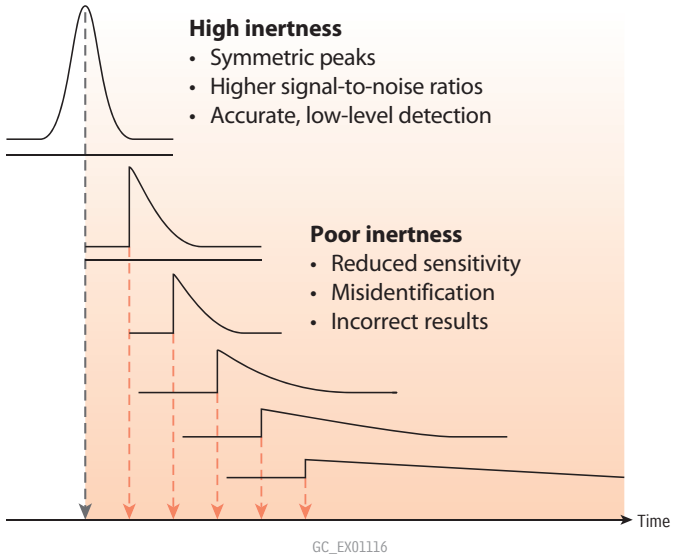


Figure 2: As column activity increases, signal decreases and retention time shifts.

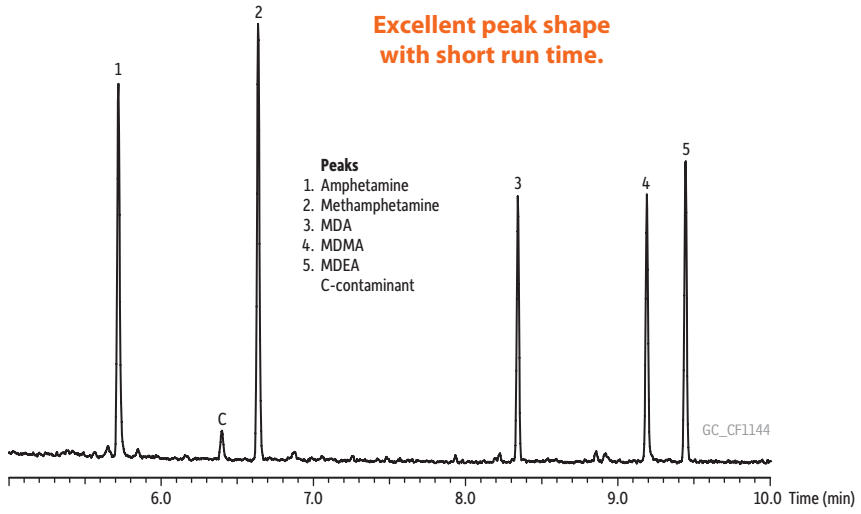


Exceptional Inertness Means Greater Certainty and Lower Detection Limits

Column inertness improves peak shape, which greatly affects the signal-to-noise ratio and, therefore, analytical sensitivity. Rxi®-5Sil MS columns are exceptionally inert, ensuring symmetric peak shape and high response for a wide range of analyte chemistries. In addition to influencing signal-to-noise ratios, column inertness also affects retention time stability, which is an important factor for correct peak identification. Inertness is critical because peak tailing will increase as column activity increases, causing retention times to shift (Figure 2). Analyzing derivatized amphetamines or cocaine and its metabolites on highly inert Rxi®-5Sil MS columns results in symmetric peak shapes and excellent low-level response (Figures 3 and 4).

Assured Performance for Forensic Applications

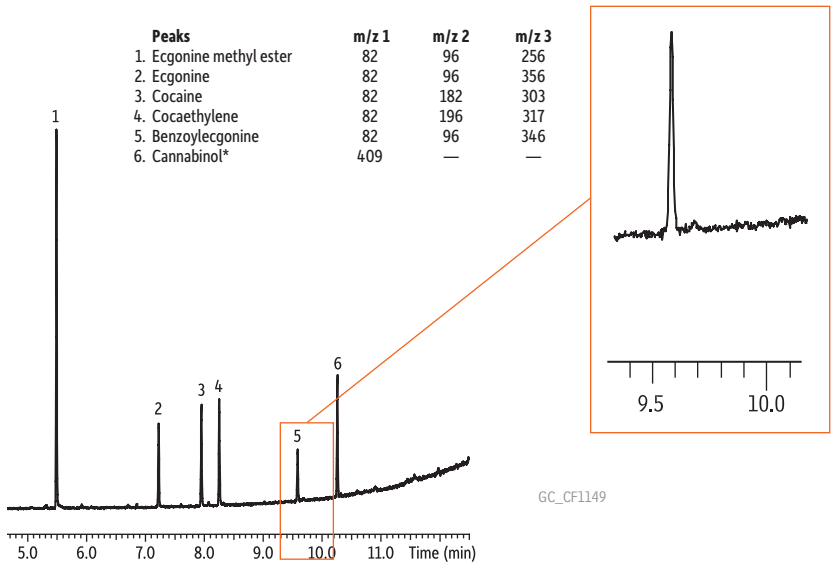
Figure 3: Robust, inert Rxi®-5Sil MS columns do not break down under harsh conditions, such as exposure to the derivatization reagents used in amphetamines analysis. Compounds shown are HFAA derivatives.



Column: Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623); **Sample:** 500 ng/mL HFAA derivatives in butyl chloride; **Injection:** Inj. Vol.: 1 µL splitless (hold 1 min); Liner: 3.5 mm splitless taper w/wool (cat.# 22286-200.1); Inj. Temp.: 250 °C; Purge Flow: 28 mL/min; **Oven:** Oven Temp: 75 °C to 300 °C at 15 °C/min; Carrier Gas: He, constant linear velocity, 45 cm/sec, 13.5 psi, 93.1 kPa @ 75 °C; **Detector:** MS, Scan; Transfer Line Temp.: 250 °C; Analyzer Type: Quadrupole; Source Temp.: 200 °C; Electron Energy: 70 eV; Solvent Delay Time: 4 min; Tune Type: PFTBA; Ionization Mode: EI; Scan Range: 40-300 amu; Scan Rate: 5 scans/sec; **Instrument:** Shimadzu 2010 GC & QP2010+ MS.

Figure 4: Low levels of derivatized cocaine and its metabolites can also be reliably separated on Rxi®-5Sil MS columns.

Peaks	m/z 1	m/z 2	m/z 3
1. Ecgonine methyl ester	82	96	256
2. Ecgonine	82	96	356
3. Cocaine	82	182	303
4. Cocaethylene	82	196	317
5. Benzoylecgonine	82	96	346
6. Cannabinol*	409	—	—



Column: Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623); **Sample:** 100 ng/mL in butyl chloride; **Injection:** Inj. Vol.: 1 µL splitless (hold 1 min); Liner: single taper w/wool (cat.# 22286-200.1); Inj. Temp.: 250 °C; Purge Flow: 20 mL/min; **Oven:** Oven Temp: 100 °C to 200 °C at 30 °C/min to 300 °C at 15 °C/min; Carrier Gas: He, constant linear velocity, 40 cm/sec, 12.5 psi, 86.2 kPa @ 100 °C; **Detector:** MS, SIM; Transfer Line Temp.: 310 °C; Source Temp.: 250 °C; Solvent Delay Time: 4 min.; Tune Type: PFTBA; Ionization Mode: EI; **Instrument:** Shimadzu 2010 GC & QP2010+ MS; **Notes:** Samples were prepared as follows: Standards brought to dryness under nitrogen, then 50 µL, BSTFA + 1%TMCS (cat.# 35606) added. 50 µL pyridine was then added, and samples were incubated at 70 °C for 30 min. After incubation, samples were diluted with butyl chloride.

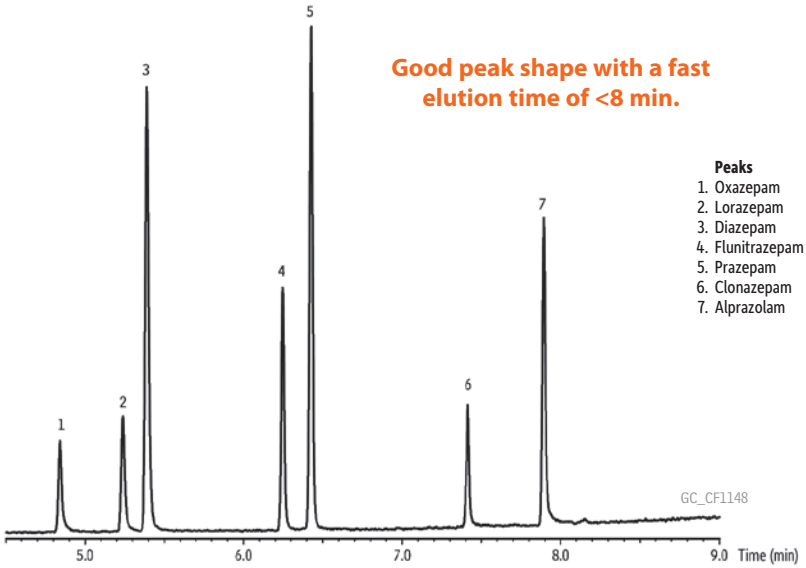
* Used as derivitazation check

Optimized Selectivity Lets You Keep Analyzing Samples Instead of Changing Columns Between Methods

While the inertness of Rxi®-5Sil MS columns exceeds typical 5 type columns, the selectivity is similar and is ideal for many toxicological applications. A wide range of analyte classes can be reliably separated on Rxi®-5Sil MS columns, including structurally-related compounds, such as benzodiazepines. Benzodiazepines are often analyzed on a fluorinated phase (e.g. Rtx®-200), but the selectivity of the Rxi®-5Sil MS column provides complete separation of all peaks of interest (Figure 5). Since a fluorinated column is no longer necessary, more time can be spent running samples with fewer time-consuming column changes between methods.

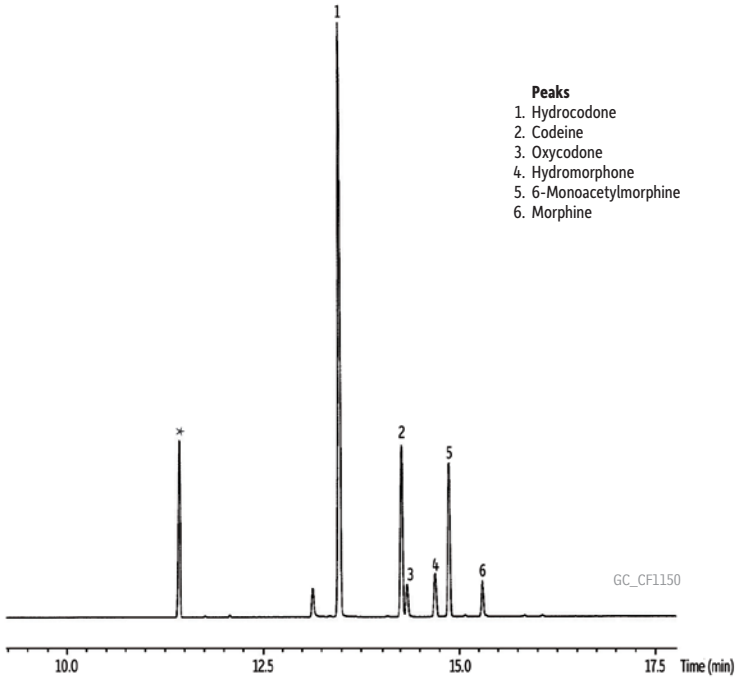
In addition to benzodiazepines, the selectivity of the Rxi®-5Sil MS column is also well-suited for the analysis of several common classes of drugs of abuse including cannabinoids, cocaine and its metabolites, opiates, and amphetamines. The Miami Dade Medical Examiner's Laboratory provides another example of how Rxi®-5Sil MS columns can simplify analyses and improve lab efficiency. The versatility and robustness of the Rxi®-5Sil MS column assisted the lab in streamlining operations by reducing time-consuming column changes and maintenance. One of the applications routinely run on this column is the analysis of opiates (Figure 6). The selectivity of the Rxi®-5Sil MS column gives excellent separation between all compounds, and very low limits of detection are achieved since bleed is minimal. In addition, the column stands up extremely well to the derivatization reagents used prior to analysis, further increasing throughput by reducing instrument downtime for maintenance. The Rxi®-5Sil MS column also produces excellent chromatography for cannibinoids (Figure 7).

Figure 5: No need to change columns to analyze benzodiazepines—Rxi®-5Sil MS columns give excellent separation of structurally-related benzodiazepines.



Column: Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623); **Sample:** 15 µg/mL in butyl chloride; **Injection:** Inj. Vol.: 1 µL splitless (hold 1 min); Liner: 3.5 mm splitless taper w/wool (cat.# 22286-200.1); Inj. Temp.: 280 °C; Purge Flow: 32.2 mL/min (20:1 split); **Oven:** Oven Temp: 200 °C to 330 °C at 15 °C/min (hold 3 min); **Carrier Gas:** He, constant linear velocity, 50 cm/sec, 23.7 psi, 163.4 kPa @ 200 °C; **Detector:** MS, Scan; Transfer Line Temp: 280 °C; Analyzer Type: Quadrupole; Source Temp.: 200 °C; Electron Energy: 70 eV; Solvent Delay Time: 4 min; Tune Type: PFTBA; Ionization Mode: EI; Scan Range: 50-350 amu; Scan Rate: 5 scans/sec; **Instrument:** Shimadzu 2010 GC & QP2010+ MS

Figure 6: Analysis of derivatized opiates on an Rxi®-5Sil MS column performed by the Miami Dade Medical Examiner's lab.

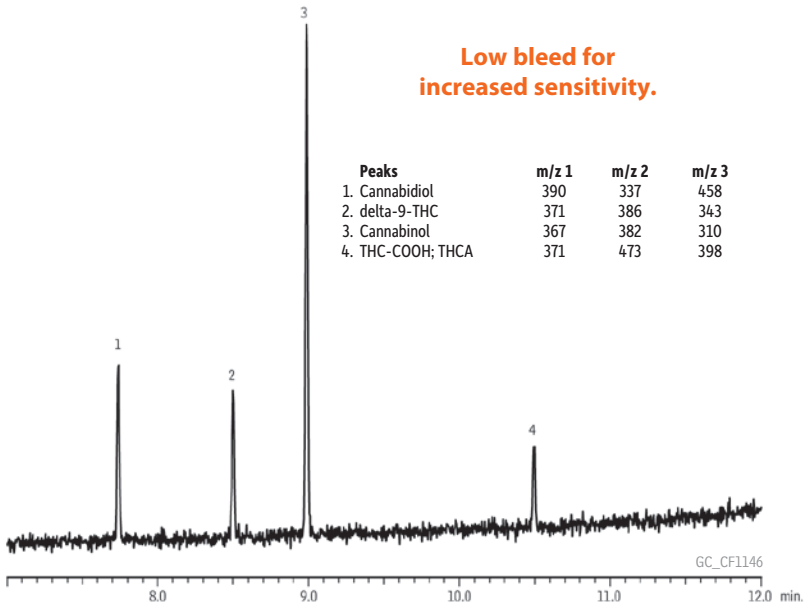


Peaks
1. Hydrocodone
2. Codeine
3. Oxycodone
4. Hydromorphone
5. 6-Monoacetylmorphine
6. Morphine

Column	Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623)
Sample	
Diluent:	Ethyl acetate
Conc.:	100 ng/mL propionic anhydride derivatives
Injection	
Inj. Vol.:	1 µL splitless (hold 1 min)
Liner:	4 mm splitless taper w/wool (cat.# 22405)
Inj. Temp.:	250 °C
Purge Flow:	100 mL/min
Oven	
Oven Temp:	65 °C (hold 1 min) to 315 °C at 15 °C/min
Carrier Gas	He, constant flow
Flow Rate:	1 mL/min
Linear Velocity:	35 cm/sec @ 65 °C
Detector	MS
Mode:	SIM
Transfer Line Temp.:	250 °C
Analyzer Type:	Quadrupole
Solvent Delay Time:	7 min
Tune Type:	PFTBA
Ionization Mode:	EI
Notes	Opiates were spiked into a blood sample and extracted by SPE, then derivatized with propionic anhydride.

Acknowledgement
Data courtesy of Miami Dade County Medical Examiner Department

Figure 7: High signal response due to column inertness and efficiency, combined with low bleed, results in maximum sensitivity for derivatized cannabinoids (50 ng/mL).



Peaks	m/z 1	m/z 2	m/z 3
1. Cannabidiol	390	337	458
2. delta-9-THC	371	386	343
3. Cannabinol	367	382	310
4. THC-COOH; THCA	371	473	398

Column	Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623)
Sample	
Diluent:	Ethyl acetate
Conc.:	50 ng/mL TMS derivatives
Injection	
Inj. Vol.:	1 µL splitless (hold 1 min.)
Liner:	3.5 mm splitless taper w/wool (cat.# 22286-200.1)
Inj. Temp.:	250 °C
Purge Flow:	21.4 mL/min
Oven	
Oven Temp:	150 °C to 330 °C at 15 °C/min (hold 3 min)
Carrier Gas	He, constant linear velocity
Linear Velocity:	40 cm/sec, 13.8 psi, 95.1 kPa @ 150 °C
Detector	MS
Mode:	SIM
SIM Program:	390, 337, 458, 367, 382, 310, 371, 386, 343, 473, 398 m/z
Transfer Line Temp.:	280 °C
Analyzer Type:	Quadrupole
Source Temp.:	200 °C
Solvent Delay Time:	4 min
Tune Type:	PFTBA
Ionization Mode:	EI
Instrument	Shimadzu 2010 GC & QP2010+ MS

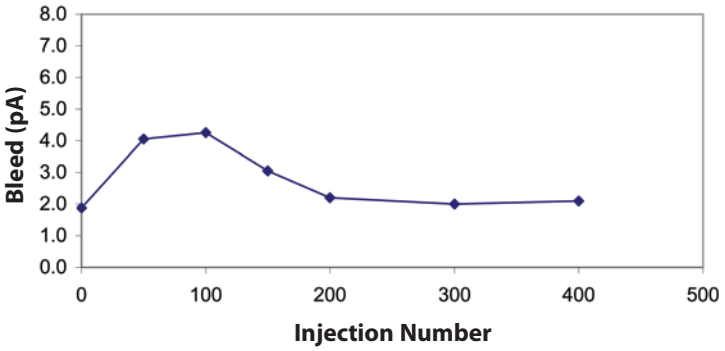
Rxi®-5Sil MS Columns…

Robust, Low-Bleed Phase Results in Better Sensitivity and Longer Column Lifetime

Many drug assays require that compounds be derivatized prior to analysis. Derivatization not only allows for GC analysis of compounds not otherwise amenable to gas chromatography, it also helps to produce unique, high molecular weight fragments that assist with GC-MS quantitation. While derivatization has its advantages, derivatization reagents and their byproducts are extremely harsh and can reduce column lifetimes by damaging the stationary phase. Phase damage usually manifests as increased bleed and tailing of active compounds. The unique Rxi®-5Sil MS stationary phase, with its embedded arylene groups, provides a more rigid matrix that is less likely to be damaged by derivatization reagents or their byproducts.

As a test of column lifetime, an Rxi®-5Sil MS column was subjected to repeated injections of high concentration HFAA, a harsh derivatization reagent, as well as prolonged exposure to the column's maximum operational temperature during each injection. Throughout lifetime testing, column bleed and inertness were tested by analyzing a mixture of active test compounds that tail severely on less inert columns. After 400 injections, no change in bleed or inertness was observed (Figures 8 and 9). The enhanced stability of Rxi®-5Sil MS columns reduces phase bleed, resulting in longer column lifetimes and improved performance with sensitive mass spectrometry detectors.

Figure 8: Low column bleed results in long column lifetimes, saving labs replacement costs.

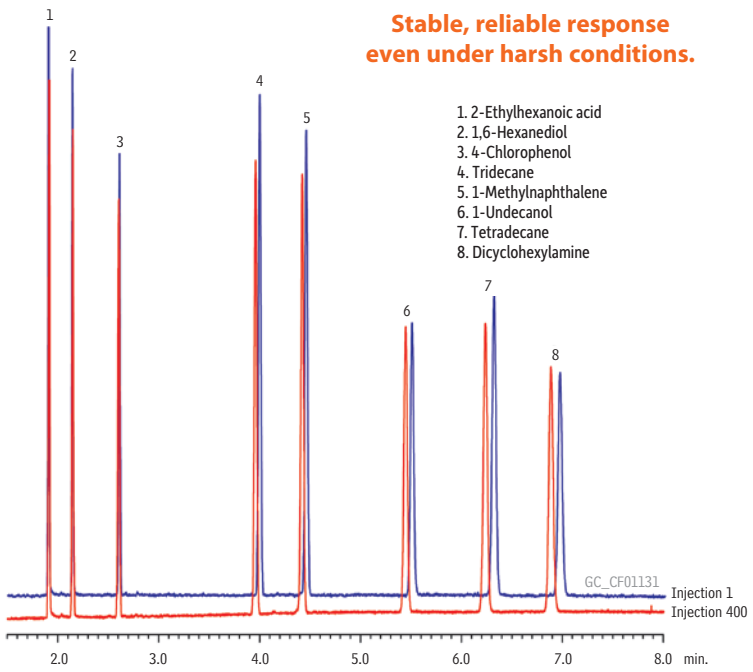


Column bleed over 400 injections of HFBA derivatization reagent. Column was held at the maximum isothermal temperature.

Conclusion

Rxi®-5Sil MS columns are ideal for toxicology labs interested in improving data quality by increasing certainty and reducing downtime. These columns have similar selectivity to conventional 5 type columns, but are significantly more inert and robust. Rxi®-5Sil MS columns provide more accurate trace-level results and reduced downtime for column changes, offering labs a valuable tool for improving methods for the routine analysis of drugs of abuse.

Figure 9: Rugged Rxi®-5Sil MS columns produce consistent retention times, even after 400 injections of derivatization reagent.



Column: Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623); **Sample:** Column test mix (cat.# 35226); **Inj.:** 1.0 µL split (split ratio 1:60), 4 mm recessed single taper (cat.# 20983); Inj. temp.: 250 °C; **Carrier gas:** helium, constant pressure; Linear velocity: 36 cm/sec @ 125 °C; Oven temp.: 125 °C; **Det:** FID @ 320 °C; Instrument: Agilent 6890

Assured Performance for Forensic Applications

Restek's low-bleed MS columns exceed requirements of the most sensitive mass spectrometers.

Rxi®-5Sil MS Columns (fused silica)
(low polarity phase; Crossbond® 1,4-bis(dimethylsiloxy)phenylene dimethyl polysiloxane)

- Engineered to be a low-bleed GC-MS column.
- Excellent inertness for active compounds.
- General-purpose columns—ideal for GC-MS analysis of drugs of abuse.
- Temperature range: -60 °C to 320/350 °C.

The Rxi®-5Sil MS stationary phase incorporates phenyl groups in the polymer backbone. This improves thermal stability, reduces bleed, and makes the phase less prone to oxidation. Rxi®-5Sil MS columns are ideal for GC-MS applications requiring high sensitivity, including use in ion trap systems.

ID	df	temp. limits	15-Meter cat.#	30-Meter cat.#
0.25 mm	0.25 µm	-60 to 320/350 °C	13620	13623
	0.50 µm	-60 to 320/350 °C	13635	13638
0.32 mm	0.25 µm	-60 to 320/350 °C	13621	13624
	0.50 µm	-60 to 320/350 °C		13639

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Description	qty.	cat.#
15 m, 0.25 mm ID, 0.25 µm Rxi-5Sil MS w/10 m Integra-Guard Column	ea.	13620-127
30 m, 0.25 mm ID, 0.25 µm Rxi-5Sil MS w/5 m Integra-Guard Column	ea.	13623-124
30 m, 0.25 mm ID, 0.25 µm Rxi-5Sil MS w/10 m Integra-Guard Column	ea.	13623-127
15 m, 0.25 mm ID, 0.50 µm Rxi-5Sil MS w/5 m Integra-Guard Column	ea.	13635-124
30 m, 0.25 mm ID, 0.50 µm Rxi-5Sil MS w/5 m Integra-Guard Column	ea.	13638-124
30 m, 0.25 mm ID, 0.50 µm Rxi-5Sil MS w/10 m Integra-Guard Column	ea.	13638-127
30 m, 0.32 mm ID, 0.50 µm Rxi-5Sil MS w/5 m Integra-Guard Column	ea.	13639-125

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Sky® 4.0 mm ID Single Taper Inlet Liner w/ Wool
For Agilent GCs equipped with split/splitless inlets

ID x OD x L	qty.	cat.#
Single Taper, Sky Technology, Borosilicate Glass with Quartz Wool		
4.0 mm x 6.5 mm x 78.5 mm	ea.	23303.1
4.0 mm x 6.5 mm x 78.5 mm	5-pk.	23303.5
4.0 mm x 6.5 mm x 78.5 mm	25-pk.	23303.25

Sky® 4.0 mm ID Single Taper Inlet Liner
For Agilent GCs equipped with split/splitless inlets

ID x OD x L	qty.	cat.#
Single Taper, Sky Technology, Borosilicate Glass		
4.0 mm x 6.5 mm x 78.5 mm	ea.	23302.1
4.0 mm x 6.5 mm x 78.5 mm	5-pk.	23302.5
4.0 mm x 6.5 mm x 78.5 mm	25-pk.	23302.25

Recommended for Split Injection

Sky® 4.0 mm ID Precision® Inlet Liner w/ Wool
For Agilent GCs equipped with split/splitless inlets

ID x OD x L	qty.	cat.#
Precision, Sky Technology, Borosilicate Glass with Quartz Wool		
4.0 mm x 6.3 mm x 78.5 mm	ea.	23305.1
4.0 mm x 6.3 mm x 78.5 mm	5-pk.	23305.5
4.0 mm x 6.3 mm x 78.5 mm	25-pk.	23305.25

Sky® 4.0 mm ID Cyclo Inlet Liner
For Agilent GCs equipped with split/splitless inlets

ID x OD x L	qty.	cat.#
Cyclo, Sky Technology, Borosilicate Glass		
4.0 mm x 6.3 mm x 78.5 mm	ea.	23312.1
4.0 mm x 6.3 mm x 78.5 mm	5-pk.	23312.5
4.0 mm x 6.3 mm x 78.5 mm	25-pk.	23312.25

More columns, inlet liners, standards, and accessories are available at **www.restek.com/cft**

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