

Rxi[®]-624SiI MS Columns

Exceptionally Inert,
Low Bleed Columns for
Volatiles Analysis

- **Optimized selectivity** for volatiles and polar compounds ensures good separations.
- **Highly inert columns** improve accuracy and allow lower detection limits, even for active compounds.
- **Most thermally stable** 624 column available; low bleed, fully MS compatible.

NEW DIMENSIONS
now available!

www.restek.com/rxi

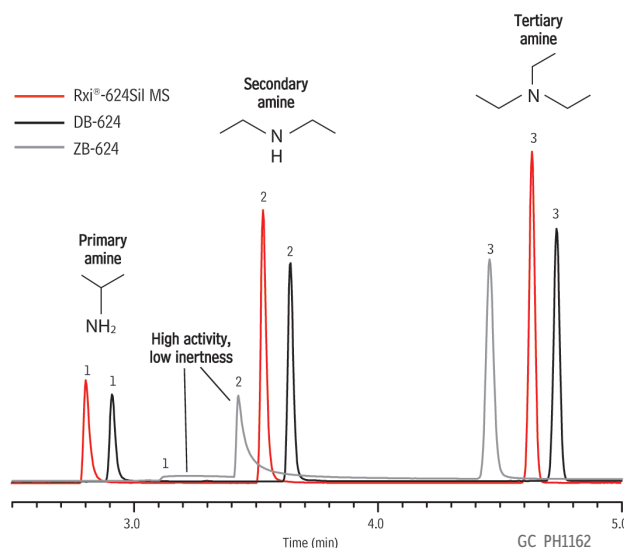
NEW! Rxi®-624Sil MS Columns: Increase Confidence in Data Accuracy

While mid polarity 624 columns are widely used for analyzing polar analytes and volatile organic compounds (VOCs), not all columns combine the selectivity needed for critical separations with the high inertness and low bleed that can further improve data quality. Whether you are developing methods for residual solvents, analyzing environmental VOCs, or running other applications for volatile organics, you can improve data quality with Rxi®-624Sil MS columns. These new columns incorporate a new stationary phase chemistry, unique column deactivation, and optimized manufacturing process that is specifically designed to provide the high inertness and thermal stability needed for greater accuracy and lower detection limits. The unique selectivity, inertness, and thermal stability of the Rxi®-624Sil MS column make it ideal for numerous applications, from detecting impurities in pharmaceuticals to monitoring environmental VOCs.

Exceptional Inertness Provides Better Peak Shape, Higher Sensitivity, and More Accurate Data

Column inertness is difficult to achieve, but critical to improving data quality. The deactivation process used for Rxi®-624Sil MS columns yields a fully passivated surface that is demonstrably more inert than other 624 columns. Comprehensive deactivation results in higher responses, more symmetrical peaks, and easy, accurate integration, even for active compounds at low levels (Figures 1 and 2). Rxi®-624Sil MS columns, with their superior deactivation, provide the inertness needed for improved linearity, greater accuracy, and lower detection limits.

Figure 1 Highly inert Rxi®-624Sil MS columns provide better peak shape and simplify integration for active compounds at low levels (5 ng on-column).



Highly inert Rxi®-624Sil MS columns give excellent peak symmetry.

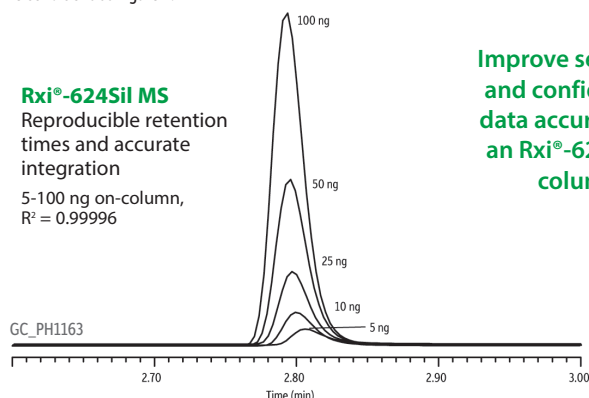
Peaks	Conc. (µg/mL)
1. Isopropylamine	100
2. Diethylamine	100
3. Triethylamine	100

Column	Rxi®-624Sil MS, 30 m, 0.32 mm ID, 1.8 µm (cat.# 13870)
Sample	DMSO
Diluent:	100 µg/mL
Conc.:	
Injection	
Inj. Vol.:	1 µL split (split ratio 20:1)
Liner:	5 mm Single Gooseneck with Wool (cat.# 22973-200.1)
Inj. Temp.:	250 °C
Oven	
Oven Temp:	50 °C (hold 1 min.) to 200 °C at 20 °C/min. (hold 5 min.)
Carrier Gas	He, constant flow
Linear Velocity:	37 cm/sec.
Detector	FID @ 250 °C
Instrument	Agilent/HP6890 GC

Figure 2 Active compounds like isopropylamine can be more accurately integrated on an Rxi®-624Sil MS column, lowering limits of quantification (LOQs) and increasing data accuracy.

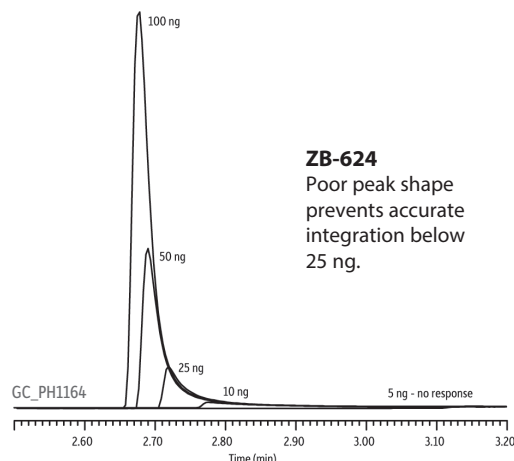
Same conditions as Figure 1.

Rxi®-624Sil MS
Reproducible retention times and accurate integration
5-100 ng on-column,
 $R^2 = 0.99996$



Improve sensitivity and confidence in data accuracy with an Rxi®-624Sil MS column.

ZB-624
Poor peak shape prevents accurate integration below 25 ng.



Lowest Bleed 624 Available—Assured GC-MS Compatibility

In addition to providing greater inertness and more accurate results for active compounds, the Rxi®-624Sil MS column offers higher temperature stability than any other column in its class (Table I, Figure 3). Even though most 624 columns provide adequate selectivity for polar compounds, poor thermal stability results in stationary phase bleed that can reduce column lifetime, decrease detector sensitivity, and interfere with the quantification of later eluting compounds. The highly effective stationary phase bonding chemistry of the Rxi®-624Sil MS column ensures extremely low bleed up to 320°C. While other 624 columns generate too much bleed to be useful for continuous mass spectrometry work, the Rxi®-624Sil MS column is fully compatible with both quadrupole and ion trap mass spectrometers. In addition to MS compatibility, higher thermal stability results in more stable baselines, longer column lifetimes, and improved method reproducibility.

Table I The Rxi®-624Sil MS column has the highest thermal stability of any 624 column.

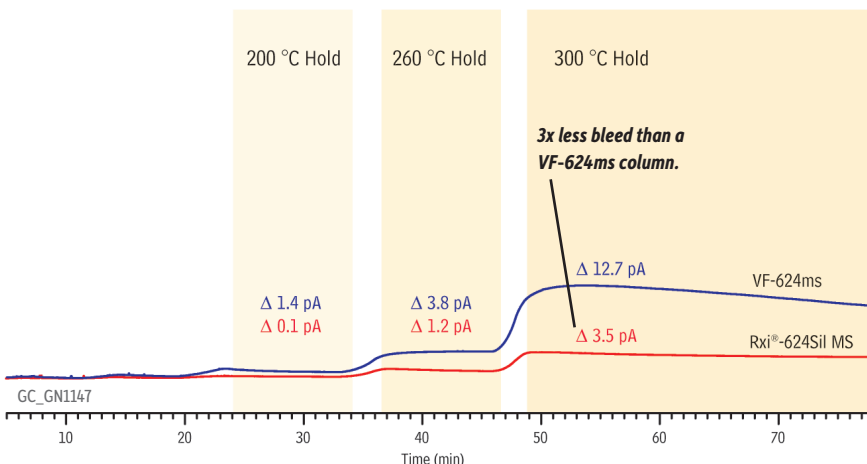
Column	Manufacturer	Maximum Programmable Temperature
Rxi-624Sil MS	Restek	320 °C
VF-624ms	Varian	300 °C
DB-624	Agilent J&W	260 °C
ZB-624	Phenomenex	260 °C

Data obtained from company website or literature for a 30 m x 0.25 mm x 1.4 µm df column.

Figure 3 The Rxi®-624Sil MS column has the lowest bleed of any column in its class and provides true GC-MS capability.

High thermal stability Rxi®-624Sil MS columns offer:

- Longer column lifetime.
- GC-MS compatibility.
- Improved method reproducibility.
- Stable baseline.



Columns: 30 m, 0.25 mm ID, 1.4 µm (Columns are of equivalent dimensions and were tested after equivalent conditioning.) Complete analytical conditions for chromatogram GC_GN1147 are available at www.restek.com

Rxi®-624Sil MS Columns (fused silica)

(midpolarity Crossbond® silarylene phase; similar to 6% cyanopropylphenyl/94% dimethyl polysiloxane)

- Low bleed, high thermal stability column—maximum temperatures up to 320 °C.
- Inert—excellent peak shape for a wide range of compounds.
- Selective—highly selective for residual solvents, great choice for USP<467>.
- Manufactured for column-to-column reproducibility—well-suited for validated methods.


ID	df	temp. limits	20-Meter	30-Meter	60-Meter	75-Meter	105-Meter
0.18mm	1.00µm	-20 to 300/320°C	13865				
0.25mm	1.40µm	-20 to 300/320°C		13868	13869 NEW		
0.32mm	1.80µm	-20 to 300/320°C		13870	13872		
0.53mm	3.00µm	-20 to 280/300°C		13871	13873 NEW	13874 NEW	13875 NEW



get more

For more information on the new Rxi®-624Sil MS column, visit www.restek.com and review our technical literature.

- Volatile Impurities Method Development (flyer PHFL1245)
- Residual Solvent Analysis: Implementing USP<467> (flyer PHFL1018A)
- Optimized Volatiles Analysis Ensures Fast VOC Separations (application note EVAN1271)



In the pharmaceutical industry, timing and certainty are everything. Time-to-market is a key driver for new drugs, and efficient batch testing is critical for releasing approved products. Whether developing new methods or conducting routine analysis, increasing productivity depends on choosing the right column for the application. Rxi®-624Sil MS columns provide enhanced retention of polar compounds and volatile analytes, as well as full MS compatibility, making them the best choice for many drug analyses.

Fast, Effective Method Development

Often, 1 and 5 type columns are used initially for GC-MS method development because of their thermal stability; however, their nonpolar character results in poor retention for polar compounds, which increases method development time. In contrast, effective methods can be developed quickly on mid polarity Rxi®-624Sil MS columns, because they provide greater retention and selectivity for polar compounds as well as good thermal stability. For example, highly volatile, polar alkyl halide genotoxic impurities are difficult to retain on 1s and 5s, but Rxi®-624Sil MS columns provide the retention needed to ensure adequate separation (Figure 4). Increased retention makes GC-MS analysis easier to control and ultimately allows faster method development.

Improving Results for Routine Analysis

Once a drug is approved, fast, reliable methods are needed for routine batch analysis. Establishing system suitability is an important part of these procedures and a major factor in overall lab productivity. Rxi®-624Sil MS columns provide the optimized selectivity and guaranteed reproducibility needed to increase pass rates. For example, batch throughput can be improved for residual solvent testing under USP <467> by using a column that provides increased resolution for system suitability components (Figure 5). Greater resolution of critical pairs means higher system suitability pass rates, which allows more batches to be analyzed per shift.

Optimized phase chemistry, complete column deactivation, and tightly-controlled manufacturing make Rxi®-624Sil MS columns the best choice for many pharmaceutical applications. With better retention of polar volatiles, lower bleed, and higher inertness, Rxi®-624Sil MS columns can improve lab productivity by allowing new methods to be developed quickly and routine applications to be run more reliably.

learn more

For more pharmaceutical applications on Rxi®-624Sil MS columns, visit www.restek.com and download flyer PHFL1245.

TECH TIP!



Tim Herring, Technical Service Specialist

When running USP <467> by headspace, using a smaller bore liner (1 mm) can improve system suitability pass rates. Larger bore liners (4 mm) are used with direct liquid injection because the sample is vaporized in the injection port and the liner must be able to accommodate the solvent expansion volume. In contrast, in headspace analysis, the sample is vaporized in a vial instead of the injection port, so a large volume liner is not needed, and in fact it can be deleterious. In headspace methods, using a smaller bore liner reduces band broadening by increasing linear velocity, allowing faster sample transfer and improving resolution.

Resolution passes USP <467> criteria when using a 1 mm liner (red line), but fails if a 4 mm liner is used (black line).

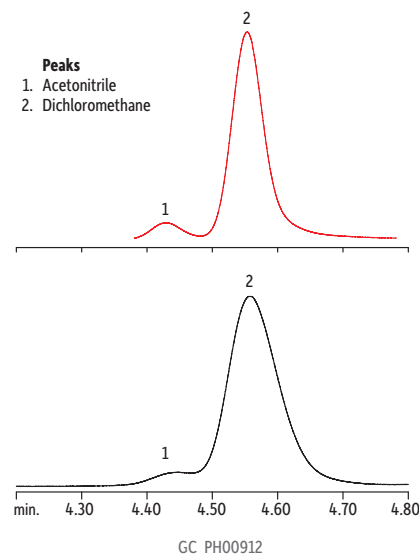
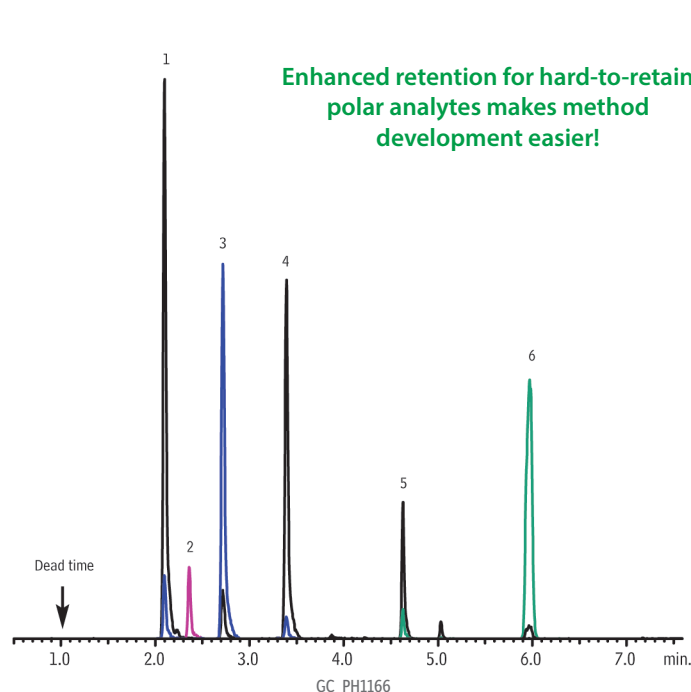


Figure 4 Polar compounds, such as alkyl halides, are highly retained on mid polarity Rxi®-624Sil MS columns, making method development faster and easier than on a nonpolar 1 or 5 type column.

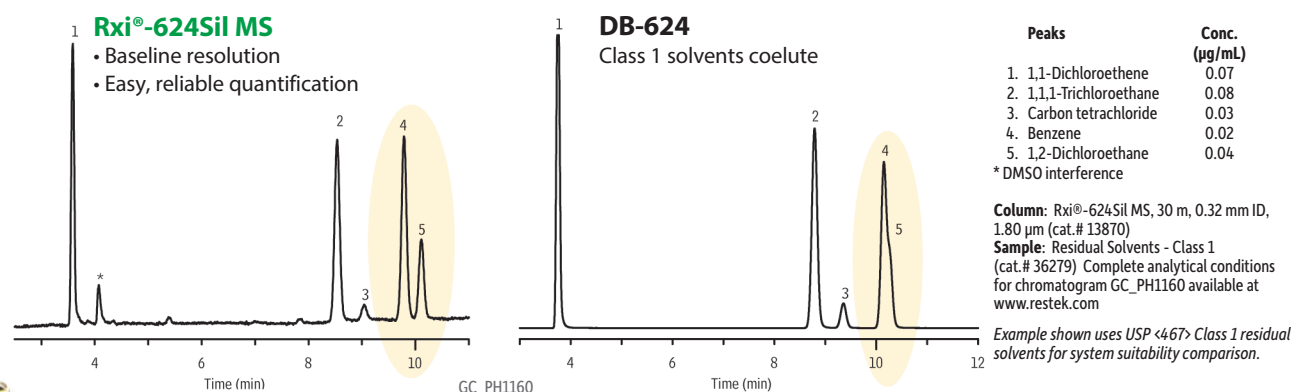


Peaks

1. 2-Chloropropane
2. Bromoethane
3. 1-Chloropropane
4. 2-Bromopropane
5. Butyl chloride
6. 1-Bromobutane

Column Rxi®-624Sil MS, 20 m, 0.18 mm ID, 1.00 µm (cat.# 13865)
Sample
Diluent: DMSO
Conc.: 1 µg/mL
Injection
Inj. Vol.: 1 µL splitless (hold 0.5 min.)
Liner: 3.5 mm Single Gooseneck Liner with wool placed 3 cm from top (middle) (cat.# 22286)
Inj. Temp.: 220 °C
Purge Flow: 3 mL/min.
Oven
Oven Temp: 40 °C (hold 3 min.) to 200 °C at 20 °C/min.
Carrier Gas He, constant flow
Linear Velocity: 40 cm/sec.
Detector MS
Mode: Scan
Transfer Line Temp.: 280 °C
Analyzer Type: Quadrupole
Source Temp.: 280 °C
Solvent Delay Time: 0.5 min.
Ionization Mode: EI
Scan Range: 30-300 amu
Scan Rate: 5 scans/sec.
Instrument Shimadzu 2010 GC & QP2010+ MS
Notes Ions displayed: 42, 43, 57, 108 m/z

Figure 5 System suitability pass rates can be improved with Rxi®-624Sil MS columns. The innovative polymer chemistry provides greater resolution of critical pairs that are difficult to separate on other 624 type columns.



Peaks

- | Peaks | Conc. (µg/mL) |
|--------------------------|---------------|
| 1. 1,1-Dichloroethene | 0.07 |
| 2. 1,1,1-Trichloroethane | 0.08 |
| 3. Carbon tetrachloride | 0.03 |
| 4. Benzene | 0.02 |
| 5. 1,2-Dichloroethane | 0.04 |

* DMSO interference

Column: Rxi®-624Sil MS, 30 m, 0.32 mm ID, 1.80 µm (cat.# 13870)

Sample: Residual Solvents - Class 1 (cat.# 36279) Complete analytical conditions for chromatogram GC_PH1160 available at www.restek.com

Example shown uses USP <467> Class 1 residual solvents for system suitability comparison.

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Increase Sample Throughput for Environmental VOCs

Fast sample throughput is a primary concern for environmental labs interested in improving productivity. Volatiles methods typically are time-consuming, but developing optimized procedures can be challenging because compound lists are extensive and analytes vary significantly in chemical characteristics. The selectivity and inertness of Rxi®-624Sil MS columns make them ideal for optimizing environmental volatiles methods for better resolution and faster analysis time.

Establishing conditions that maximize sample throughput can be difficult, because conditions optimized for speed can result in problematic coelutions, while conditions optimized for resolution can result in long analysis times. The exceptional inertness of Rxi®-624Sil MS columns produces highly symmetrical peaks for active compounds, which improves resolution and allows separations to be maintained even under faster conditions. Here, an optimized method was developed using an Rxi®-624Sil MS column to maintain adequate resolution, while throughput was maximized by synchronizing purge and trap cycles with instrument cycles.

Improve Productivity and Resolve Critical Pairs

Initially, several critical pairs were chosen for computational modeling using Pro ezGC software. The temperature program first determined by the software provided the best resolution, but also resulted in an analysis time of 19 minutes. Since the purge and trap cycle time was 16.5 minutes, other conditions were evaluated to see if adequate resolution could be maintained using a faster instrument cycle. The final program, shown in Figure 6, reduced instrument downtime by better synchronizing purge and trap cycles with instrument cycles, and also provided excellent resolution. Using these conditions, up to 36 samples can be analyzed following EPA Method 8260 in a typical 12-hour shift.

Labs interested in optimizing both sample throughput and resolution of VOCs can adopt the synchronized conditions established here on Rxi®-624Sil MS columns to maximize productivity and assure accurate, reliable results.

For the complete application, visit www.restek.com/adv002



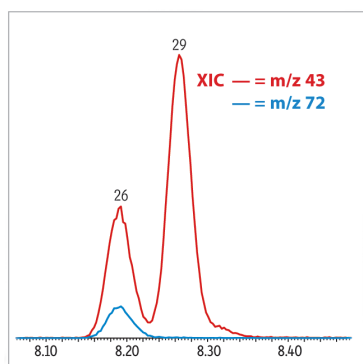
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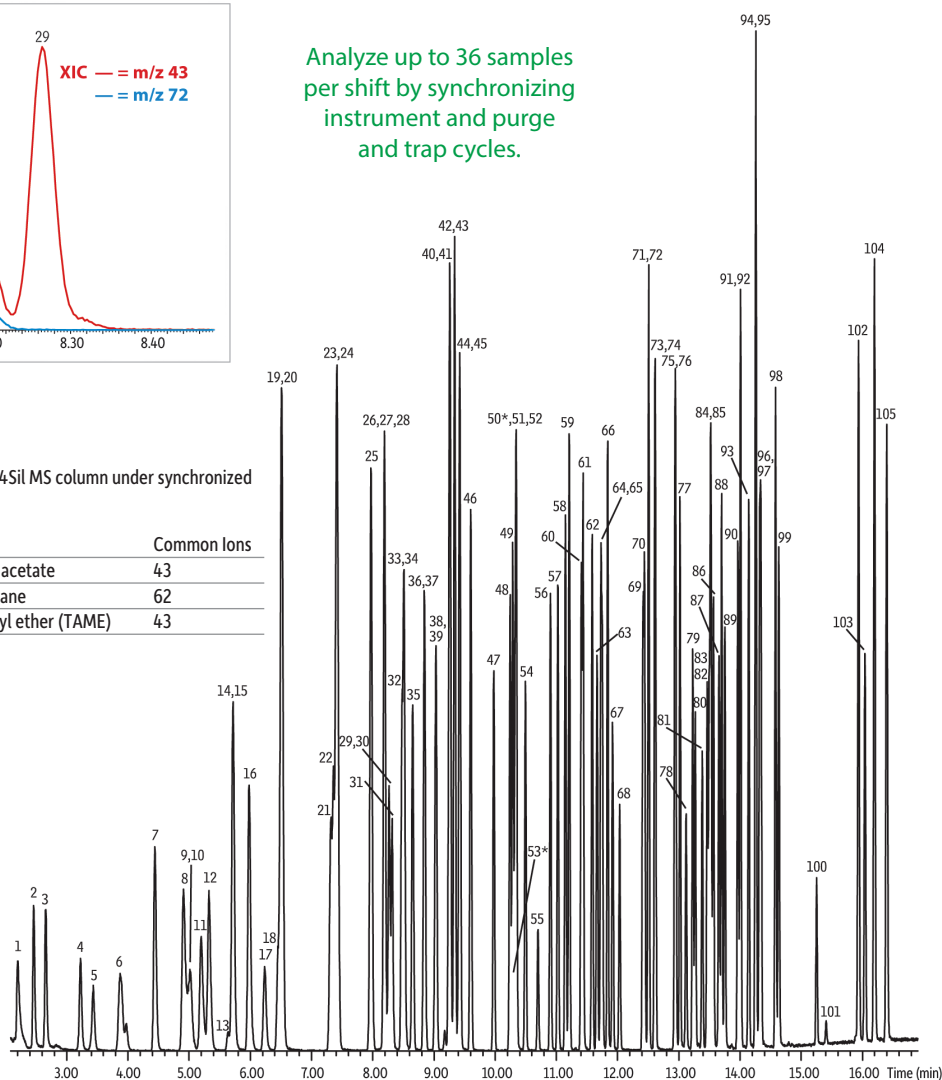
Figure 6 Using an Rxi®-624Sil MS column under optimized conditions increases productivity by assuring good resolution and minimal downtime when analyzing environmental volatiles.



Analyze up to 36 samples per shift by synchronizing instrument and purge and trap cycles.

Critical pairs resolved using an Rxi®-624Sil MS column under synchronized conditions:

Peak #s	Compounds	Common Ions
26/29	2-butanone (MEK)/ethyl acetate	43
41/42	benzene/1,2-dichloroethane	62
41/45	benzene/ <i>tert</i> -amyl methyl ether (TAME)	43



For peak identifications and conditions visit www.restek.com and enter chromatogram GC_EV1169 in the search function.

Rxi®-624Sil MS Columns (fused silica)

(midpolarity Crossbond® silarylene phase; similar to 6% cyanopropylphenyl/94% dimethyl polysiloxane)

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U.S. • 110 Benner Circle • Bellefonte, PA 16823 • 1-814-353-1300 • 1-800-356-1688 • fax: 1-814-353-1309 • www.restek.com

China • phone: +86-10-5629-6620 • fax: +86-10-5814-3980 • cn.restek.com

France • phone: +33 (0)1 60 78 32 10 • fax: +33 (0)1 60 78 70 90 • www.restek.fr

Germany • phone: +49 (0)6172 2797 0 • fax: +49 (0)6172 2797 77 • www.restekgmbh.de

Italy • phone: +39-02-7610037 • fax: +39-02-70100100 • www.superchrom.it

Japan • phone: +81 (3)6459 0025 • fax: +81 (3)6459 0025 • e-mail: restekjapan@restek.com

UK • phone: +44 (0)1494 563377 • fax: +44 (0)1494 564990 • www.thamesrestek.co.uk

