

# Accelerate Development



# Rxi®-624Sil MS

## The Go To GC Column for Fast, Effective Volatile Impurities Method Development

In drug development, time-to-market is everything, but finding the right column can be laborious and time-consuming. Commonly, "624" (6% cyanopropyl phenyl/94% dimethyl polysiloxane) type columns are used for GC/FID impurity analyses to provide the necessary selectivity, but when mass spectrometry is needed, method development often starts with lower bleed "1" (100% dimethyl polysiloxane) and "5" (5% diphenyl/95% dimethyl polysiloxane) type columns. Now, you can get to market faster and more efficiently by using a single column that combines these attributes—the new Rxi®-624Sil MS column. With enhanced retention and selectivity of polar compounds, compatibility with mass spec detectors, and unsurpassed inertness, Rxi®-624Sil MS columns are the most broadly applicable GC columns available to the pharmaceutical industry. Speed up successful method development by making Rxi®-624Sil MS columns your "go to" column of choice for polar impurities.

## Go To...the right column first

For better retention of polar analytes and improved accuracy, peak shape, and response for active compounds.

## Go To... mass spec directly

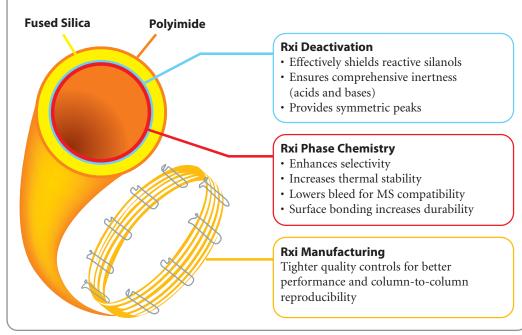
With the lowest bleed 624 column available; stable up to 320 °C, for easy transfer of methods to GC/MS.

### Go To...the next batch faster

With the best-in-class G43 for USP methods.

## How did we create the Rxi Column Family?

We've optimized phase chemistry, column deactivation, and our manufacturing process to ensure the comprehensive performance that makes Rxi®-624Sil MS columns the best starting point for method development.

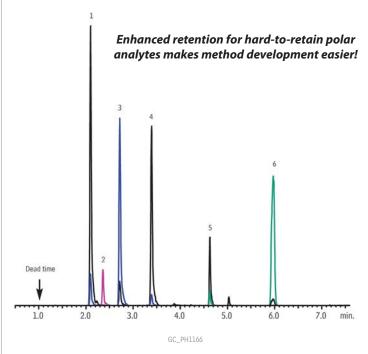


# Go To...the Right Column First

#### **Balanced Retention Simplifies Method Development For Polar Impurities**

While "1s and 5s" are often used initially in GC/MS method development because of their thermal stability, their nonpolar character results in poor retention for polar compounds and costs additional development time. In contrast, midpolarity Rxi®-624Sil MS columns provide improved retention and selectivity for polar compounds and are also more compatible with polar injection solvents. Highly volatile, polar alkyl halide genotoxic impurities, for example, are difficult to retain on 1s and 5s, but the Rxi®-624Sil MS column provides higher retention capacity, making GC/MS analysis easier to control and allowing faster method development (Figure 1).

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



Peaks	RT (min.)
1. 2-Chloropropane 2. Bromoethane 3. 1-Chloropropane 4. 2-Bromopropane 5. Butyl chloride 6. 1-Bromobutane	2.10 2.36 2.72 3.393 4.627 5.973

Column Rxi $^{\circ}$ -624Sil MS, 20 m, 0.18 mm ID, 1.00  $\mu$ m (cat.# 13865) Sample Diluent: Conc :  $1 \mu \mathrm{g/mL}$  each compound Injection  $1\,\mu\text{L}$  splitless (hold 0.5 min.) Inj. Vol.: 3.5mm Single Gooseneck Liner with wool placed 3cm Liner: from top (middle) (cat.# 22286) Inj. Temp.: Purge Flow: 3 mL/min. Oven 40 °C (hold 3 min.) to 200 °C at 20 °C/min.

Oven Temp: 40 °C (hold 3 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

 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 201

Instrument Shimadzu 2010 GC & QP2010 + MS Notes: EIC of 42, 43, 57, 108 m/z



Visit www.restek.com/rxi for detailed comparisons and to learn how exceptional Rxi® inertness, bleed, and reproducibility can improve your data.

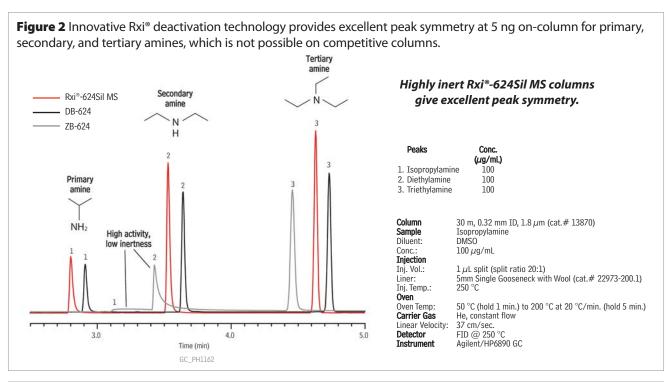


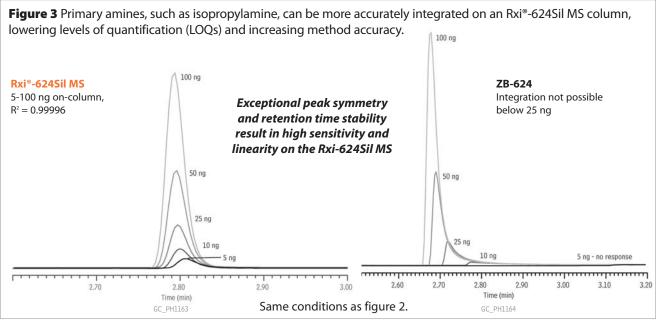
The versatility of an Rxi®-624Sil MS column makes it a perfect fit for Quality By Design.

# Go To...the Right Column First

## Balanced Inertness Gives Higher Data Quality— Excellent Peak Symmetry and Reproducibility for Active Compounds

In addition to offering better retention of polar analytes, Rxi®-624Sil MS columns are exceptionally inert, reducing the need to switch columns when developing methods for active compounds, such as amines. Amines are commonly found on pharmaceutical impurities and can interact with surface silanols resulting in a tailing peak. Proper deactivation is the best way to combat this, and Rxi® technology provides the most balanced deactivation, assuring good peak symmetry for both basic and acidic compounds. Columns that are not effectively deactivated for basic compounds produce unacceptable peak tailing (Figure 2). In contrast, the Rxi®-624Sil MS column provides excellent peak shape, which leads to consistent peak integration, improved linearity, and higher method sensitivity (Figure 3).





# Go To...the Right Column First

Other active compounds, such as glycols, also exhibit peak tailing as a result of reactivity with the chromatographic system. For example, when analyzing ethylene glycol and diethylene glycol in glycerin according to a new FDA Guidance for Industry, only the Rxi®-624Sil MS column gives the selectivity and peak symmetry needed for these reactive compounds (Figure 4). Satisfy this and other industry guidances quickly, by choosing the right column, the first time.

Figure 4 Rxi®-624Sil MS columns provide the best overall inertness and selectivity for ethylene glycol and diethylene glycol impurities in glycerin, glycol, or sorbitol solutions.

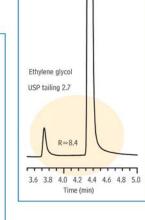
#### Best in Class—Rxi®-624Sil MS

	Rxi®-624Sil MS	ZB-624	DB-624
USP tailing (ethylene glycol)	1.8	2.7	1.8
Resolution (ethylene glycol/ propylene glycol)	9.7	8.4	7.2



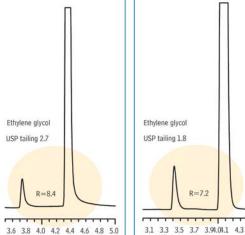
Propylene glycol

HO-



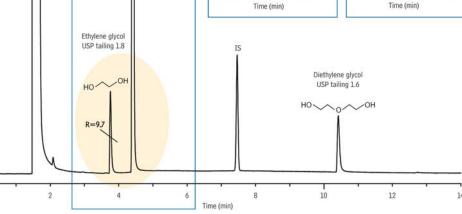
ZB-624 Column

Propylene glycol



DB-624 Column

Propylene glycol



GC PH1151

Peaks

Ethylene glycol

Propylene glycol

Diethylene glycol

2,2,2-Trichloroethanol (IS)

Column 30 m, 0.32 mm ID, 1.80 μm (cat.# 13870) Sample methanol Injection Ini. Vol.:  $1 \mu L$  split (split ratio 10:1) 5mm Single Gooseneck with Wool (cat.# 22973-200.1) Inj. Temp.: 100 °C (hold 4 min.) to 120 °C at 50 °C/min. (hold 10 min.) Oven Temp:

to 220 °C at 50 °C/min. (hold 6 min.) He, constant flow

Carrier Gas Linear Velocity: 40 cm/sec. FID @ 250 °C Detector Instrument

Columns tested: Rxi®-624Sil MS, ZB-624, and DB-624 Notes

#### **Innovation & Service**

"Having a background in LC/MS/MS does not automatically qualify one to run GC/MS. Julie Kowalski spent time with me to help me decide which column would be the best for my application as well as which consumables I would need to do routine maintenance. The time and knowledge she shared with me saved me multiple headaches and will keep me a loyal Restek customer!"

Richard, Biologist National Institute of Health

#### How can we help you today?

Contact support@restek.com or your local Restek representative for helpful, knowledgeable technical support.



RT (min.)

3.757

4.422

7.461

10.416

Conc.

(ma/mL)

0.05

2.0

## Go To... Mass Spec Directly

#### High Thermal Stability and Low Bleed for GC/MS Compatibility

While midpolarity 624 type columns offer better retention of polar analytes than 1s and 5s, most 624s have low thermal stability and generate too much column bleed to be useful for mass spec work. However, the Rxi®-624Sil MS column is fully compatible with mass spectrometry, due to stabilizing technology that delivers the highest thermal stability and lowest bleed of any polar capillary column in its class (Table I, Figure 5). Eliminate the need to change columns when mass spec is required—unlike other 624 columns, Rxi®-624Sil MS columns take your method directly to GC/MS. Keep the same 624 retention and selectivity, but leave the bleed behind.

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

Column	Manufacturer	Highest Temperature Limit (Isothermal)
Rxi®-624Sil MS	Restek	320 °C
VF-624ms	Varian	300 °C
DB-624	Agilent J&W	260 °C
ZB-624	Phenomenex	260 °C

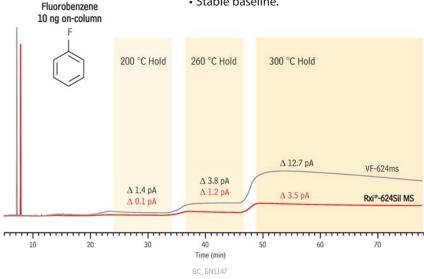


Data obtained from company website or literature for a 30 m x 0.25 mm x 1.4  $\mu$ m df column.

Figure 5 The Rxi®-624Sil MS column has the lowest bleed of any column in its class, providing true GC/MS capability.

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

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



Column 30 m, 0.25 mm ID, 1.4  $\mu$ m (cat.# 13868) Sample Fluorobenzene (cat.# 30030)

Diluent: methanol  $200 \,\mu \text{g/mL}$ Conc.: Injection

1 µL split (split ratio 20:1) Ini. Vol.:

4mm Split Liner with Wool (cat.# 20781) Liner:

Inj. Temp.: 220 °C Oven

Oven Temp: 40 °C (hold 5 min.) to 60 °C at 20 °C/min. (hold 5 min.) to 120 °C at 20 °C/min. (hold 5 min.)

to 200 °C at 20 °C/min. (hold 10 min.) to 260 °C at 20 °C/min. (hold 10 min.) to 300 °C at 20 °C/min. (hold 20 min.) He, constant flow

Carrier Gas Linear Velocity: 40 cm/sec. Detector FID @ 250 °C Instrument Agilent/HP6890 GC

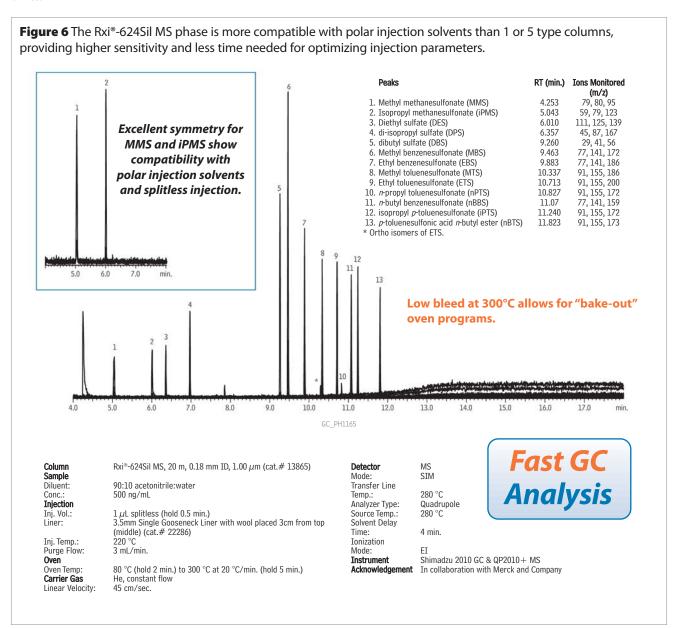
Columns are of equivalent dimensions and were tested after equivalent conditioning.



# Go To... Mass Spec Directly

#### Selective, Retentive, and Compatible with Polar Injection Solvents

The Rxi®-624Sil MS column combines the thermal stability of a mass spec friendly column with the selectivity, retention, and injection solvent compatibility needed to analyze polar impurities. For example, when analyzing mesylate, besylate, and tosylate genotoxic impurities by GC/MS, the Rxi®-624Sil MS column provides excellent selectivity, stability, and sensitivity (Figure 6). The innovative stationary phase is compatible with a variety of injection solvents, allowing splitless injection techniques to be used for heightened sensitivity. Bleed-free thermal stability allows oven "bake-out" programs to be used for contaminant removal and longer column lifetimes.





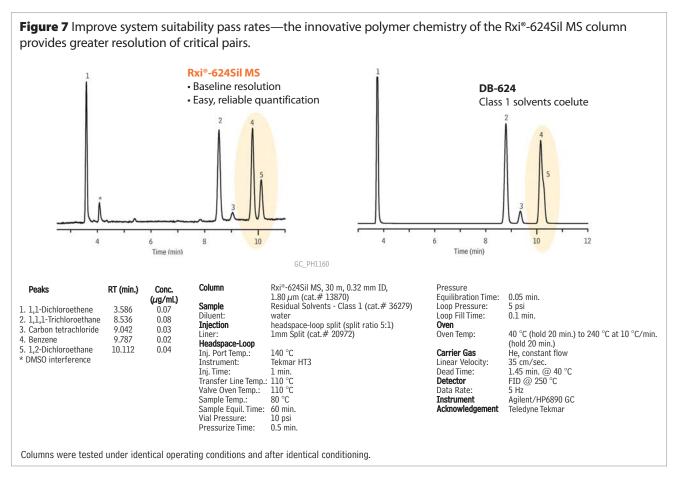
Restek carries a full line of innovative GC/MS accessories built to make your life easier.

Visit www.restek.com for a complete selection.

## Go To...the Next Batch Faster

#### Improve Pass Rates with the Best-In-Class G43 for USP <467>

System suitability is a major factor in overall lab productivity, and  $Rxi^{\circ}$ -624Sil MS columns provide the optimized selectivity and guaranteed reproducibility needed to increase pass rates. For example, batch throughput can be improved for USP <467> residual solvents analysis by using a column that provides increased resolution and sensitivity for system suitability components (Figures 7 and 8). Benefits include industry-leading resolution of acetonitrile and dichloromethane, as well as benzene and 1,2-dichloroethane. No other 624 type column performs as well as  $Rxi^{\circ}$ -624Sil MS columns for these critical system suitability requirements.







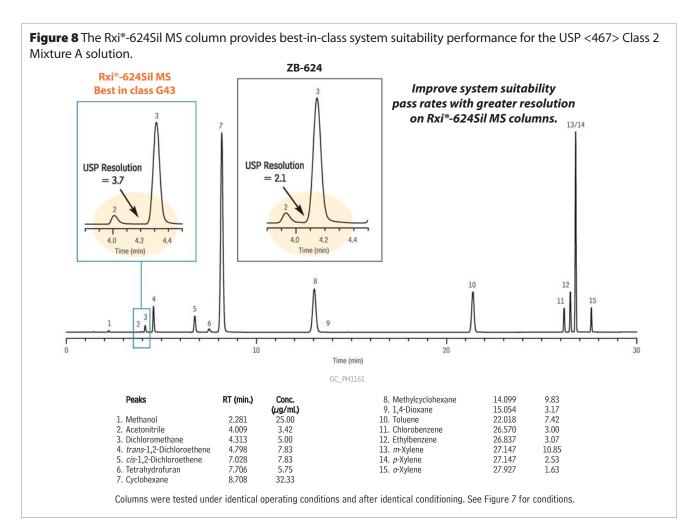
## Custom Residual Solvent Mixes

A perfect match for validated residual solvent methods

Save time and money with mixes prepared to your specific solvent set and concentrations. The more you buy the less you pay per ampule!

Easy Online Order Form! www.restek.com/customusp

## Go To...the Next Batch Faster



#### Screw-Thread Headspace Vials (18mm)

Description	Volume	Color	Dimensions	100-pk.	1000-pk.
Headspace Vial	10mL	Clear	22 x 45mm	23084	23085
Headspace Vial	10mL	Amber	22 x 45mm	23088	23089
Headspace Vial	20mL	Clear	22 x 75mm	23082	23085
Headspace Vial	20mL	Amber	22 x 75mm	23086	23089



Caps not included.

#### Magnetic Screw-Thread Caps (18mm)

Description	Septa Material	100-pk.	1000-pk.
Magnetic Caps and Septa	PTFE/Silicone	23090	23091
Magnetic Caps and Septa	PTFE/Silicone for SPME	23092	23093
Magnetic Caps and Septa	PTFE/Red Chlorobutyl	23094	23095



#### **Hot Swap Capillary Column Nuts**

Quickly change columns for USP <467> Procedures A and B using a **Hot Swap Capillary Column Nutl** 

Description	qty.	cat.#
For use with "compact" Agilent-style ferrules.		
Hot Swap Capillary Column Nut	ea.	22348
For use with standard 1/1/2"-type ferrules.		
Hot Swap Capillary Column Nut	ea.	22347



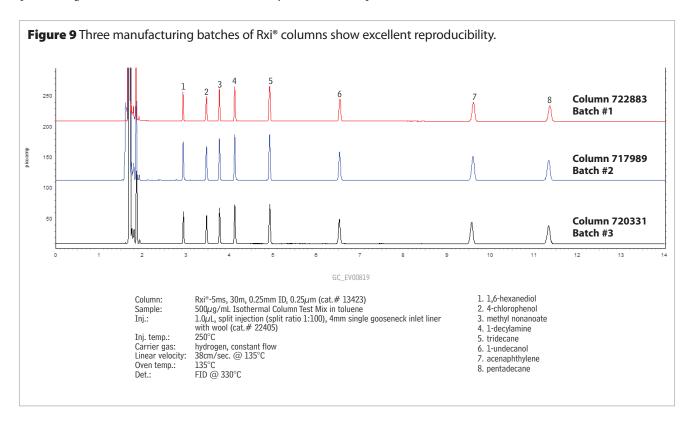
NOTE: For proper operation, oven fan must be kept operational during change out or risk of burn may occur.



## Go To...the Next Batch Faster

#### **Guaranteed Reliable Column-to-Column Performance**

Reliable column-to-column performance also contributes to lab productivity as less column variation means faster setup and more consistent results. Rxi® column technology has enabled Restek to tighten our quality control standards and guarantee consistency. Columns from multiple manufacturing batches show the excellent reproducibility obtained using the new Rxi® manufacturing process (Figure 9). All Rxi® columns are individually tested to assure performance.



## Go To...Rxi®-624Sil MS Columns for Faster Method Development

Optimized phase chemistry, column deactivation, and manufacturing make Rxi®-624Sil MS columns the "go to" column for pharmaceutical method development. With better retention of polar compounds than 1 and 5 type columns, lower bleed than any other 624 column, and unsurpassed inertness, Rxi®-624Sil MS columns offer the most comprehensive performance, allowing you to develop successful methods quickly, easily, and reliably. Try one for your next method today.

#### **Innovation & Service**

"When my research group needed a GC column for a chiral separation, Restek was the only company that offered to provide us with test columns to evaluate. The willingness of Restek to work with us to find a solution to our separation problem is exceptional."

#### Joe Dinnocenzo,

#### **Professor of Chemistry**

Director, Center for Photoinduced Charge Transfer University of Rochester

#### How can we help you today?

Contact support@restek.com or your local Restek representative for helpful, knowledgeable technical support.

# **Technical Opportunities**

Expand your knowledge and improve your results with Restek.

- Download our free technical literature.
- View free technical webinars.
- Contact us for on-site GC training.



# Compare and Save

#### Restek Offers an Exclusive Line of Innovative and Cost-Effective GC Products

#### Rxi®-624Sil MS Columns (fused silica)

(mid polarity Crossbond® silarylene phase; equivalent 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, including acidic and basic 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 (µm)	temp. limits	20-Meter	30-Meter	60-Meter	
0.18mm	1.00	-20 to 300/320°C	13865			
0.25mm	1.40	-20 to 300/320°C		13868		
0.32mm	1.80	-20 to 300/320°C		13870	13872	
0.53mm	3.00	-20 to 280/300°C		13871		



#### **Restek Electronic Leak Detector**

Why have a small leak turn into a costly repair? Protect your data and analytical column by using a Restek Leak Detector.

Description	qty.	cat.#	
Leak Detector with Hard-Sided Carrying Case and Universal Charger Set (US, UK, European, Australian)	ea.	22839	
Soft-Side Storage Case	ea.	22657	
Small Probe Adaptor	ea.	22658	



Avoid using liquid leak detectors on a GC! Liquids can be drawn into the system.

#### **GC/MS Cleaning Kit**

Description	qty.	cat.#
Mass Spec Cleaning Kit with Dremel Tool	kit	27194
Mass Spec Cleaning Kit without Dremel Tool	kit	27195
Mass Spec Cleaning Kit Replacement Parts Kit		
(includes cloths, micro mesh sheets, small and large gloves)	kit	27196



#### **ETP Electron Multipliers**

for Mass Spectrometry

- · Air stable.
- 2-year shelf life guarantee.
- Discrete dynode design results in extended operating life.

Description	qty.	cat.#	
Electron Multipliers for Agilent GC/MS and LC/MS			
For Agilent 5970 GC/MS	ea.	23072	
For Agilent 5971, 5972, GC GC/MS	ea.	23073	
For Agilent 5973 & 5975 GC/MS (includes mount for initial installation)*†	ea.	23074	
For Agilent 5973 & 5975 GC/MS and LC/MSD (Replacement Multiplier)*†	ea.	23075	
For Agilent LC/MSD (includes mount for initial installation)*†	ea.	23076	
Electron Multiplier for Applied Biosystems (Sciex)			
For API 300, 3000 & 4000 Applied Biosystems	ea.	23077	
Electron Multiplier for Thermo Finnigan GC/MS			
For Thermo TRACE DSQ, DSQII, and Polaris-Q GC/MS	ea.	23081	



 $\dagger$ This unit is designed for use in the 5975, 5973 GC and the LC/MSD.



## In Stock Now!

USP-equivalent residual solvent Class 1 & 2 mixes & singles.

Visit www.restek.com/usp467 for a complete selection





<sup>\*</sup>First time installation requires a mount which includes the mechanical housing. After initial installation, only the replacement electron multiplier is required.

# **Go To Restek** for Custom Residual Solvent Methods

Develop Residual Solvent Methods Faster by "Outsourcing" to Restek's Technical Support Team

Speed up residual solvent method development by letting Restek do the work for you. We have benchmarked ICH Class 1, 2, and 3 residual solvents on our most popular columns and will model your specific separations with our Pro ezGC software. We can get you started quickly and accurately with an optimized set of analytical conditions.

- Fast, effective method development with highly accurate results.
- Customized to your specific solvent list.
- Includes recommended column format and analytical conditions.

Contact Restek's Technical Support group at **support@restek.com** and let us do the work for you!

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