

# RESTEK ADVANTAGE Newsletter

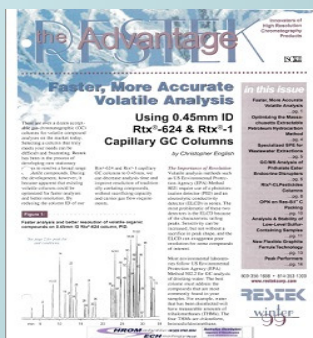
## 1996-2000 : Quarterly

## ADV Overview

2000



1999



1998



1997



1996



Archive Only : some products may be obsolete / redundant and technologies upgraded !

... see later Restek Updates

INT'l Suppl

Jun 94 5.3

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**ECHnology** Pty Ltd

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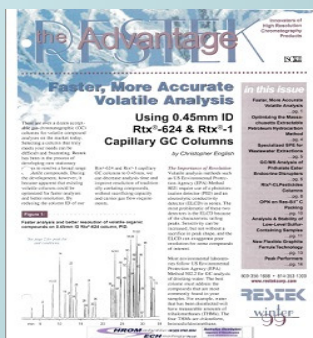
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# RESTEK ADVANTAGE Newsletter

## 1991-1995 : Quarterly

## ADV Overview

1994

**THE RESTEK ADVANTAGE**

**Columns for Blood Alcohol Analysis**  
Faster Analysis Time and Baseline Resolution

Today's law enforcement and forensic laboratories are faced with the task of analyzing blood alcohol content (BAC) in a timely and accurate manner. The RESTEK ADVANTAGE columns provide the perfect solution for this task. They offer faster analysis times and better baseline resolution than traditional columns.

**In this issue...**

- Blood Alcohol Analysis
- Fast Analysis Time
- Baseline Resolution
- Column Selection
- Peak Performance
- Flow Rate
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- Detector
- Injection System
- Sample Preparation
- Method Development
- Quality Control
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April 94 5.2

**THE RESTEK ADVANTAGE**

**Rtx®-1701**  
The Highest Quality 1701 Columns Available

The RESTEK ADVANTAGE Rtx®-1701 columns are the highest quality 1701 columns available. They are made from high-purity silica and have a uniform particle size distribution. This results in excellent flow characteristics and high efficiency.

**In this issue...**

- Rtx®-1701 Columns
- High Purity Silica
- Uniform Particle Size
- Excellent Flow Characteristics
- High Efficiency
- Column Selection
- Peak Performance
- Flow Rate
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Aug 94 5.4

**THE RESTEK ADVANTAGE**

**Analyze Flavor Volatiles in Alcoholic Beverages**  
Using Restek's New Silcoflex® Carbobond® Packed Columns

The RESTEK ADVANTAGE Silcoflex® Carbobond® packed columns are the perfect solution for analyzing flavor volatiles in alcoholic beverages. They offer excellent separation and resolution for a wide range of compounds.

**In this issue...**

- Silcoflex® Carbobond® Columns
- Flavor Volatiles
- Alcoholic Beverages
- Separation and Resolution
- Column Selection
- Peak Performance
- Flow Rate
- Temperature
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- Injection System
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Oct 94 5.4

**THE RESTEK ADVANTAGE**

**Analyzing Free Fatty Acids Without Derivatization**

The RESTEK ADVANTAGE columns provide the perfect solution for analyzing free fatty acids without the need for derivatization. They offer excellent separation and resolution for a wide range of fatty acids.

**In this issue...**

- Free Fatty Acids
- Without Derivatization
- Separation and Resolution
- Column Selection
- Peak Performance
- Flow Rate
- Temperature
- Detector
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Dec 94 5.6

1993

**THE RESTEK ADVANTAGE**

**Micropacked GC Columns**  
Improve the Separation of Low Molecular Weight Compounds

The RESTEK ADVANTAGE micropacked GC columns provide the perfect solution for improving the separation of low molecular weight compounds. They offer excellent separation and resolution for a wide range of compounds.

**In this issue...**

- Micropacked GC Columns
- Low Molecular Weight Compounds
- Separation and Resolution
- Column Selection
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- Flow Rate
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- Detector
- Injection System
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Jan 93 4.2

**THE RESTEK ADVANTAGE**

**ezGC™ Software**  
Simplifies GC Method Development

The RESTEK ADVANTAGE ezGC™ software simplifies GC method development. It provides a user-friendly interface for developing and optimizing GC methods.

**In this issue...**

- ezGC™ Software
- GC Method Development
- User-Friendly Interface
- Developing and Optimizing
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Mar 93 4.2

**THE RESTEK ADVANTAGE**

**A Thin Film (0.25µm) 0.53mm ID Rtx®-1701 Provides the Best Overall Resolution of Pesticides in the Least Amount of Time**

The RESTEK ADVANTAGE Rtx®-1701 column with a thin film (0.25µm) provides the best overall resolution of pesticides in the least amount of time. It offers excellent separation and resolution for a wide range of pesticides.

**In this issue...**

- Rtx®-1701 Column
- Thin Film (0.25µm)
- 0.53mm ID
- Pesticides
- Resolution
- Column Selection
- Peak Performance
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Sept 93 4.3

**THE RESTEK ADVANTAGE**

**Purge and Trap of Volatile Organics Using Narrowbore Capillary Columns**

The RESTEK ADVANTAGE narrowbore capillary columns provide the perfect solution for the purge and trap of volatile organics. They offer excellent separation and resolution for a wide range of volatile organics.

**In this issue...**

- Narrowbore Capillary Columns
- Purge and Trap
- Volatile Organics
- Separation and Resolution
- Column Selection
- Peak Performance
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Dec 93 4.4

1992

**THE RESTEK ADVANTAGE**

**Industrial Solvent Analysis Simplified**

The RESTEK ADVANTAGE columns provide the perfect solution for simplifying industrial solvent analysis. They offer excellent separation and resolution for a wide range of industrial solvents.

**In this issue...**

- Industrial Solvent Analysis
- Simplified
- Separation and Resolution
- Column Selection
- Peak Performance
- Flow Rate
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- Detector
- Injection System
- Sample Preparation
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Jan 92 3.1

**THE RESTEK ADVANTAGE**

**Rtx®-65TG Alkone Fast, Selective Analysis of Triglycerides**

The RESTEK ADVANTAGE Rtx®-65TG Alkone column provides the perfect solution for the fast, selective analysis of triglycerides. It offers excellent separation and resolution for a wide range of triglycerides.

**In this issue...**

- Rtx®-65TG Alkone Column
- Fast, Selective Analysis
- Triglycerides
- Separation and Resolution
- Column Selection
- Peak Performance
- Flow Rate
- Temperature
- Detector
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Sept 92 3.5

**THE RESTEK ADVANTAGE**

**Rtx®-624, for the Analysis of Volatile Organic Pollutants**

The RESTEK ADVANTAGE Rtx®-624 column provides the perfect solution for the analysis of volatile organic pollutants. It offers excellent separation and resolution for a wide range of volatile organic pollutants.

**In this issue...**

- Rtx®-624 Column
- Volatile Organic Pollutants
- Analysis
- Separation and Resolution
- Column Selection
- Peak Performance
- Flow Rate
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- Detector
- Injection System
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Nov 92 3.6

1991

**THE RESTEK ADVANTAGE**

**Drug Testing by Capillary GC**

The RESTEK ADVANTAGE columns provide the perfect solution for drug testing by capillary GC. They offer excellent separation and resolution for a wide range of drugs.

**In this issue...**

- Drug Testing
- Capillary GC
- Separation and Resolution
- Column Selection
- Peak Performance
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Sept 91 2.4

**THE RESTEK ADVANTAGE**

**Vu-Tight™ - A New View to Packed Column Conversion**

The RESTEK ADVANTAGE Vu-Tight™ provides a new view to packed column conversion. It offers a simple and easy way to convert packed columns to capillary columns.

**In this issue...**

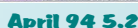
- Vu-Tight™
- Packed Column Conversion
- Simple and Easy
- Capillary Columns
- Separation and Resolution
- Column Selection
- Peak Performance
- Flow Rate
- Temperature
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- Injection System
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Nov 91 2.5

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## 1994



1991

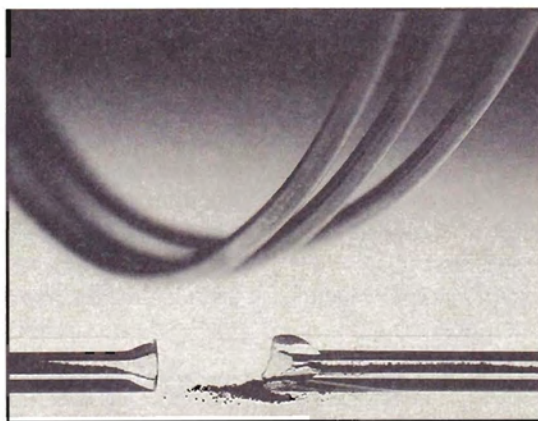


# THE RESTEK

## ADVANTAGE

### Analyze Flavor Volatiles in Alcoholic Beverages Using Restek's New Silcosteel® CarboBlack™ Packed Columns

The overall flavor and quality of alcoholic beverages is determined by the amount of acids, alcohols, and aldehydes present in distilled spirits. The Association of Official Analytical Chemists (AOAC) methods provide information on acetic acid analysis and determinations for fusel oils, methanol, ethanol, and higher alcohols.<sup>1</sup> Gas chromatography provides a rapid and simplified method to analyze all of these components simultaneously without performing any preliminary extractions. Glass packed columns are commonly used for such analyses, but they are prone to breakage and adsorption. Restek's new Silcosteel® CarboBlack™ columns are made from stainless steel coated with a deactivated fused silica inner layer, providing improved inertness, durability and flexibility compared to traditional glass packed columns. CarboBlack™ columns yield excellent separation and reproducible quantitation of the important volatile components in alcoholic beverages such as scotch whiskey and rum.



*No more mess from broken glass packed columns when you use Silcosteel® CarboBlack™ columns.*

#### Silcosteel® CarboBlack™ columns offer greater versatility over glass packed columns

Stainless steel tubing provides greater flexibility, durability, and easier handling than glass columns. These columns also offer superior column reproducibility due to more stringent tolerances for inside diameters. The typical ID tolerance for stainless steel is 0.001" compared to .0025" for glass. In addition, the ID tolerance of glass tubing is further affected during the coiling process. This is attributable to burner temperature inconsistencies and coiling speed. This results in poor column-to-column reproducibility which is not a concern with Silcosteel® tubing. The flexibility and strength of stainless steel allows columns to be coiled to a 3" diameter without fear of breakage.

Five percent Carbowax® 20M on 80/120 mesh CarboBlack™ B columns are available in 1/8" OD with a 2mm ID. A new Packed Column Inlet Adaptor Fitting is available to convert 1/4"

Five percent Carbowax® 20M on 80/120 mesh CarboBlack™ B columns are available in 1/8" OD with a 2mm ID. A new Packed Column Inlet Adaptor Fitting is available to convert 1/4"

#### *in this issue...*

Analyze Flavor Volatiles in Alcoholic Beverages  
Using Restek's New Silcosteel® CarboBlack™ Packed Columns

Detection of Antioxidants in Food by Capillary GC  
Rtx®-50 column provides excellent resolution and peak shape

Standards Spotlight  
High Concentration Composite Fuels

Aromatics in Gasoline  
New aromatic standards necessary for calibration using the proposed ASTM method

- |   |  |    |
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| 1 | New - Re-engineered Split/Splitless Injection Port for HP 5890 GCs   | 10 |
|   | <i>New injector offers several features not available with the original design</i>   |    |
| 4 | Hints for the Capillary Chromatographer  | 12 |
|   | <i>Helpful hints on using Flame Ionization Detectors</i>   |    |
| 6 | Peak Performers  | 14 |
|   | <i>Restek's new Replacement Chemical Traps for HP 5890 GCs, GC Installation Kit, mini-Lam™ Split Sleeve for HP 5890 GCs, Dual Column mini-Lam™ direct injection tee, Injector Nut for Shimadzu 17A GCs</i> |    |



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injection ports for use with this 1/8" tubing. This adaptor allows on-column injection with minimal sample backflash. The column can be installed directly into the detector port using a 1/4" to 1/8" reducing ferrule. Unlike traditional packed columns, the new CarboBlack™ columns have been pre-conditioned, eliminating the need for overnight conditioning. Only 20-30 minutes of conditioning at 170°C following column installation is required.

#### CarboBlack™ columns permit accurate acid determination

Acid concentration is indicative of maturity in distilled spirits such as Scotch whiskey. Fresh-distilled whiskey contains a low concentration of acids, whereas total acid content increases as whiskey is aged.<sup>2</sup> The predominant acid in whiskey is acetic acid. AOAC Method #945.08 recommends a titration method for acetic acid determination.<sup>1</sup> Packed column chromatography is a practical alternative since it provides accurate qualitative and quantitative results for acids. The glass wool end plugs are treated with phosphoric acid to prevent adsorption of these acidic components. To ensure minimal acid adsorption, new columns can be pre-treated by injecting several microliters of a 0.1% phosphoric acid solution. Note the good peak shape of the acetic acid in the scotch sample on the CarboBlack™ column in Figure 1.

#### Achieve excellent peak symmetry and resolution of alcohols

Percent alcohol concentration can be accurately determined on CarboBlack™ columns. Methanol, ethanol, and fusel oils (aliphatic alcohols between ethanol and isoamyl alcohol) can be analyzed for individual concentrations or for total alcohol

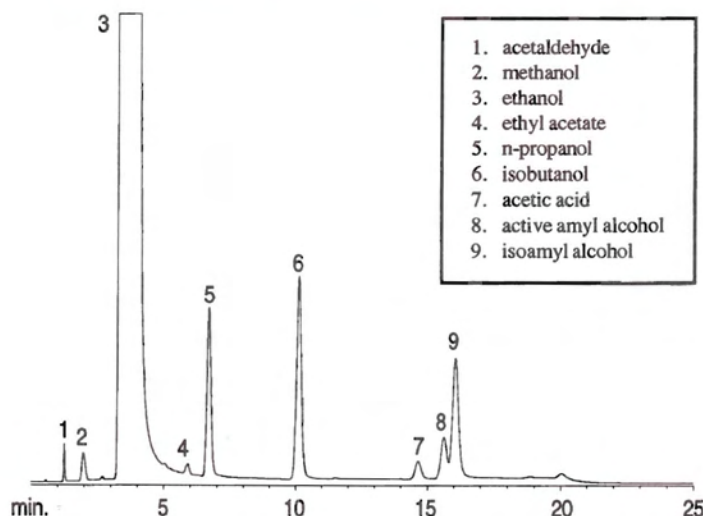
content. Ethanol has the highest concentration of the alcohols, and determines proof value. Methanol and isopropanol determination allow detection of denaturants in alcoholic beverages.<sup>3</sup> Beverage analysts often experience poor methanol peak shape. Note the excellent peak shape of methanol and its resolution from ethanol on the CarboBlack™ columns as shown in Figures 1 and 2.

The two predominate fusel oils, active-amyl and isoamyl alcohol, are difficult to separate. These compounds, along with isobutanol, predominately influence the aroma strength of the alcoholic beverages.<sup>2</sup> Resolution of these components is difficult to achieve even with capillary columns. CarboBlack™ packed columns yield 60% resolution between active-amyl and isoamyl alcohols as shown in Figures 1 and 2.

#### Accurately determine carbonyl concentration in alcoholic beverages

Recognition of aldehydes, ketones, and esters is important in analyses of distilled and fermented beverages. Acetaldehyde, the predominate aldehyde in most alcoholic beverages, exhibits a pungent odor that is softened by acetal formation during the maturation process.<sup>2</sup> Ethyl formate and ethyl acetate concentrations increase consistently with age and are reliable markers for age determination, especially of whiskeys. Eight-year-old Bourbon contains about 19 times more ethyl acetate and 10 times more ethyl formate as unaged Bourbon.<sup>3</sup> Acetone determination detects any adulteration or contamination present.<sup>3</sup> All of these carbonyl compounds in distilled spirits are completely resolved on a CarboBlack™ column, as shown in Figure 2.

**Figure 1 - Unbreakable Silcosteel® CarboBlack™ B packed columns provide excellent resolution of Scotch whiskey.**



2m, 1/8" OD x 2mm ID, 5% Carbowax 20M 80/120 CarboBlack™ B column (cat.# 80105)

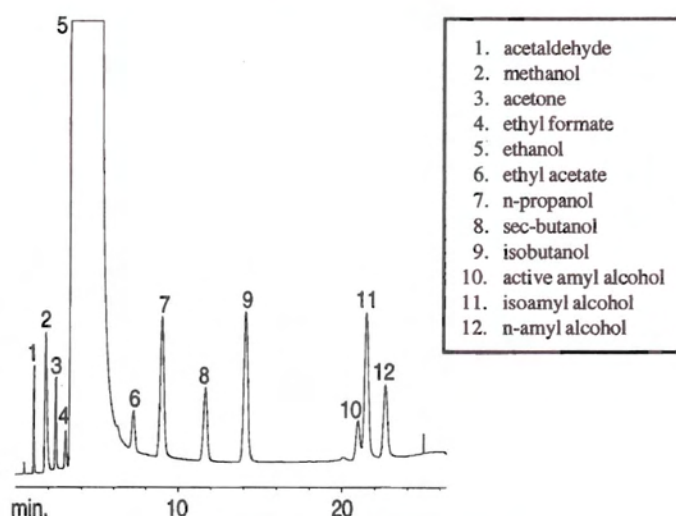
0.5µl on-column injection of Scotch, on-column concentration: neat

Oven temp.: 70°C to 150°C @ 4°C/min.

Inj./det. temp.: 200°C/250°C Detector: FID

Carrier gas: nitrogen Column flow rate: 20ml/min.

**Figures 2 - CarboBlack™ B columns resolve important flavor volatiles in fresh-distilled and aged rum.**



2m, 1/8" OD x 2mm ID, 5% Carbowax 20M 80/120 CarboBlack™ B column (cat.# 80105)

0.5µl on-column injection of fusel oils in rum, on-column concentration: neat

Oven temp.: 65°C (hold 5 min.) to 150°C @ 4°C/min.

Inj./det. temp.: 200°C/250°C Detector: FID

Carrier gas: nitrogen Column flow rate: 20ml/min.



The new Silcosteel® CarboBlack™ columns can be used to effectively analyze the important flavor volatiles in distilled spirits such as scotch and rum. All components are resolved to provide accurate and reproducible quantitation. Even active compounds such as alcohols and acetic acid exhibit excellent response. These new columns provide more versatility over glass packed columns traditionally used for alcoholic beverage analysis. Stainless steel tubing provides easier handling, eliminates breakage, and offers greater precision of column

dimensions. The fused silica inner layer ensures inertness. Flexibility of stainless steel allows these packed columns to be wound to smaller diameters to accommodate ovens of various sizes. A new adaptor allows for on-column injection into any 1/4" injection port.

## References

1. AOAC, *Official Book of Methods of AOAC*, 15th ed., 1990.
2. Suomalainen, H. and L. Nykäne, "Composition of Whiskey Flavour", *Process Biochemistry*, July, 1970.
3. Deman, *Principles of Food Chemistry*, 1990. pp. 48-50.

## CarboBlack™ Packed Columns Product Listing

To order, specify Restek catalog number and instrument configuration. For example, a 2m, 1/8" OD, 2mm ID 1% Rt-1000 on 60/80 CarboBlack™ B, used in an HP 5890 GC would be part number: 80207-810.

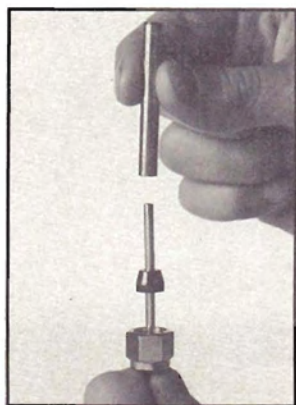
Phase	Packing	Length	OD	ID	Catalog #	Application
5% Carbowax® 20M	80/120 CarboBlack™ B	2m	1/8"	2mm	80105-???	Flavor volatiles in alcoholic beverages
5% Carbowax® 20M	60/80 CarboBlack™ B	1.8m	1/8"	2mm	80106-???	Blood alcohols
6.6% Carbowax® 20M	80/120 CarboBlack™ B	2m	1/8"	2mm	80107-???	Alcoholic beverages & fermentation components
0.3% Carbowax® 20M, 0.1% H3PO4	60/80 CarboBlack™ C	0.75m	3/16"	4mm	80111-???	Free acids
4% Carbowax® 20M, 0.8% KOH	60/80 CarboBlack™ B	2m	1/8"	2mm	80116-???	Amines
0.2% Carbowax® 1500	60/80 CarboBlack™ C	2m	1/8"	2mm	80121-???	Alcohols, esters, ketones
0.2% Carbowax® 1500	80/100 CarboBlack™ C	2m	1/8"	2mm	80122-???	Alcohols, esters, ketones
0.1% Rt-1000	80/100 CarboBlack™ C	1.8m	1/8"	2mm	80205-???	Phenols, solvents
1% Rt-1000	60/80 CarboBlack™ B	2.4m	1/8"	2mm	80206-???	EPA 601
1% Rt-1000	60/80 CarboBlack™ B	2m	1/8"	2mm	80207-???	EPA 624
3% Rt-1500	80/120 CarboBlack™ B	3.05m	1/8"	2mm	80211-???	Solvents
1% Rt-1510	60/80 CarboBlack™ B	3.05m	1/8"	2mm	80216-???	Solvents
1.5% XE 60, 1% H3PO4	60/80 CarboBlack™ B	1.8m	1/8"	2mm	80305-???	Sulfur compounds
0.19% Picric Acid	80/100 CarboBlack™ C	2m	1/8"	2mm	80311-???	C4 unsaturates

### Instrument Configuration

General Configuration, fits most GCs	-800
HP 5880, 5890, 5987	-810
Varian 3700, Vista series, FID	-820
Perkin Elmer 900-3920, Sigma 1,2,3	-830
(other configurations available, please call your local distributor)	

### Restek Part #

\*Restek's CarboBlack™ B is equivalent to Carbowax™ B and Carbograph™ 1. Restek's CarboBlack™ C is equivalent to Carbowax™ C and Carbograph™ 2.



## Packed Column Inlet Adapter Fittings

Many GCs are shipped with an adapter sleeve to allow 1/8" or 3/16" columns to be used in 1/4" injection ports for on-column analysis. This inlet adapter sleeve is bulky and adds mass to the injection port. Restek's low mass inlet adapters fit over the top of a 1/8" or 3/16" Silcosteel® packed column to center the column perfectly in a 1/4" injection port. A slot is positioned at the top of the adapter to prevent carrier gas flow occlusion and a chamfered guide directs the syringe needle to the center of the packed column. The adapter fitting seals at the base of the 1/4" injection port using a 1/4" to 1/8" or 1/4" to 3/16" reducing ferrule (supplied). A reducing ferrule is provided for column installation into the detector.

### Packed Column Inlet Adapter Fitting:

- (1/4" to 1/8"): cat.# 21651
- (1/4" to 3/16"): cat.# 21650

### 1/4" to 1/8" Reducing Ferrules:

- Vespel®/graphite: cat.# 20222, 10-pk.
- Graphite: cat.# 20225, 10-pk.



# Detection of Antioxidants in Food by Capillary GC

Foods containing fats and oils are prone to lipid oxidation which promotes off-flavors and limits shelf-life. In order to inhibit the oxidation process, food preservatives, or antioxidants, are often added. Antioxidant food additives retard oxidative rancidity caused by the atmosphere and delay discoloration in meats, meat products, and fruits and vegetables. Commonly used antioxidants include phenolic compounds such as BHA(butylated hydroxy anisole), BHT(butylated hydroxy toluene), PG(propyl gallate), and TBHQ(tert-butyl-hydroquinone). Recent attention has focused on "natural" antioxidants such as tocopherols and tocotrienols. These antioxidants possess Vitamin E activity and are currently very popular for their dual function in both food and health preservation.

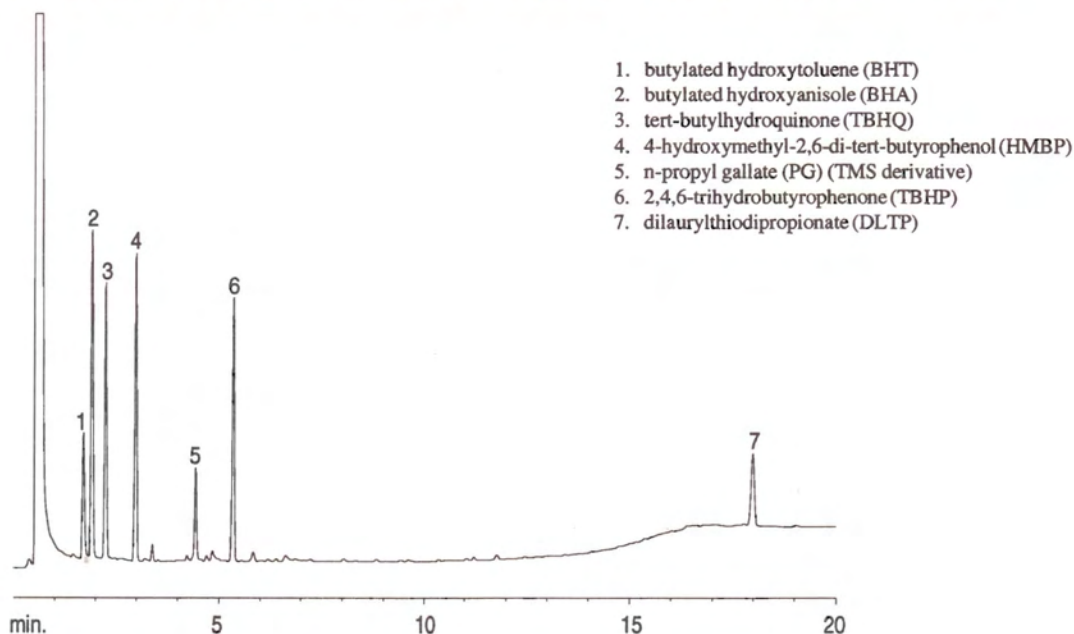
Capillary GC is often used for food antioxidants analysis since capillary columns provide rapid, accurate detection. Methods typically recommend non-polar columns such as the Rtx®-1 or Rtx®-5. However, when analyzing several different antioxidants simultaneously, coelutions can occur when using non-polar columns. By using an intermediate polarity column, coelution problems can be eliminated and excellent resolution achieved. The selectivity and thermal stability of the Rtx®-50 column provides an ideal choice for the analysis of antioxidants, providing excellent resolution and peak shape.

## Synthetic Antioxidants

Primary antioxidants such as BHA, BHT, TBHQ, and PG, terminate the free radical chains susceptible to lipid oxidation. Secondary antioxidants such as DLTP (dilauryl thiopropionate) function by decomposing the lipid hydroperoxides into stable end products.<sup>1</sup> The United States Food and Drug Administration (FDA) has specific regulations on phenolic antioxidant addition, because many, like BHT, are toxic above certain levels. The GRAS (Generally Recognized As Safe) limit for direct addition to food is 0.02% (200ppm), based on the fat content of the food.<sup>1</sup> If added to food packaging, which is an indirect addition to food, the maximum allowable limit is 50ppm or 0.005% in the food item.<sup>1</sup> This limit applies to a maximum concentration allowable for antioxidants used alone or a total concentration for combinations of antioxidants. As mandated by the FDA, antioxidant concentration levels in food samples must be monitored to ensure such regulatory standards.

Several methods have been developed for the analysis of regulated antioxidants in food.<sup>1,2,3</sup> Although analysis of primary or secondary antioxidants can be performed on non-polar columns, simultaneous determination requires the selectivity of a more polar column. Figure 1 shows the analysis of seven regulated antioxidants, including silylated propyl

**Figure 1 - The Rtx®-50 capillary column provides rapid analysis of 7 FDA-regulated food antioxidants.**



30m, 0.53mm ID, 0.50µm Rtx®-50 (cat.# 10540)  
1.0µl direct injection of 7 regulated food antioxidants using a Uniliner®  
on-column concentration: 100ppm  
Oven temp.: 165°C (hold 1 min.) to 310°C @ 10°C/min. (hold 10 min.)  
Inj./det. temp.: 290°C/310°C Detector: FID  
Carrier gas: helium Linear velocity: 89cm/sec. set @ 100°C  
Detector sensitivity: 8 x 10<sup>-11</sup> AFS Column flow: 11.7cc/min.



gallate on an Rtx®-50 column. The GRAS limits can be easily achieved on the Rtx®-50 by using the direct injection mode. Baseline resolution of all components is achieved in less than 20 minutes due to the 310°C maximum temperature limit of the Rtx®-50 column.

### Tocopherol Antioxidants

Tocopherols are primary antioxidants that quench free radicals created by oxidation of unsaturated bonds in fats.<sup>4</sup> They can be extracted from natural sources such as nuts, seed oils, and soybeans, or created synthetically. Alpha-tocopherol has the most biological potency, however,  $\delta$ -tocopherols have the most antioxidant potency. Therefore, concentrates used as food antioxidants contain high levels of  $\gamma$ - and  $\delta$ -tocopherols, and smaller amounts of  $\alpha$ -tocopherol.<sup>4</sup> Usage levels of mixed tocopherols range from 0.015% to 0.045% based on oil or fat content.<sup>5</sup> The use of mixed tocopherols as antioxidants are generally regarded as safe by the FDA.<sup>6</sup> Tocopheryl acetate, a stable form of Vitamin E, is also added to food products. Although tocopheryl acetate is not an antioxidant itself, in an acidic environment, it slowly hydrolyzes and tocopherol is released.<sup>4</sup>

Because tocopherols are found in a variety of sample matrices, labs may be required to perform sample preparation including saponification, solvent extraction, and silylation. Tocopherols are commonly analyzed as derivatives, but can also be chromatographed in their free form. Figure 2 shows the analysis

of underivatized  $\alpha$ -,  $\gamma$ -, and  $\delta$ -tocopherols,  $\gamma$ -tocotrienol and tocopheryl acetate on a 30 meter, 0.53mm ID, 0.5 $\mu$ m Rtx®-50 column. All components exhibit good peak shape and are baseline resolved in less than 20 minutes.

The Rtx®-50 is an excellent choice for phenolic and tocopherol antioxidants. The inertness and high thermal stability of the Rtx®-50 permits all components to be analyzed, with the exception of propyl gallate, in their free form. All components are well resolved to provide qualitative and quantitative accuracy. The 310°C maximum operating temperature of the Rtx®-50 column reduces analysis time while maintaining a stable baseline during temperature programming.

### References

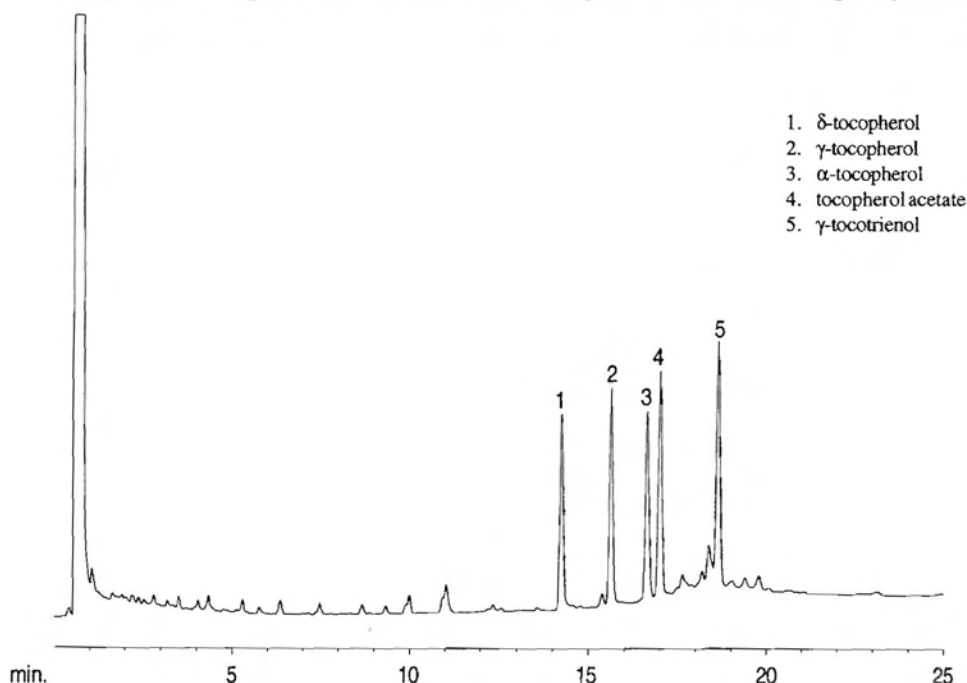
- 1) AOAC, *Food Additives Analytical Manual: Volumes I and II*, 1992.
- 2) AOAC, *Official Methods of Analysis, Volumes I and II*, 1990.
- 3) AOCS, *Official Methods and Recommended Practices*, 1994.
- 4) Hudson, B.F.J., *Food Antioxidants*, 1990.
- 5) Han, D., O.S. Yi and H.K. Shin, *Journal of Food Science*, Vol. 55, pp.247, 1990.
- 6) Food and Drug Administration proposal 184.1894, Oct. 27, 1978.

## Product Listing

### Rtx®-50

30 meter, 0.53mm ID, 0.50 $\mu$ m  
cat.# 10540

**Figure 2 - Tocopherol antioxidants can be analyzed on the Rtx®-50 capillary column.**



1.  $\delta$ -tocopherol
2.  $\gamma$ -tocopherol
3.  $\alpha$ -tocopherol
4. tocopheryl acetate
5.  $\gamma$ -tocotrienol

30m, 0.53mm ID, 0.50 $\mu$ m Rtx®-50 (cat.# 10540)

1.0 $\mu$ l direct injection of Vitamin E food antioxidants

on-column concentration: 50ppm

Oven temp.: 200°C to 290°C @ 5°C/min. (hold 15 min.)

Inj./det. temp.: 290°C/310°C Detector: FID

Carrier gas: helium Linear velocity: 89cm/sec. set @ 100°C

Detector sensitivity: 8 x 10<sup>-11</sup> AFS Column flow: 11.7cc/min.



# Aromatics in Reformulated Gasoline by GC/MS

The US Clean Air Act amendments of 1990 require that after 1994 only reformulated gasoline be sold in certain areas which have not attained specified ozone levels. The reformulation of gasoline is designed to reduce air pollution by lowering the emission of volatile organic compounds (VOCs), nitrogen oxides ( $\text{NO}_x$ ) and toxic compounds. In order to meet the reduction of air pollution, a new set of specifications<sup>1,2</sup> will go into effect on January 1, 1995 setting limits on the volatility and composition of gasoline. As part of these new regulations the Environmental Protection Agency (EPA) has specified that GC/MS be used for the determination of total aromatics in gasoline. In addition, this test method can also be used to measure the volume % of benzene as an alternative to ASTM test method D3606.

In order to meet the EPA requirements, the American Society of Testing and Materials Committee on Petroleum Products and Lubricants (ASTM D2) is developing a standard test method, which is currently under evaluation by an intralaboratory cooperative test program. In addition to a GC/MS and column, the procedure requires specific mixtures of aromatic hydrocarbons for qualitative and quantitative calibration of the system prior to sample analysis. It would be time consuming and expensive if each laboratory had to obtain the 28 individual hydrocarbons, perform purity analyses on each, and prepare two sets of the 5 calibration levels. Restek now offers kits containing the aromatic standards necessary for calibration using the proposed ASTM method. These standards undergo extensive quality control to ensure the purity of the starting materials and the accuracy of the final mixtures.

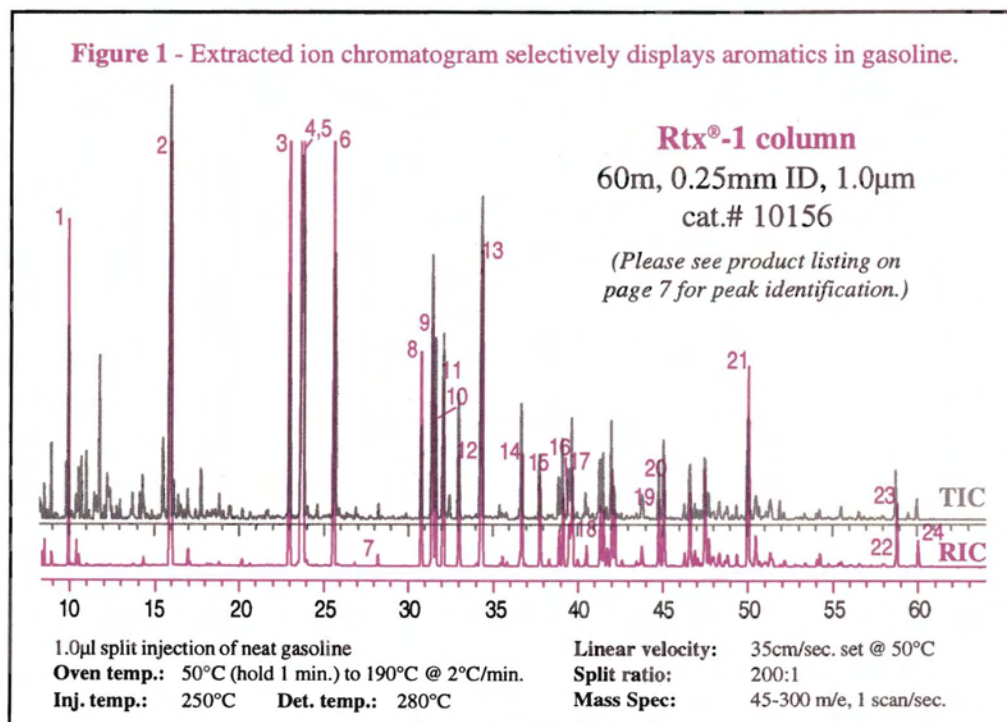
## Peaks are Integrated Using Ions Specific for Aromatic Hydrocarbons

The ASTM proposed method specifies the analysis of gasoline using a 60 meter methyl silicone capillary column and a quadrupole GC/MS system operated in the scan mode from 45-300 dalton. Either a split injection with direct interface to the GC/MS or a cool on-column injection with an open split interface may be used. For the purpose of this study, the split injector was selected with a 60m, 0.25mm ID, 1.0 $\mu\text{m}$  Rtx®-1 column. Figure 1 was obtained from the analysis of a gasoline sample, showing both the total ion chromatogram (TIC) and a reconstructed chromatogram (RIC) with specific ions for aromatics extracted. The 60 meter column provides resolution of the aromatic hydrocarbons in the complex gasoline sample. Aromatics are identified, with the aid of the mass spectra, and appropriate response factors are applied. The Mass % of each aromatic detected is calculated using the internal standard technique and the individual amounts are summed to report a total aromatic concentration.

## Pure Calibration Materials for Quantitative Accuracy

Since the proposed test method will be used by refineries and regulators to determine the aromatic concentration in gasoline, it is crucial that the method be accurate. Response factor calibration, using an accurate standard, is a critical part of the analytical procedure. However, before a calibration mixture can be prepared, the chemical purity of the aromatic components must be determined. Restek uses several complimentary analytical techniques including gas chromatography with flame ionization detection (GC/FID), GC/MS, refractive index (RI), melting point (T<sub>mp</sub>) and differential scanning calorimetry (DSC) to determine the identity and purity of raw materials used in standards preparation.

Gas chromatography is a valuable tool for calculating an aromatic compound's purity, but only if the column and conditions are optimized to resolve possible organic impurities. All volatile hydrocarbons in Restek's reformulated gasoline standard mixes are analyzed using a 105m, 0.53mm ID, 3.0 $\mu\text{m}$  Rtx®-502.2 column (cat.# 10910), which has good selectivity for resolving aromatics and high capacity for the major component. A second GC/FID analysis for substituted benzenes is also performed using a more polar Stabilwax® column which gives better resolution of possible coeluting isomers. GC/MS analysis is also done on all raw materials to confirm identity in case of mislabeling.



1. 40 CFR Part 80, *Federal Register*, 59(32):7716-7878, Feb.16, 1994.

2. W.H. Keesom, M.J. Humbach; "Effective Gasoline Reformulation", *Standardization News*, 22 (6) June 1994.



Refractive index provides a complementary qualitative technique and is especially informative when combined with GC/MS and DSC. The melting point of an organic compound provides another qualitative analysis in addition to RI and MS but most importantly the presence of water and inorganic impurities can be determined<sup>3</sup>. DSC provides a very powerful tool for purity analysis because it measures impurities which are not detectable by GC. DSC analysis was particularly helpful for the analysis of 1,2-diethyl benzene containing an impurity of 1,4-diethyl benzene. These compounds have boiling points which differ by only 0.4°C and are therefore difficult to purify, but the melting points differ by 12°C. In this case, DSC provides a better qualitative identification than GC/MS, because the isomers have similar spectra and retention times. All of these individual tests are important quality control checks on the raw materials because it is very difficult to determine the purity of the mixture after the standard has been prepared.

### Construct a 5 Point Calibration Curve with Convenient Standards Kits

Two sets of calibration blends are available containing the aromatics specified in the ASTM proposed test method. The appropriate internal standards are included in the mixtures for calculation of relative response factors. Each kit contains five ampuls of standards at the correct concentration to construct a five point calibration curve and complete the calibration of the GC/MS system. A data pack containing complete quality assurance documentation is available for each of these standards. The data pack includes the raw material purities by each of the tests mentioned, gravimetric amounts determined by NIST traceable balance, and quantitative verification by GC/MS. In addition to these kits, individual standards at each concentration level are also available.

3. ASTM Test Method E-928 "Mol Percent Purity by Differential Scanning Calorimetry" *ASTM Book of Standards* Vol14.02.

## ASTM/EPA Aromatics Standards Product Listing

Peak #	Compound	Mix #1	Mix #2	Mix #3	Mix #4	Mix #5
1	benzene	30mg/ml	15mg/ml	10mg/ml	5mg/ml	1mg/ml
2	toluene	150	100	50	30	10
3	ethylbenzene	50	25	10	5	1
4	m-xylene	60	30	10	5	1
5	p-xylene	60	30	10	5	1
6	o-xylene	60	30	10	5	1
IS1	benzene-d6	20	20	20	20	20
IS2	ethylbenzene-d10	20	20	20	20	20
<b>Cat. #</b>						
	single ampul	36200	36201	36202	36203	36204
	single ampul w/dp	36200-500	36201-500	36202-500	36203-500	36204-500
	5-pack	36200-510	36201-510	36202-510	36203-510	36204-510
	5-pack w/dp	36200-520	36201-520	36202-520	36203-520	36204-520
	10-pack w/dp	36300	36301	36302	36303	36304

**Complete ASTM/EPA Aromatics Set #1 Kit (1ml each of above 5 mixes):** 36205, per kit      36205-500, per kit with data pack

Peak #	Compound	Mix #1	Mix #2	Mix #3	Mix #4	Mix #5
7	isopropylbenzene	30	15	10	5	1
8	n-propylbenzene	30	15	10	5	1
9	1-methyl-3-ethylbenzene	30	15	10	5	1
10	1-methyl-4-ethylbenzene	30	15	10	5	1
11	1,3,5-trimethylbenzene	30	15	10	5	1
12	1-methyl-2-ethylbenzene	30	15	10	5	1
13	1,2,4-trimethylbenzene	50	25	10	5	1
14	1,2,3-trimethylbenzene	30	15	10	5	1
15	indan	30	15	10	5	1
16	1,4-diethylbenzene	30	15	10	5	1
17	butylbenzene	30	15	10	5	1
18	1,2-diethylbenzene	30	15	10	5	1
19	1,2,4,5-tetramethylbenzene	20	10	5	2.5	1
20	1,2,3,5-tetramethylbenzene	20	10	5	2.5	1
21	pentamethylbenzene	20	10	5	2.5	1
22	naphthalene	20	10	5	2.5	1
23	2-methylnaphthalene	20	10	5	2.5	1
24	1-methylnaphthalene	20	10	5	2.5	1
IS2	ethylbenzene-d10	20	20	20	20	20
IS3	naphthalene-d8	10	10	10	10	10
<b>Cat. #</b>						
	single ampul	36206	36207	36208	36209	36210
	single ampul w/dp	36206-500	36207-500	36208-500	36209-500	36210-500
	5-pack	36206-510	36207-510	36208-510	36209-510	36210-510
	5-pack w/dp	36206-520	36207-520	36208-520	36209-520	36210-520
	10-pack w/dp	36306	36307	36308	36309	36310

**Complete ASTM/EPA Aromatics Set #2 Kit (1ml each of above 5 mixes):** 36211, per kit      36211-500, per kit with data pack



# Standards Spotlight



## XHc Underground Storage Tank Monitoring Standards and JP-8 Military Fuel Standards

- Suitable for matrix spiking & recovery studies • Highest concentration commercially available • Full data pack for audit compliance • Two convenient volumes for every product • Produced under an ISO 9001 registered quality system •

Restek is pleased to introduce the highest concentration underground storage tank monitoring standards available today - our XHc (eXtra High Concentration) product line. These standards are prepared at a concentration of 50,000µg/ml in a solvent suitable for the type of analysis being performed.

Each standard is packaged in either 1ml or 5ml volumes. The mixtures are placed in an amber ampul, purged with nitrogen, and flame sealed for extended shelf life. The standards are shipped with an ampul breaker and extra label for your convenience. In addition, the 1ml ampuls are that supplied are silane treated and come with a screw cap vial for storage of any unused standard.

All the products are available with a full data pack for audit compliance. Each data pack contains detailed information on product manufacture and testing to easily pass the most stringent quality audit a laboratory might encounter. Our customer choice packaging offers either a 10% discount or a FREE data pack for 5-pack purchases, and a 10% discount PLUS FREE data pack for 10-pack purchases.

### XHc Unleaded Gasoline Composite Standard

50,000µg/ml in purge & trap grade methanol.

#### Packaged 1ml per ampul:

cat.# 30205	each
30205-500	each w/data pack
30205-510	5-pack
30205-520	5-pk. w/data pack
30305	10-pk. w/data pack

#### Packaged 5ml per ampul:

cat.# 30206	each
30206-500	each w/data pack
30206-510	5-pack
30206-520	5-pack w/data pack
30306	10-pack w/data pack

### XHc Aviation Gas Standard

50,000µg/ml in purge & trap grade methanol.

#### Packaged 1ml per ampul:

cat.# 30207	each
30207-500	each w/data pack
30207-510	5-pack
30207-520	5-pack w/data pack
30307	10-pack w/data pack

#### Packaged 5ml per ampul:

cat.# 30208	each
30208-500	each w/data pack
30208-510	5-pack
30208-520	5-pack w/data pack
30308	10-pack w/data pack

### XHc Jet Fuel A Standard

50,000µg/ml in methylene chloride.

#### Packaged 1ml per ampul:

cat.# 31242	each
31242-500	each w/data pack
31242-510	5-pack
31242-520	5-pack w/data pack
31342	10-pack w/data pack

#### Packaged 5ml per ampul:

cat.# 31243	each
31243-500	each w/data pack
31243-510	5-pack
31243-520	5-pack w/data pack
31343	10-pack w/data pack

### XHc Fuel Oil #4 Standard

50,000µg/ml in methylene chloride

#### Packaged 1ml per ampul:

cat.# 31244	each
31244-500	each w/data pack
31244-510	5-pack
31244-520	5-pack w/data pack
31344	10-pack w/data pack

#### Packaged 5ml per ampul:

cat.# 31245	each
31245-500	each w/data pack
31245-510	5-pack
31245-520	5-pack w/data pack
31345	10-pack w/data pack

### XHc Fuel Oil #5 Standard

50,000µg/ml in methylene chloride

#### Packaged 1ml per ampul:

cat.# 31246	each
31246-500	each w/data pack
31246-510	5-pack
31246-520	5-pack w/data pack
31346	10-pack w/data pack

#### Packaged 5ml per ampul:

cat.# 31247	each
31247-500	each w/data pack
31247-510	5-pack
31247-520	5-pack w/data pack
31347	10-pack w/data pack

### XHc Fuel Oil #6 Standard

50,000µg/ml in methylene chloride

#### Packaged 1ml per ampul:

cat.# 31248	each
31248-500	each w/data pack
31248-510	5-pack
31248-520	5-pack w/data pack
31348	10-pack w/data pack

#### Packaged 5ml per ampul:

cat.# 31249	each
31249-500	each w/data pack
31249-510	5-pack
31249-520	5-pack w/data pack
31349	10-pack w/data pack



**XHc JP-4 Standard**

50,000µg/ml in methylene chloride

**Packaged 1ml per ampul:**

cat.# 31250	each
31250-500	each w/data pack
31250-510	5-pack
31250-520	5-pack w/data pack
31350	10-pack w/data pack

**Packaged 5ml per ampul:**

cat.# 31251	each
31251-500	each w/data pack
31251-510	5-pack
31251-520	5-pack w/data pack
31351	10-pack w/data pack

**XHc JP-5 Standard**

50,000µg/ml in methylene chloride

**Packaged 1ml per ampul:**

cat.# 31252	each
31252-500	each w/data pack
31252-510	5-pack
31252-520	5-pack w/data pack
31352	10-pack w/data pack

**Packaged 5ml per ampul:**

cat.# 31253	each
31253-500	each w/data pack
31253-510	5-pack
31253-520	5-pack w/data pack
31353	10-pack w/data pack

**XHc JP-8 Standard**

50,000µg/ml in methylene chloride

**Packaged 1ml per ampul:**

cat.# 31254	each
31254-500	each w/data pack
31254-510	5-pack
31254-520	5-pack w/data pack
31354	10-pack w/data pack

**XHc JP-8 Standard (cont.)****Packaged 5ml per ampul:**

cat.# 31255	each
31255-500	each w/data pack
31255-510	5-pack
31255-520	5-pack w/data pack
31355	10-pack w/data pack

**XHc Kerosene Composite Standard**

50,000µg/ml in methylene chloride

**Packaged 1ml per ampul:**

cat.# 31256	each
31256-500	each w/data pack
31256-510	5-pack
31256-520	5-pack w/data pack
31356	10-pack w/data pack

**Packaged 5ml per ampul:**

cat.# 31257	each
31257-500	each w/data pack
31257-510	5-pack
31257-520	5-pack w/data pack
31357	10-pack w/data pack

**XHc Diesel Fuel #2 Composite Standard**

50,000µg/ml in methylene chloride

**Packaged 1ml per ampul:**

cat.# 31258	each
31258-500	each w/data pack
31258-510	5-pack
31258-520	5-pack w/data pack
31358	10-pack w/data pack

**Packaged 5ml per ampul:**

cat.# 31259	each
31259-500	each w/data pack
31259-510	5-pack
31259-520	5-pack w/data pack
31359	10-pack w/data pack

**XHc Mineral Spirits Standard**

50,000µg/ml in methylene chloride

**Packaged 1ml per ampul:**

cat.# 31260	each
31260-500	each w/data pack
31260-510	5-pack
31260-520	5-pack w/data pack
31360	10-pack w/data pack

**Packaged 5ml per ampul:**

cat.# 31261	each
31261-500	each w/data pack
31261-510	5-pack
31261-520	5-pack w/data pack
31361	10-pack w/data pack

**JP-8 Standard**

5000µg/ml in 1ml methylene chloride, 1ml per ampul.

cat.# 31262	each
31262-500	each w/data pack
31262-510	5-pack
31262-520	5-pack w/data pack
31362	10-pack w/data pack

**Restek chemical  
standards are  
produced under an  
ISO 9001 registered  
quality system!**

**Additional EPA Drinking Water Monitoring Standards**

The EPA is now requiring laboratories to monitor and report three additional compounds in drinking water. These compounds are: methylene chloride, 1,1,2-trichloroethane, and 1,2,4-trichlorobenzene. To meet these requirements, Restek is offering an additional calibration mixture and revised kit for drinking water analysis. See Restek's *Chromatography Products 1994/95* catalog (page 43) for a complete description of the DW-VOC Mix #1 & #2, plus Trihalomethane Mix.

**DW-VOC Mix #3**

200µg/ml each in purge & trap grade methanol, 1ml per ampul.

methylene chloride  
1,1,2-trichloroethane  
1,2,4-trichlorobenzene

cat.# 30209	each
30209-500	each w/data pack
30209-510	5-pack
30209-520	5-pack w/data pack
30309	10-pack w/data pack

**Revised DW-VOC Kit**

Contains 1ml each of these products:

501 Trihalomethane Mix (cat.# 30036)  
DW-VOC Mix #1 (cat.# 30037)  
DW-VOC Mix #2 (cat.# 30038)  
DW-VOC Mix #3 (cat.# 30209)

cat.# 30210	each kit
30210-500	each kit w/data pack



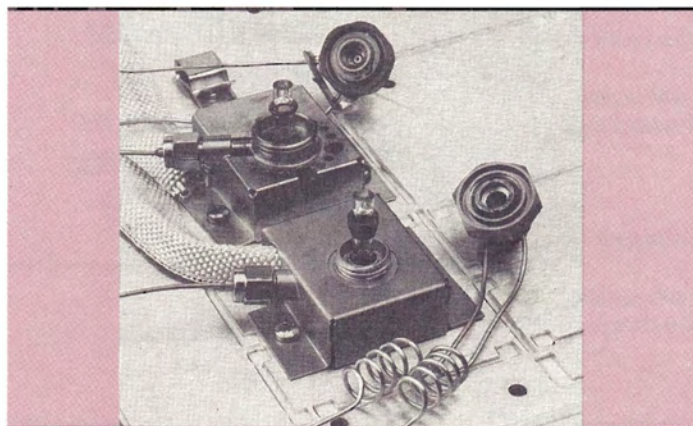
# New - Restek's Split/Splitless Injection Port for HP 5890 GCs

After many years of using HP 5890 GCs in our Applications, R&D, and Quality Assurance labs and getting feedback from our customers, Restek has re-engineered the HP split/splitless injector. Our new injector offers several features not available with the original design.

## Improved Split/splitless Weldment Assembly

- Split/splitless weldment re-design allows the use of 1/4" type graphite or Vespel®/graphite ferrules instead of rubber or graphite o-rings.\*
- Locking pin and slot assembly eliminates the need for a metal bracket to prevent the inlet lines from snapping.
- Narrow weldment recess prevents trapped septa particles.

**Figure 1 - Restek's injection port seals the sleeve tightly with a Swagelok®-type 1/4" ferrule eliminating rubber o-ring melt down.**



The o-ring that seals the sleeve in the top of the injector is a potential source for leaks. Although rubber o-rings seal well at temperatures below 200°C, they can become brittle and leak when operated for prolonged periods above 200°C. Graphite o-rings can handle higher temperatures, but do not seal as easily as rubber o-rings. We experimented with various designs and determined that a 1/4" type ferrule, installed tapered end down, gave the most consistent, leak-free results. Our new design accepts either Vespel® or graphite ferrules and delivers a leak-free seal, even at injection port temperatures as high as 400°C. The photo in Figure 1 illustrates the taper inside the injection port weldment utilizing standard ferrules to ensure a leak tight seal.\*

The original equipment incorporates a metal bracket that locks into a slot on the top of the injection port. This design prevents the assembly from spinning and breaking off the inlet gas lines. Our new design incorporates a pin and slot assembly that

prevents the split/splitless weldment from spinning and eliminates the bulky metal bracket assembly. The original split/splitless weldment incorporates a multi-piece assembly that permits a large recess between the top and bottom orifice. Septum particles can be trapped in this recess, causing ghost peaks and baseline disturbances. Restek's new design significantly reduces this recess and eliminates trapped septa particles.

## Re-designed Injector Base Improves Seal and Simplifies Column Installation

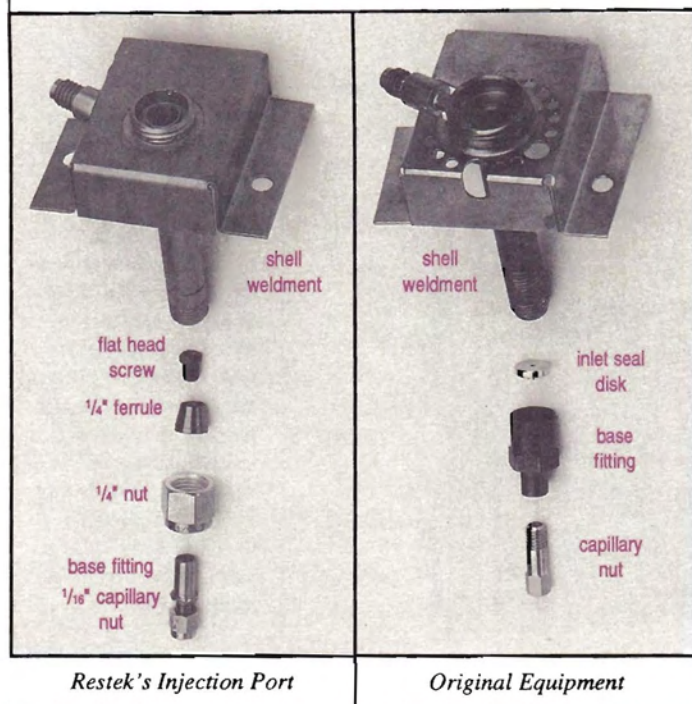
- Base inlet sealing disk eliminated and replaced with a standard 1/4" ferrule.
- Sleeve supported with an easy to remove, inexpensive slotted flat head stainless steel screw.
- Base fitting designed to use standard 1/16" Swagelok®-type ferrules.
- Base fitting protrudes from the insulated cup making column installation easier.
- Original sleeve dimensions and column insertion distances maintained (from back of nut).

In the original equipment injector design, the sleeve is supported in the injector by a metal inlet seal that sits inside of the injector fitting. When the injector nut is tightened, a knife edge at the base of the injection port cuts into the metal inlet seal and creates a seal by compressing metal against metal. The inlet seals require frequent replacement due to sample contamination and cannot be re-used, otherwise a leak will occur. Restek has re-designed the base of the injection port to accept a Swagelok®-type ferrule to seal the injector fitting to the injector shell weldment. An inexpensive slotted, stainless steel flat head screw fits into the base fitting, supporting the sleeve in the injection port and maintaining the same flow path as the original design. This screw can easily be cleaned and re-installed without fear of leakage and is inexpensive enough to replace when severely contaminated. Both gold plated and Silcosteel® versions are available to minimize decomposition of active compounds. Figure 2 shows a comparison between the original equipment design and Restek's design. Restek's design eliminates the inlet seal while maintaining the original column insertion distances from the back of HP's standard capillary inlet nut. The injector fitting is designed to seal with standard 1/16" graphite or Vespel® capillary ferrules with the threads protruding from the insulated cover. This allows visual confirmation of column installation.

*\*Standard 1/4" ID graphite and Vespel®/graphite ferrules fit over split sleeves (OD ~ 6.35mm) but a slightly enlarged ferrule ID is required for splitless sleeves (OD 6.5mm) and is available from Restek.*

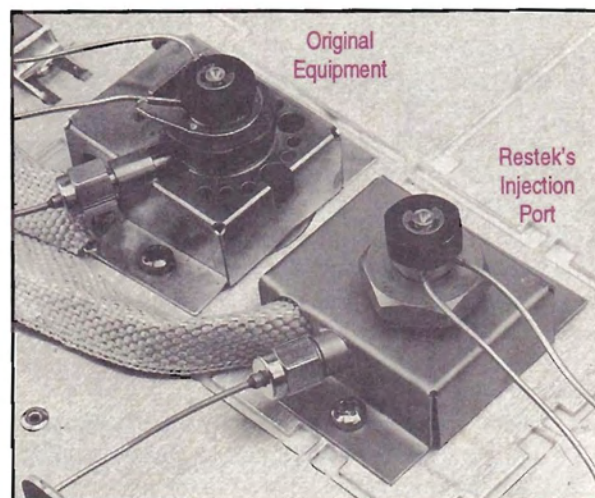


**Figure 2 -** The original equipment design requires a metal inlet seal which presses against a knife edge at the base of the injector shell weldment. Restek's design seals the inlet base fitting with a standard 1/4" Swagelok®-type ferrule and utilizes an inexpensive flat head machine screw to achieve the same flow profile at the base of the injector.



#### Restek's Split/Splitless Injection Port for HP 5890 GCs:

- Swagelok®-type ferrules seal around sleeve.
- Narrower needle gap eliminates trapped septum particles.
- Locking pin guide.
- Standard 1/4" ferrule used instead of sealing disk.
- Easily removable and re-useable slotted base screw utilized to maintain flow profile.
- Original sleeve dimensions and column insertion distances maintained (26mm from back of nut).



### Product Listing

Product	Quantity	Cat.#
<b>Complete Injection Port Assembly includes*:</b> base fitting, split/splitless weldment, shell weldment, stainless steel base screw, septum nut, 1/16" and 1/4" stainless steel nuts, 1/4" graphite ferrules		
<b>Injection Port Kit for HP 5890 GCs</b>	kit	21625
<b>Ferrules for split sleeves (6.35mm OD):</b>		
1/4" graphite ferrules	10-pk.	20210
1/4" Vespal®/graphite ferrules	10-pk.	20221
<b>Ferrules for splitless sleeves (6.5mm OD):</b>		
6.55mm ID Graphite Splitless Ferrules (1/4" type)	10-pk.	20260
6.55mm ID Vespal®/Graphite Splitless Ferrule (1/4" type)	10-pk.	20261
<b>Replacement Parts</b>		
<b>Flat head base screws:</b>		
S.S. Base Screws for Restek 5890 Injection Port	10-pk.	21633
	50-pk.	21634
Silcosteel® Base Screws for Restek 5890 Injection Port	10-pk.	21631
	50-pk.	21632
Gold-plated Base Screws for Restek 5890 Injection Port	2-pk.	21629
	10-pk.	21630
Needle Guide Septa Nut / 26 gauge	each	21309
Base Fitting for Restek 5890 Injection Port	each	21626
Split/Splitless Weldment for Restek 5890 Injection Port	each	21627
Shell Weldment for Restek 5890 Injection Port	each	21628

\*Does not include inlet sleeve, 1/16" type capillary ferrule, or split/splitless sleeve ferrules. Order separately.



# Hints for the Capillary Chromatographer



## Using Flame Ionization Detectors

The flame ionization detector (FID) is the most widely used GC detector. The FID is a destructive, mass flow dependent detector that utilizes a flame source to ionize compounds. Compounds responding to FIDs include all organic compounds that ionize in a hydrogen and air flame. Many of these compounds are found in petrochemical, industrial, environmental, clinical, and flavor and fragrance analyses.

The FID is considered a universal detector since it responds to a wide variety of organic compounds. However, carbon dioxide, carbon monoxide, nitrogen, oxygen, carbon disulfide, and inert gases produce little or no response by FID. Formaldehyde, formic acid and heavily halogenated compounds produce a low or non-linear response, because of the few carbon/hydrogen bonds present.

### Detector Operation

A signal for an FID is produced when a sample combusts in a hydrogen/air flame and ionizes (Figure 1). Because the FID is a mass sensitive detector, it will respond to the amount of material passing through the flame at a given time. The response of a component is directly proportional to the amount injected.

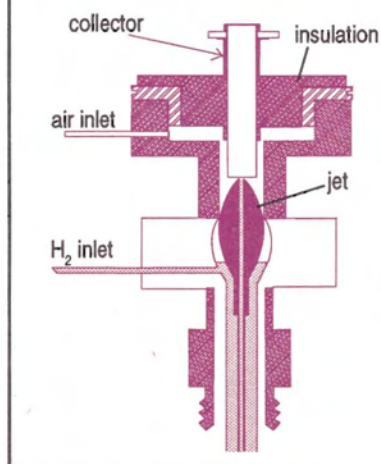
FIDs can detect concentrations of saturated compounds as low as 20pg. However, FIDs are not practical for quantitating compounds below 0.1ng because baseline noise from air movement around the collector, electrical noise, gas contaminants, and column bleed reduces FID sensitivity (signal/noise ratio). In addition, the presence of compounds with minimal carbon/hydrogen bonds (i.e., carbonyl, alcohols, halogens, amines, etc.) also make detectability below 0.1ng difficult. When an increase in concentration no longer produces an increase in response, the detector has reached its maximum linear range. The FID has the largest linear range of all the common GC detectors,  $10^7$  times the minimum detectable level of a hydrocarbon.

FID jet orifices range from 0.011" to 0.030". In general, the smaller the jet orifice, the more sensitive an FID. Internal diameters of 0.011" are used for narrow bore capillary columns (< 0.25mm ID) when high sensitivity is desired. However, smaller orifices are prone to plugging and are impractical for packed columns or thick film capillary columns. Some packed columns with high phase loadings require a 0.030" jet. In most cases, jet orifices of 0.018" are used.

### Detector Gases

FID gas flow rates are generally optimized at 30cc/min. hydrogen, 30cc/min. carrier plus make-up gas, and 300cc/min. air.

**Figure 1 - Flame Ionization Detector Design**



*Hydrogen gas is heated as it flows around the side of the jet and is mixed with the make-up gas and sample effluent exiting from the column. The combined gases travel through the jet tail and combust as they exit the jet orifice. Because there is an applied voltage across the jet, a current is produced by the ionized particles as they reach the collector and is measured by an electrometer.*

Sensitivity can be increased by decreasing the hydrogen flow rate relative to the carrier and make-up gas. Usually, a 1:1.25 ratio of hydrogen to carrier gas (25cc/min  $H_2$ , 30cc/min carrier + make-up) increases sensitivity. However, at lower hydrogen flow rates, the flame is prone to extinguishing as large volumes (>2 $\mu$ l) of water elute. A higher than optimum hydrogen flow rate is usually necessary when using water as a solvent to ensure the flame remains lit at all times. Increasing the air flow rate above 300cc/min. has little effect on FID sensitivity. However, increasing the air flow rate to 400-450 ml/min. will extend the linearity of the FID when high concentrations of analytes are injected. Since there are variations in detector design, consult your instrument manual for optimum gas settings.

Carrier, hydrogen, and air should be of high purity or the FID background noise will increase. Excessive moisture in the hydrogen or air is particularly a problem. Make-up gas is frequently recommended with capillary columns to optimize detector performance, reduce dead volume, and minimize band broadening. Without make-up gas to augment the typical 5cc/min. carrier gas flow rate of a capillary column, the flame burns rich which results in increased noise and non-linearity.

### Detector Maintenance

FIDs can become contaminated from stationary phase bleed and sample residue. Most stationary phase bleed consists of low molecular weight silicone polymer fragments that form a white silica powder when burned in the flame. This contamination can deposit on the jet tip and collector increasing background



noise. In cases of severe contamination, noise spikes occur as the silica particles are carried into the flame. Other stationary phases such as Carbowax® and high molecular weight sample contaminants form a residue that coats the detector surfaces. As the coating builds, noise increases and sensitivity diminishes.

Figure 2 shows a chromatogram obtained before and after a new jet was installed. In the first chromatogram, active compounds such as benzoic acid and 2,4-dinitrophenol (peaks 3 & 5) are tailing and produce a reduced response. By installing a new jet, peak shape and response could be restored. The problem was identified as stationary phase contamination from a Carbowax® column that was previously installed in the GC. Because the Carbowax® column has a lower maximum temperature than an XTI®-5 column (250°C vs. 360°C), the stationary phase decomposed when the detector temperature was increased to elute higher molecular weight compounds.

In addition to stationary phase bleed and sample residue, glass fragments or short pieces of fused silica tubing can break off and become lodged in the small jet orifice causing peak tailing or adsorption. This can occur during installation as the column is inserted into the jet and pushed as close to the orifice as possible. Often, excessive tailing solvent or sinusoidal baseline signals indicate a fused silica fragment lodged in the jet.

Inexpensive FID cleaning kits are available from most chromatography suppliers and contain jet reamers, tube brushes, and emery cloth to make cleaning easy. Avoid using kits with brass brushes which leave metal fragments inside the FID. To clean an FID, cool the detector, disassemble the collector assembly, and remove the jet. It is important to inspect the FID base for particles by shining a flashlight into the oven and through the fitting until the light exits the top of the FID where the jet is attached. Rotate the light to look for particles or contamination

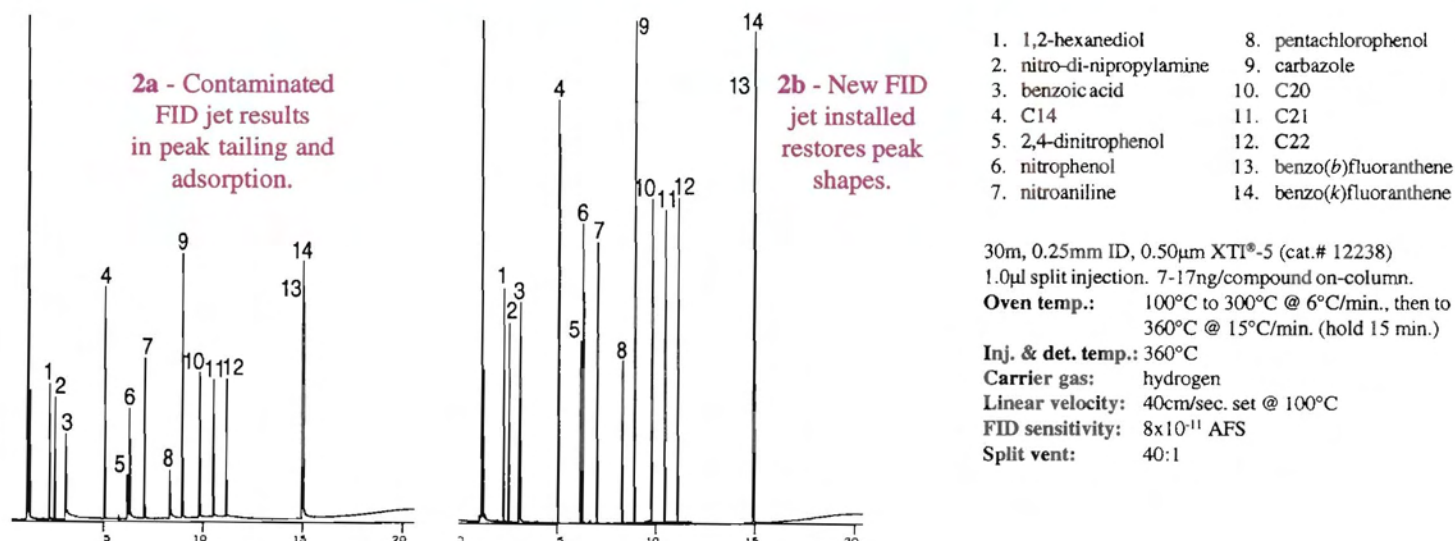
that might be on the FID base wall. Clean the detector base with a 1/4" brush. Next clean the jet orifice with a jet reamer and a stainless steel toothbrush. Shine a flashlight through the jet bore and look for dull spots or particles lodged in the bore. Clean the jet in an ultrasonic bath with solvents such as methylene chloride to remove the contamination. Discard the jet if it can not be cleaned. Finally, clean the electrometer metal contacts and spring clips with emery cloth to ensure a noise free signal. Wipe off the metal dust by using a Kimwipes® or cloth moistened with solvent. Reassemble and allow the detector temperature to reach 150°C before installing a column or lighting the flame.

### FID Operating Hints

The FID temperature should always be operated above the final oven temperature to prevent stationary phase or sample components from condensing in the detector. Since the column is installed in the detector's heated zone, never heat the FID above the stationary phases maximum operating temperature or decomposition and excessive bleed will occur. Never set an FID temperature below 100°C or water vapor from the flame will condense in the detector. Always install the capillary column as close to the jet orifice as possible to prevent sample adsorption or decomposition on the metal surfaces of the jet. Inert Silcosteel® FID jets should be used with active samples to prevent adsorption or decomposition. In addition, ceramic jets for high temperature applications are used by some instrument companies for MXT® or fused silica lined metal capillary columns to prevent grounding of the electrical signal.

Due to its ease of use and nearly universal response to most organic compounds, the FID has become the most commonly used GC detector. Proper care and routine maintenance is required to keep an FID working at maximum sensitivity with minimum background noise.

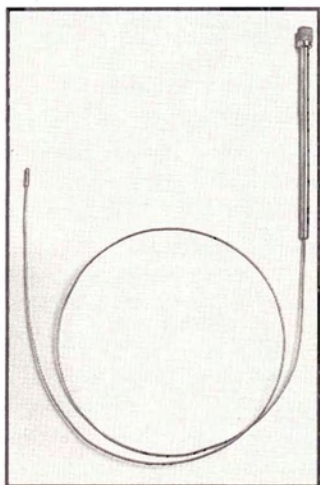
**Figure 2 - Peak tailing and adsorption occur when sample residue or stationary phase bleed deposits in the FID jet tail and interacts with the sample stream.**





# Peak Performers

## Replacement Chemical Trap for HP 5890 GCs



GC carrier gas line contamination is a difficult problem to diagnose and correct. Carrier gas line contamination can occur from carrier gas impurities or by sample backflash. Contamination can appear as ghost peaks or a rising baseline not attributed to the inlet, column, or detector. HP 5890 GCs incorporate a small chemical filter in the carrier gas line to reduce the possibility of contamination. However, the trap capacity is low and must be periodically changed or contamination will occur. It is recommended the traps be replaced annually under normal operating conditions or more frequently under heavy usage.

Restek's chemical trap is easy to install and attaches to the same fittings as HP's chemical trap. It incorporates built-in frits and adsorbents to remove both moisture and hydrocarbons. Additionally, Restek's trap can be regenerated to remove trapped contaminants and restored to its original performance.

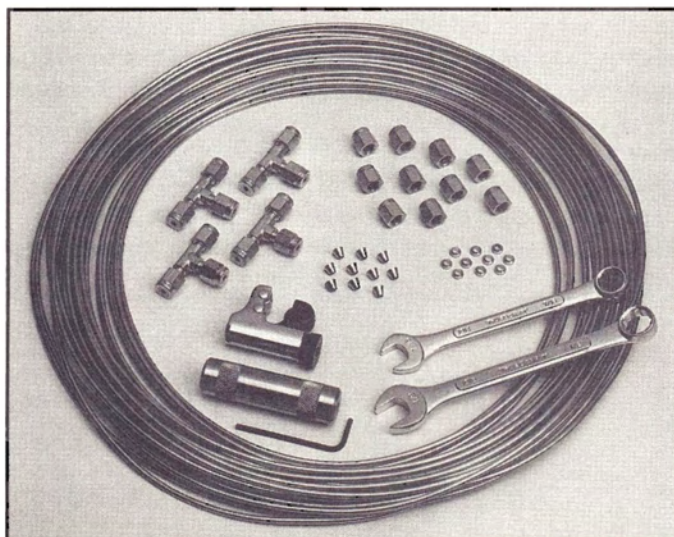
**Replacement Chemical Trap for HP 5890 GCs**  
cat.# 21610, each

## GC Installation Kit

Now, a GC Installation kit to make your life easier! This kit contains the necessary tubing and fittings to add an additional GC to your lab bench. Also included in the kit are four 1/8" tees, so GC gases like hydrogen, helium, nitrogen, and air can be routed to the new inlet or detector from the existing gas lines. Additional parts, such as purifiers or regulators, may be ordered separately to customize the GC installation to your lab specifications.

Kit includes: one Imp™ tubing cutter, one 1/8" x 1/4" reamer, one 7/16" wrench, one 1/2" wrench, four 1/8" brass tees, ten 1/8" brass nuts, ten brass front and back ferrules, and 50' of pre-cleaned 1/8" copper tubing.

**GC Installation Kit:** cat.# 21325



## Injector Nut for Shimadzu 17A GCs

*Uses standard 1/16" Swagelok®-type ferrules*

Restek has developed a special inlet fitting for Shimadzu 17A GCs that allows the use of standard 1/16" Swagelok®-type ferrules. Either Vespel® or graphite type ferrules can be used. (Similar to Shimadzu part# 221-32509.)  
**cat.# 21895, kit**

### Trademarks

CarboBlack™, mini-Lam™, MXT®, Rtx®, Silcosteel®, Uniliner®, and XTT® are trademarks of Restek Corporation. CARBOWAX is a trademark of Union Carbide Corp. Carbo-pack is a trademark of Supelco, Inc. Carbograph is a trademark of Alltech. Vespel is a trademark of E.I. DuPont Nemours & Co., Inc. Swagelok is a trademark of Crawford Fitting Co. Imp is a trademark of Imperial Eastman. Kimwipes is a trademark of Kimberly-Clark Corporation. Restek capillary columns are manufactured under U.S. patent 4,293,415, licensed by Hewlett-Packard Company.



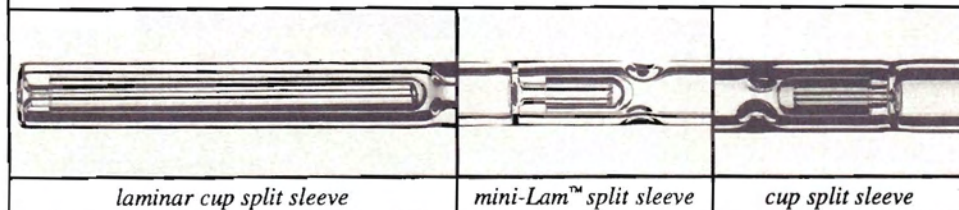
## **mini-Lam™ Split Sleeve for HP 5890 GCs**

### *Same benefits as the laminar cup at a lower price!*

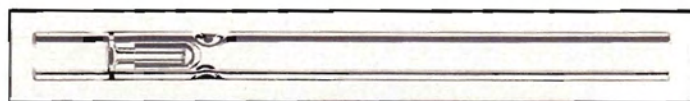
Grob<sup>1</sup> has published results showing the laminar cup splitter as the best split inlet sleeve to prevent un-vaporized sample from reaching the column. Injection volumes as large as 5µl can be made without having un-vaporized sample reach the column. Un-vaporized sample will result in molecular weight discrimination or non-linear response. Grob has determined that the narrow channels between the laminar cup and sleeve wall are responsible for enhanced evaporation since the gap at the bottom of the laminar cup traps the liquid sample until it is fully vaporized. Other sleeve designs are not as effective in completely vaporizing the sample, even with volumes as small as 2µl.

Although the laminar cup provides the best sample vaporization and minimizes discrimination, it is costly to manufacture. The complexity of the sleeve design and precision required to seal several glass chambers at the outlet of the sleeve (Figure 1) greatly increases the price when compared to a simple splitless sleeve. Restek has recently developed a new sleeve, the *mini-Lam™*, that maintains the same benefits of a laminar cup, but at a lower cost. The design of the new *mini-Lam™* utilizes a shortened, inverted laminar cup. Because the head of the shortened laminar cup faces upward, septa particles and sample

**Figure 1 - The *mini-Lam™* split sleeve offers the benefits of a laminar cup sleeve (complete sample vaporization) while reducing trapped septa particles and sample residue common with a standard inverted cup split sleeve.**



residue are not trapped in the cup body as seen with standard cup sleeves. The *mini-Lam™* sleeve can also be used for dual column analysis in a split/splitless injection port using a two-hole ferrule. The wider base of the *mini-Lam™* provides enough room for the installation of two capillary columns, whereas the standard laminar cup splitter could not.



#### **mini-Lam™ Split Sleeve for HP 5890 GCs**

cat.# 20990, each

cat.# 20991, 5-pk.

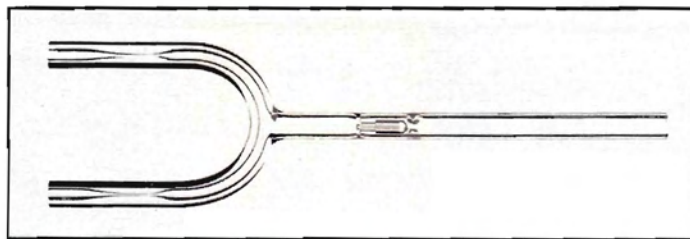
## **Dual Column *mini-Lam™* Direct Injection Tee for 1/4" Packed Column Inlets**

*Complete vaporization of the sample and visual confirmation of the column connection*

Direct injection into two columns simultaneously has become increasingly popular for dual column confirmational analysis. Previously, Restek only offered a cyclo dual column direct injection tee that utilized a glass screw to vaporize the sample prior to reaching the tee split point. Based on Dr. Grob's work<sup>1</sup>, we now have a *mini-Lam™* direct injection tee which allows complete vaporization and permits larger sample volumes.

The *mini-Lam™* direct injection sleeve is designed to fit into 1/4" packed column injectors. The tee incorporates a press-tight taper in each outlet leg that delivers a perfect, dead volume free connection to each 0.32 or 0.53mm ID analytical column (OD ranging from 0.4 to 0.8mm) and allows visual confirmation of the column connection. The open top design makes it easy to pack with glass wool to keep dirty sample residue from contami-

nating the cup. A complete *mini-Lam™* dual column direct injection kit is available that includes a deactivated 1/4" glass tee, 1/4" nut and ferrule, two 1/4" to 1/16" reducing unions, and ferrules. Deactivated replacement glass tees are also available.



**mini-Lam™ Direct Injection Tee Kit:** cat.# 20436, kit

**mini-Lam™ Direct Injection Tee:** cat.# 20435, each

1. K. Grob, *HRC & CC*, 15 (1992) 190.





" some" **promos / Products / Offers** in the **ADVNews**  
have been since been progressively superceded  
/ UPDATED OR Since Discontinued  
**CHECK** THE latest Restek ADVantage Newsletter, Restek ESSENTIALS  
. . . Or The Restek Catalog . . . Or other Restek publications for updates  
**www.chromtech.net.au** or NEW site 2015 > **www.chromalytic.net.au**



# THE RESTEK

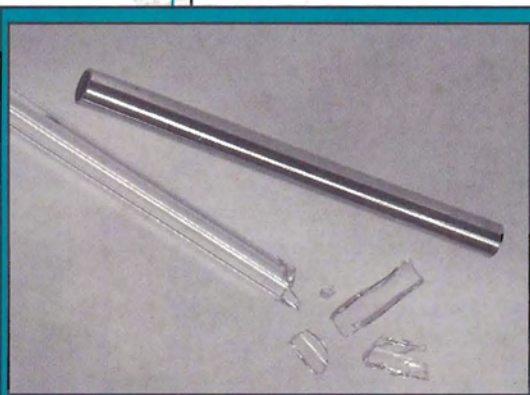
## ADVANTAGE

### Innovative Silcosteel® Metal Inlet Sleeves

Restek has developed a new low cost, high performance inlet sleeve for the HP 5890/6890 split/splitless injector. Advances made by our manufacturing group enable us to introduce a stainless steel inlet sleeve with inertness equivalent to deactivated glass. Utilizing our advanced Silcosteel® process, metal sleeves are coated with a fused silica-like layer and then deactivated with a high temperature silanization process. These sleeves are ideal for split/splitless applications that require frequent changing of inlet sleeves to keep the injector contamination free. These sleeves are also excellent for portable GCs since they do not break during transportation or installation.

#### SILCOSLEEVE™ METAL INLET SLEEVES UTILIZE RESTEK'S SILCOSTEEL® SURFACE

Silcosteel® is a process that bonds a thin, uniform layer of flexible fused silica on the stainless steel. The surface is then deactivated and made inert by the same process used in our high quality fused silica and MXT® capillary columns. Because the Silcosteel® layer is flexible, it will not crack or break off the surface of the stainless steel.



- Equivalent inertness to glass sleeves.
- Excellent response for pesticides, phenols and other active compounds.
- Silcosleeve™ inlet sleeves won't crack, chip, or break like glass sleeves.
- Inexpensive and cost effective.





# PINNACLE™ TO-11:

## The Definitive HPLC Column for Formaldehyde Analysis

EPA method TO-11 describes a procedure for the determination of formaldehyde as well as 14 other aldehydes and ketones in ambient indoor and outdoor air. Formaldehyde is of particular concern due to its widespread occurrence and tendency to photochemically react to produce many pollutants such as peroxyacetyl nitrate compounds, peroxides, and ozone. Its major source of emissions is due to incomplete combustion of hydrocarbons such as fossil fuels and waste incineration. It is also released from many chemical and industrial processes in which it is used. Short term exposure to formaldehyde and other carbonyl compounds can cause irritation of the eyes, skin, and mucous membranes, while chronic or high level exposure may damage the lungs and other vital organs.

The TO-11 method outlines a procedure in which the sample is collected by drawing

air through cartridges containing a solid adsorbent coated with dinitrophenylhydrazine (DNPH). Following collection the sample is eluted from the cartridge as the DNPH derivative and then analyzed by HPLC with UV detection. Although this procedure was written specifically for formaldehyde, it also lists 14 other aldehydes and ketones which can be analyzed with modification of the basic chromatographic procedure.

For the analysis of formaldehyde alone, the method recommends a single 25cm Zorbax™ ODS column with a water/acetonitrile mobile phase. Using this column and conditions, formaldehyde is retained approximately 7 minutes. Using a 15cm, 5µm Pinnacle™ TO-11 column, the retention is decreased to under 2 minutes. The analysis time can be further reduced by using a shorter, high efficiency 3µm Pinnacle™ column.

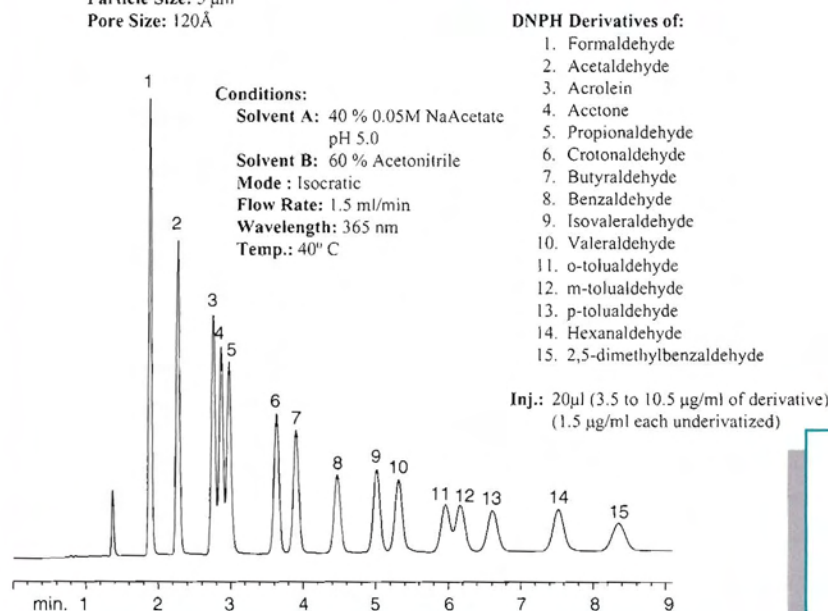
To separate all 15 compounds listed in the method the procedure recommends two 25cm Zorbax™ ODS columns in series and a 55 minute gradient with a 15 minute reequilibration. This long gradient and use of two columns is required because a regular ODS column does not have the proper selectivity to easily separate acetone, acrolein, and propionaldehyde as well as the tolualdehyde isomers. By using a Pinnacle™ TO-11 column which has been specifically optimized for this analysis, all 15 compounds can be easily separated isocratically in less than 10 minutes. For greater resolution of the closely eluting triplicates a 15cm x 3µm or 25cm x 5µm may be used.

The knowledgeable applications group at Restek has made similar improvements to other environmental methods as well. Application Briefs on the HPLC analysis of explosives, carbamates, and PAHs are available upon request. Call your local distributor to request more information on these applications.

**Figure 1 - PINNACLE™ TO-11 elutes all 15 components in Method TO-11 isocratically in less than 10 minutes.**

Column: Pinnacle™ TO-11  
Catalog Number: 9172565  
Dimensions: 150 x 4.6 mm  
Particle Size: 5 µm  
Pore Size: 120Å

*Call your local distributor to request a copy of  
Restek's HPLC Catalog!*



## PINNACLE™ TO-11

ID (mm)	Particle Size	Length (mm)	cat. #
4.6	3 µm	100	9172315
	3 µm	150	9172365
	5 µm	150	9172565
	5 µm	250	9172575
3.2	3 µm	100	9172313
	3 µm	150	9172363
	5 µm	150	9172563
	5 µm	250	9172573

— FAST ANALYSIS —  
— IMPROVED RESOLUTION —  
— ISOCRATIC SEPARATION —  
— BINARY MOBILE PHASE —  
— DECREASED SOLVENT USAGE —





# Get "FAME"ous!

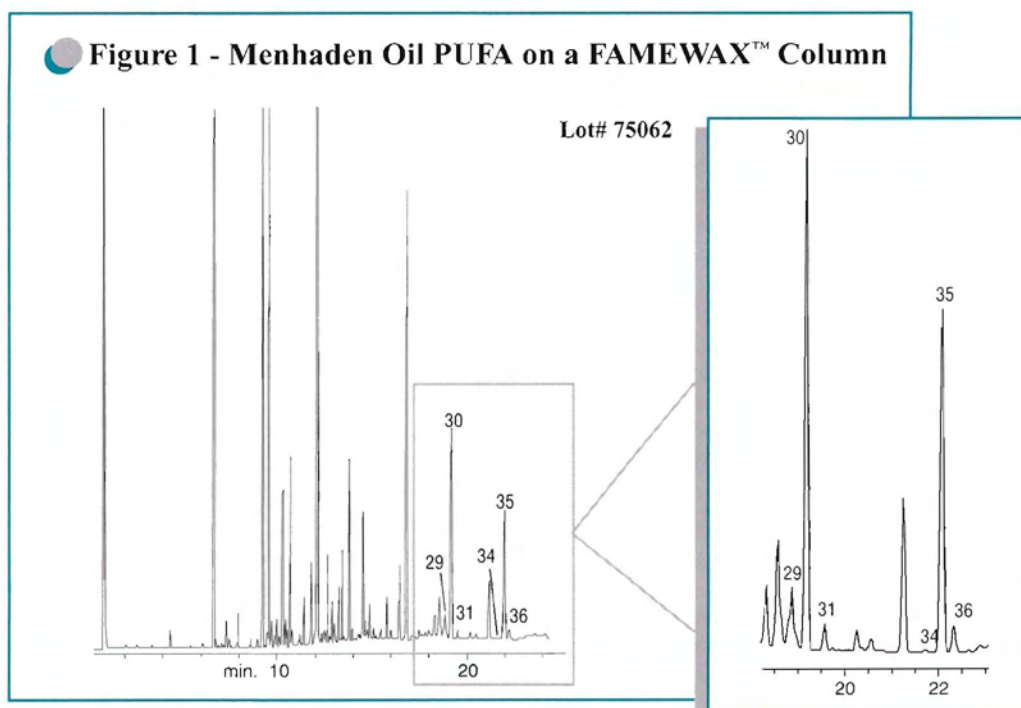
**Restek's new FAMEWAX™ column for fast and efficient FAME analysis.**

Current methods for the analysis of fatty acid methyl esters (FAMES) have been designed using Carbowax® columns that provide a specific elution order and separation of polyunsaturated fatty acids (PUFAs) in 35 to 50 minutes. Restek's new 0.25 and 0.32mm ID FAMEWAX™ columns can provide necessary baseline resolution for complex PUFA samples in less than 22 minutes! 0.53mm ID FAMEWAX™ columns are also available for concentrated FAME samples and for conversion from packed to capillary columns.

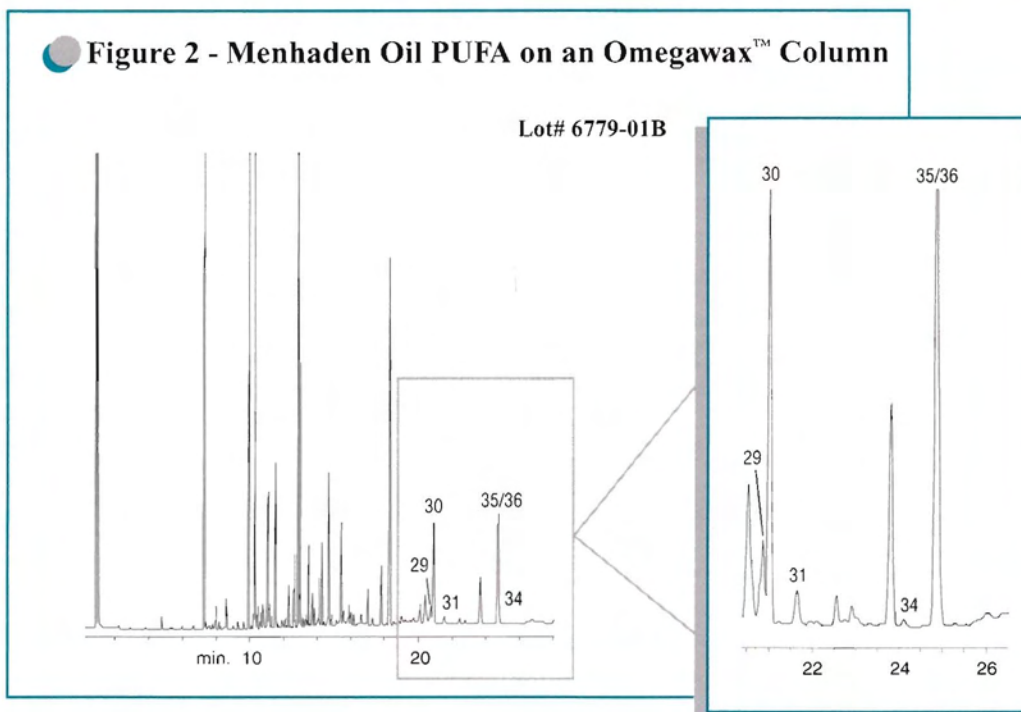
Capillary column performance requirements for PUFA analysis are specified in AOCS and AOAC methods. The American Oil Chemists Society (AOCS) Method CE 1b-89 "FAMES analysis by capillary GLC" requires baseline resolution of C21:5n3 and C23:0 (internal standard [IS]) and C24:0 and C22:6n3 (DHA). The Association of Official Analytical Chemists (AOAC) Official Method #991.39 "Fatty Acids in Encapsulated Fish Oils and Fish Oil Methyl and Ethyl Esters" requires the same elution pattern as Carbowax® 20M and additional resolution of C23:0(IS) from C22:4n6.

FAMEWAX™ columns meet all the criteria listed in the methods in significantly less time, with faster flow and temperature program rates than other Carbowax® columns. The menhaden oil PUFA analysis in Figure 1 shows that C21:5n3 and C23:0 (IS) are well resolved, as are C24:0, C22:6n3 (DHA) and C24:1n9 with a total analysis time of only 22 minutes. Figure 2 shows the same analysis on the Supelco Omegawax® 250 column with identical GC conditions. Peaks C21:5n3 and C23:0 are not baseline resolved, nor are C22:6n3 and C24:1n9. To achieve resolution of these components on

**Figure 1 - Menhaden Oil PUFA on a FAMEWAX™ Column**



**Figure 2 - Menhaden Oil PUFA on an Omegawax™ Column**



the Omegawax® column, the program rate must be decreased to 2 or 3°C/minutes, increasing analysis time by 59%!

The 30m, 0.32mm, 0.25µm FAMEWAX™ column also meets the criteria for PUFA analysis. Larger diameter 0.32 mm ID col-





umns have 4-5 times more sample capacity (400-500ng vs. 50-100ng). The increased sample capacity minimizes overloading of more concentrated samples with minimal loss in column efficiency.

The 30m, 0.53mm, 0.5µm FAMEWAX™ column has the sample capacity to accommodate direct and on-column injections. This wide bore column allows conversion from packed to capillary columns without the expense of adding a capillary injector to your GC. Although attaining the resolution requirements for PUFA analysis is difficult for most wide bore PEGs, Figure 3 illustrates that the 0.53mm ID FAMEWAX™ column can provide sufficient resolution for PUFA analysis.

Unlike similar columns from other manufacturers, all FAMEWAX™ columns are tested with two test mixtures. Each batch of polymer is tested with a menhaden type

oil, and each column is tested with a Grob type test mix. The menhaden oil test ensures proper column polarity indicated by the separation and elution order of PUFAs. Also, 0.25mm and 0.32mm ID FAMEWAX™ columns must pass the resolution criteria outlined in the official methods for complex PUFA matrices. The Grob mix ensures inertness, efficiency, film thickness consistency and minimal column bleed.

Save yourself time and money. Try Restek's new FAMEWAX™ columns for fast and efficient FAME analyses. The 0.25mm and 0.32mm ID FAMEWAX™ columns provide optimum PUFA analyses in less time than other columns. The 0.53mm ID provides capillary conversion in packed column instruments, maximum sample capacity, and the necessary resolution for a wide variety of FAME analyses, including complex PUFA analysis. All FAMEWAX™ columns are tested with two test mixtures to pro-

vide you with the highest quality column for FAME analyses anywhere. We guarantee it!

## FAMEWAX™ Columns

- Significantly reduces analysis times.
- Specially tested to ensure column reproducibility. We guarantee it!
- 0.25mm and 0.32mm ID columns available for complex PUFA analysis.
- Also available in 0.53mm ID columns.

## FAMEWAX™ Columns

30m, 0.25mm ID, 0.25µm  
FAMEWAX™:  
cat.# 12497

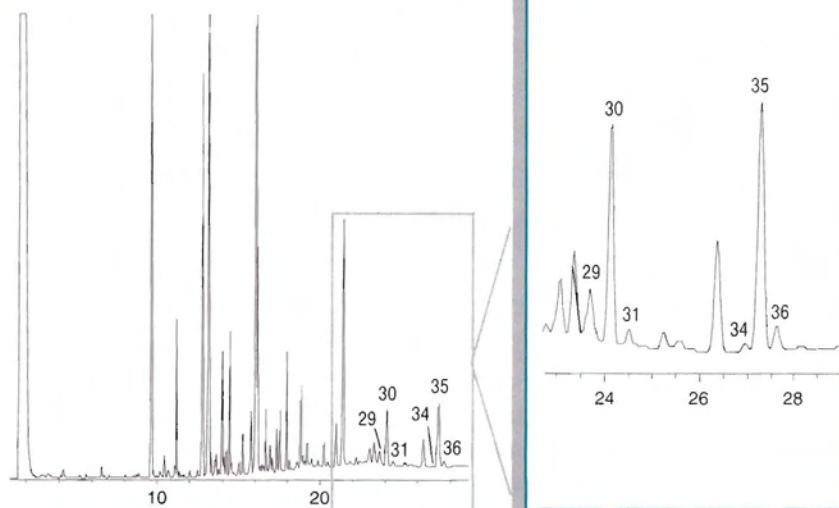
30m, 0.32mm ID, 0.25µm  
FAMEWAX™:  
cat.# 12498

30m, 0.53mm ID, 0.50µm  
FAMEWAX™:  
cat.# 12499

## Compound List and Conditions for Figures 1-3

29. C21:5n3	34. C24:0
30. C23:0 (IS)	35. C22:6n3
31. C22:4n6	36. C24:1n9

**Figure 3 - The 0.53mm ID FAMEWAX™ provides the necessary resolution for PUFAs using direct injection.**



**Figure 1**

30m, 0.25mm ID, 0.25µm FAMEWAX (75062)

**Figure 2**

30m, 0.25mm ID, 0.25µm Omegawax (6779-01B)  
0.8µl split injection of menhaden oil PUFA with C23:0 (IS)

On-column concentration 100-150ng.

**Oven temp.:** 120°C to 220°C @ 7°C/min. (hold 20 min.)

**Inj. & det. temp.:** 220°C

**Carrier gas:** hydrogen

**Linear velocity:** 60cm/sec. set @ 120°C

**FID sensitivity:** 8 x 10<sup>-11</sup> AFS

**Split ratio:** 50:1

**Figure 3**

30m, 0.53mm ID, 0.50µm FAMEWAX (82764B)

1.0µl direct injection of menhaden oil.

On-column concentration 2500ng total.

**Oven temp.:** 120°C (hold 2 min.) to 220°C @ 6°C/min. (hold 20 min.)

**Inj. & det. temp.:** 230°C

**Carrier gas:** hydrogen

**Linear velocity:** 32cm/sec. set @ 120°C

**FID sensitivity:** 8 x 10<sup>-11</sup> AFS





# Integra-Guard™ Takes the Frustration Out of Analyzing Residual Solvents

The United States Pharmacopeia (USP) has published several methods for the analysis of residual solvents in pharmaceutical products. USP 467 outlines five methods for Organic Volatile Impurities (OVI) that utilize several different sample introduction techniques and analytical columns. Methods I and V have become the most popular because of the simplified sample introduction technique. Both of these methods involve the direct dissolution of the pharmaceutical product in water followed by direct aqueous injection. Since many pharmaceutical products contain inorganic additives and non-volatile components, the USP methods recommend the use of a 5 meter guard column to protect the analyti-

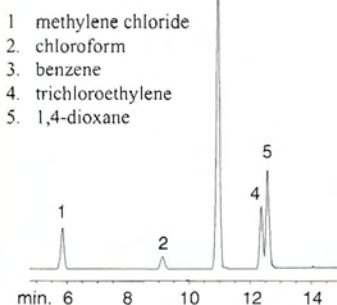
cal column. Although the use of a guard column can greatly increase the life expectancy of your analytical column, making a consistent leak-free connection has been difficult.

To eliminate the headache of making those difficult connections between the guard column and the analytical column, the Wizards at Restek have developed Integra-Guard™ (capillary columns with integrated guard columns). These columns are available with the two most common stationary phases used for OVI analysis. Figure 1 shows the analysis of common solvents on a 30 meter, 0.53mm ID, 5.0µm Rtx®-G27 Integra-Guard™ column (5% phenyl/95%

methyl polysiloxane). Figure 2 shows the same analysis on a 30 meter, 0.53mm ID, 3.0µm Rtx®-G43 Integra-Guard™ column (6% cyanopropylphenyl polysiloxane). Both columns are manufactured with high purity, bonded stationary phases for low bleed and unsurpassed inertness. Each column is tested to meet stringent requirements to insure reproducible analysis.

Save time and money and improve your analysis by using Rtx®-G27 and Rtx®-G43 Integra-Guard™ columns to analyze residual solvent impurities in pharmaceutical products. In addition, Restek offers several high quality calibration standards for USP 467 methods. Data packages with complete quality assurance information are available for all Restek standards.

**Figure 1 - USP 467 analysis on a 30m, 0.53mm ID, 5.0µm Rtx®-G27 Integra-Guard™ column**



1.0µl direct injection of USP 467 Mix #1 (cat.# 36001).  
**Oven temp.:** 35°C (hold 5 min.) to 175°C @ 8°C/min. to 260°C @ 35°C/min.  
**Inj. / det. temp.:** 200°C / 240°C  
**Carrier gas:** helium  
**Linear velocity:** 30cm/sec. set @ 35°C  
**FID sensitivity:** 1 x 10<sup>-11</sup> AFS

## Columns

30m, 0.53mm ID, 5.0µm  
Rtx-G27 Integra-Guard™  
cat.# 10279-126

30m, 0.53mm ID, 3.0µm  
Rtx-G43 Integra-Guard™  
cat.# 16085-126

## Standards

**USP 467 Mix #1 (in DMSO)**  
cat.# 36001, ea.  
cat.# 36101, 10-pk.

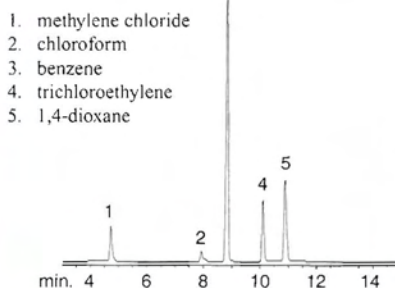
**USP 467 Mix #2 (in methanol)**  
cat.# 36002, ea.  
cat.# 36102, 10-pk.

**USP 467 Mix #3 (in DMSO)**  
cat.# 36004, ea.  
cat.# 36104, 10-pk.

**International USP 467 Mix (in methanol)**  
cat.# 36003, ea.  
cat.# 36103, 10-pk.

See Restek's Product Guide for more information on USP 467 standard mixes.

**Figure 2 - USP 467 analysis on a 30m, 0.53mm ID, 3.0µm Rtx®-G43 Integra-Guard™ column**



1.0µl direct injection of USP 467 Mix #1 (cat.# 36001).  
**Oven temp.:** 35°C (hold 5 min.) to 175°C @ 8°C/min. to 260°C @ 35°C/min.  
**Inj. / det. temp.:** 200°C / 240°C  
**Carrier gas:** helium  
**Linear velocity:** 34cm/sec. set @ 35°C  
**FID sensitivity:** 1 x 10<sup>-11</sup> AFS

- Integrated guard column eliminates leaks.
- Meets all requirements of USP 467 methods.
- Bonded phase columns for low bleed & excellent inertness.



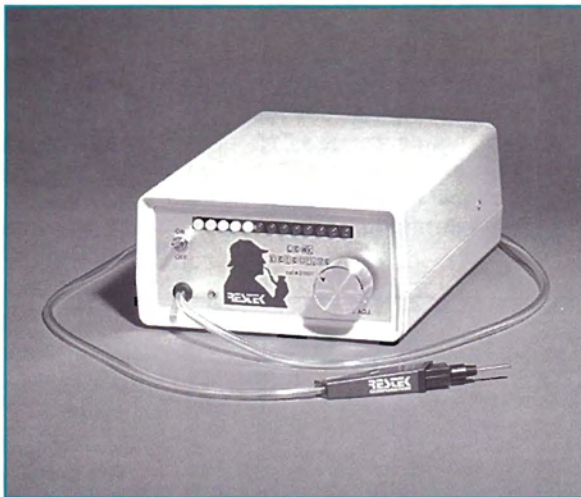


# Restek's Leak Detective™

Restek's Leak Detective™ electronic leak detector is the convenient and affordable solution for GC leak detection. It responds in less than 2 seconds to trace leaks of gases\* with thermal conductivities different than air. Helium or hydrogen can be detected at  $3 \times 10^{-4}$  cc/sec. or an absolute concentration less than 200ppm. Leaks are indicated by an audible alarm, as well as an LED readout. Two 9-volt batteries provide 10-12 hours of continuous operation, or the unit can be used with an AC adaptor (both included).

## Features

- Lowest cost thermal conductivity leak detector available.
- Compact, light weight, hand-held design.
- Contamination-free leak detection.
- Detects helium or hydrogen trace leaks at  $\geq 3 \times 10^{-4}$  cc/sec. or  $\geq 200$ ppm.



## Detecting Leaks

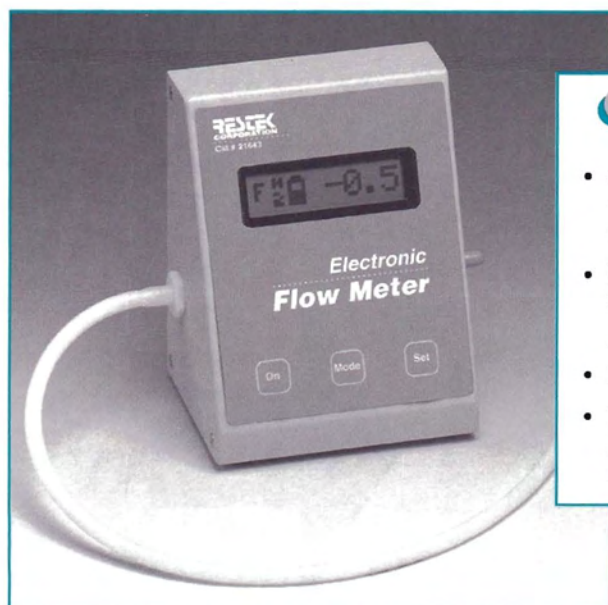
*Leaks in a GC system can cause problems ranging from increased detector noise, baseline instability, and short column lifetime, to wasting expensive carrier gases. Electronic leak detectors, like Restek's Leak Detective™ allow analysts to detect minute gas leaks undetected by liquid leak detectors without risk of contamination. Electronic leak detectors are an absolute necessity with capillary GC!*

## Leak Detective

cat.# 21607 (110 Volts)  
cat # 21609 (220 Volts)

\* Not designed for use in explosive atmospheres.

# Restek's Veri-Flow 500 Flow Meter



## New Features

- Calculates linear velocity based on column ID.
- Includes standard RS 232 ComPort with printer function.
- Has auto zero function.
- Includes rechargeable battery pack and charger.

## Other Features

- Reads flow accurately to 500mL/min.
- Measures N<sub>2</sub>, He, H<sub>2</sub>, 5% Ar/Me, and Air.
- Measures split flow and mass flow.
- Has pulse free operation that will not interfere with EPCs.
- Includes auto "off" battery saver.
- Includes NIST traceable calibration certificate.
- Includes capillary column adaptor.

## Veri-Flow 500 Flow Meter

110 V, cat.# 21643  
220 V, cat.# 21645



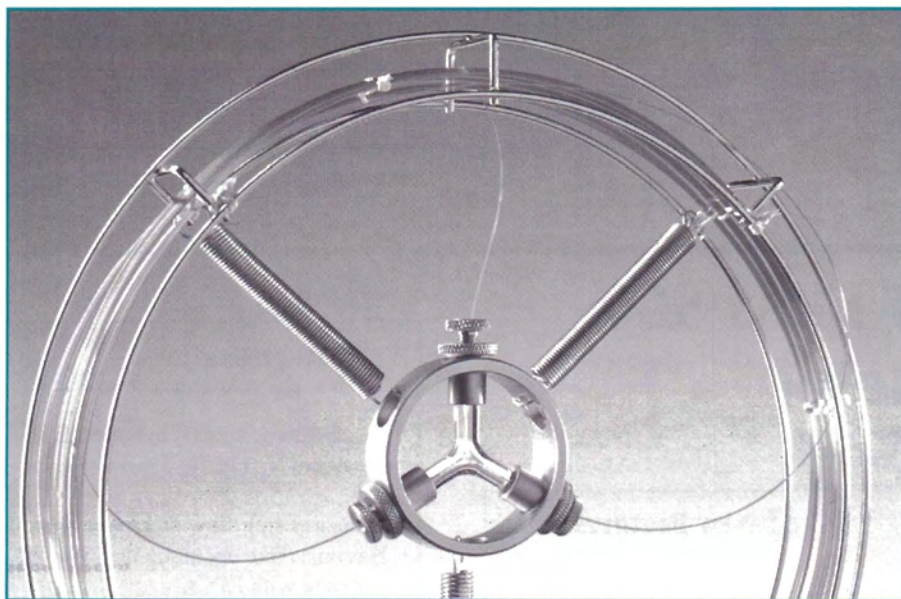


# Improved Pesticide Column Kits

Restek developed pesticide column kits in the early 90's to meet the requirements of the EPA's CLP chlorinated pesticide protocol. By connecting two capillary columns with different stationary phases to a "Y" connector and 5 meter guard column, analysts gained the advantage of running both primary and confirmational analyses simultaneously. However, the awkward angle at which the columns were inserted into the connector caused some columns to leak, disconnect, or even break. Restek solved this problem by introducing the Angled "Y" Press-Tight® connector and the "Y" Vu-Union® connector. The "Y" Vu-Union® incorporates a secondary ferrule seal that makes it almost impossible for the connection to come apart, making it ideal for higher temperature analyses. With these solutions in hand, analysts still found get-

## Features

- Pre-assembled kits offer convenience over purchasing parts separately.
- All pesticide column kits are tested for flow and leaks prior to shipping.
- Custom column combinations are available with either an Angled "Y" Press-Tight® connector or a "Y" Vu-Union® connector. Call your local distributor for more information.



## Pesticide Column Kits

*0.32mm ID & other phase combinations are available.*

Column Combo's (30m, 0.53mm ID, 0.50µm)	Cat.#	"Y" Vu-Union® connector suffix #	Angled "Y" Press-Tight® connector suffix #
Rtx-5/-1701	10950	-495	-496
Rtx-5/-35	10951	-495	-496
Rtx-35/-1701	10952	-495	-496

ting a leak free connection required "some special magic" Now analysts can rely on Restek's chemists to make leak-free dual column connections for them.

## "Y" VU-UNION® CONNECTOR

Restek's "Y" Vu-Union® connector is the latest advancement in "Y" connectors. It has quickly gained the reputation as the most reliable connector for pesticide column kits. The primary seal is visible, to assure the connection is made properly without dead volume or leakage. The secondary seal utilizes standard graphite ferrules to physically hold the columns in place and assure a "leak free" connection remains "leak free." Not only are the columns (two 30 meter analytical columns and one 5m x 0.53mm ID guard column) protected in Restek's unique capillary column cage, but the Vu-Union® connector is securely suspended inside the column cage with springs. This configuration reduces stress on the fused silica tubing and acts as a "shock absorber" from the turbulence created inside the GC oven.

## ANGLED "Y" PRESS-TIGHT® CONNECTOR

Restek's Angled "Y" Press-Tight® connector has the correct angle to assure easy, long lasting connections. Each one is carefully tested to insure a leak free seal. The guard column is carefully tucked inside the cage and is ready to use when you receive the column.

*Simply decide which columns you want to connect and the type of connector you wish to use and let us make the connection for you.*

**Order Yours  
TODAY**





# "Sample Ready" Packed Columns...

## ...Require Less Than 20 Minutes Stabilization Time!

- Fully conditioned and "sample ready" for maximum productivity.
- Silcosteel® tubing inert as glass.
- Deactivated with same process used for capillary columns.
- Rugged and flexible.
- Universal and versatile, can be bent to any instrument or detector configuration.
- Tighter tubing tolerances result in improved efficiency and reproducibility over glass.

Since the inception of glass deactivation technologies in the 1970's, packed column technology has remained virtually unchanged. In 1994, Restek introduced a breakthrough in packed columns by offering a metal packed column with efficiency and inertness exceeding the best glass packed columns. Restek's Silcosteel® process and deactivation chemistries have produced the most advanced surface for the production of packed columns on the market today.

Because the tubing material is stainless steel and the fused silica layer is flexible, Silcosteel® packed columns are truly universal. Columns can be easily moved from one instrument to another. Silcosteel® packed columns offer analysts the flexibility and versatility previously seen only by capillary column users.

All packings offered by Restek are fully conditioned and "sample ready". With only

15-20 minutes of stabilization time, your GC is ready to run. This improves throughput and lab efficiency. Restek offers a wide range of packings and liquid phases including Silcoport™, the most inert support available for packed columns. In fact, our porous polymer and Molecular sieve columns are the best we've ever seen!

### New Silcosteel® Packed Columns:\*

#### 2m x 1/8" OD x 2mm ID; Preconditioned

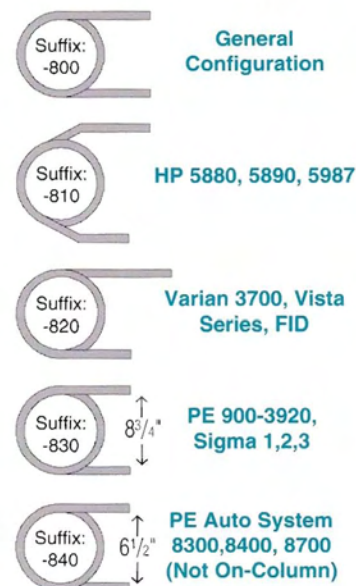
Chromosorb 101 80/100: cat.# 80435  
Chromosorb 102 80/100: cat.# 80434  
Hayesep Q 80/100: cat.# 80433  
Porapak Q 80/100: cat.# 80427  
Porapak QS 80/100: cat.# 80426  
Porapak R 80/100: cat.# 80425  
Tenax TA 60/80: cat.# 80431  
Tenax TA 80/100: cat.# 80432  
Molesieve 5A 60/80: cat.# 80428  
Molesieve 5A 80/100: cat.# 80429

#### 3.05m x 1/8" OD x 2mm ID; Fully activated

Molesieve 5A 80/100: cat.# 80430  
  
2m x 3/16" OD x 4mm ID  
1.5% Rt 2250 / 1.95% Rt 2401  
on 100/120 SilcoPort: cat.# 80254

\* Please specify catalog number and instrument configuration suffix when ordering.

### Instrument Configurations



Call your local distributor for more information on Silcosteel® packed columns or to receive Restek's Packed Column Catalog.

# SILCOSTEEL®

*Combining the durability of stainless steel with the inertness of glass.*





# Restek Advances PLOT columns – Again!

Porous polymers have been used for nearly three decades for separating light hydrocarbons and solvents. The columns traditionally used have been 2mm ID columns packed with 80/100 mesh material. The wizards at Restek have now made capillary PLOT columns available that utilize the same porous polymer materials. The Rt-S and Rt-Q PLOT columns offer a choice to the analyst who desires the selectivity of the divinylbenzene porous polymers but the efficiency of capillary columns. Figure 1 illustrates light hydrocarbons analyzed on an Rt-S and Rt-Q PLOT column. The incorporation of different vinyl monomers into the divinylbenzene porous polymer matrix produces noticeable selectivity variations. The Rt-S column provides baseline separation of ethylene and acetylene. PLOT columns offer higher efficiency when compared to packed columns, resulting in faster analyses.

Because of the large amount of porous polymer in packed columns, long conditioning times are necessary. However, with PLOT columns the actual amount of porous polymer is significantly lower. Not only does this shorten conditioning times and increase throughput, it also reduces the overall bleed level (critical with high temperature applications where low background levels are desired).

## TRUE SELECTIVITY OF TRADITIONAL POROUS POLYMER PACKINGS

Restek starts with high purity monomers for production of porous polymer PLOT columns. This ensures the columns will have a selectivity identical to that of the Porapak® and HayeSep® packings. When switching from packed columns to capillary columns, very little method development is required because the elution patterns remain the same. The Rt-Q PLOT column is a divinylbenzene homopolymer, whereas the Rt-S PLOT column is a divinylbenzene and 4-vinylpyridine copolymer.

## PLOT Columns

- PLOT columns offer an efficient alternative to packed columns.
- Selectivity identical to HayeSep® and Porapak® packings.
- All PLOT columns immobilized to eliminate particle generation.
- Unbreakable MXT-Q and MXT-Msieve 13X.
- Each column individually tested for reproducibility.

## NO PARTICLE TRAPS NECESSARY

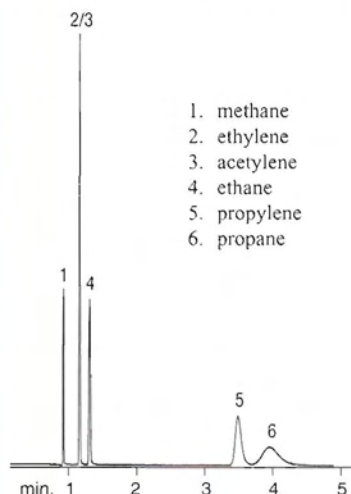
Because PLOT columns are made with submicron particles attached to the column wall, finding a good binder is critical. Restek has discovered a binding material that eliminates particle generation from PLOT columns. This process eliminates detector spiking and disruption of the valving system.

## NEW LINE OF UNBREAKABLE MXT PLOT COLUMNS

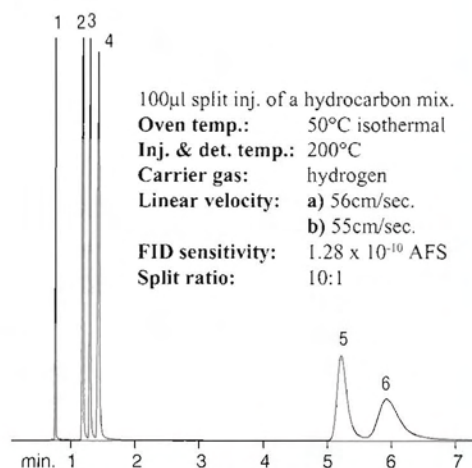
The wizards at Restek have coated PLOT columns utilizing Silcosteel® fused silica lined tubing. The Silcosteel® PLOT columns offer the analyst an unbreakable option to standard fused silica tubing with no compromise in performance. MXT® PLOT columns must meet or exceed the same rigorous specifications as fused silica PLOT columns. Because the tubing is stainless steel, field and process analyzers are no longer at the mercy of brittle fused silica tubing. Current metal PLOT columns are

Figure 1 - Selectivity changes with the incorporation of different monomers in the porous polymer.

a) 30m, 0.53mm ID Rt-Q (cat.# 19716)



b) 30m, 0.53mm ID Rt-S (cat.# 19712)



100µl split inj. of a hydrocarbon mix.  
Oven temp.: 50°C isothermal  
Inj. & det. temp.: 200°C  
Carrier gas: hydrogen  
Linear velocity: a) 56cm/sec.  
b) 55cm/sec.  
FID sensitivity: 1.28 x 10<sup>-10</sup> AFS  
Split ratio: 10:1





available with the Q porous polymer or Molecular sieve 13X zeolite. Figure 2 shows the analysis of light hydrocarbons on an MXT®-Q PLOT column and the analysis of permanent gases on an MXT®-Msieve 13X PLOT column.

### COLUMN TO COLUMN REPRODUCIBILITY GUARANTEED

Each porous polymer PLOT column is tested with a hydrocarbon test mix to insure proper phase thickness and selectivity. Propane is used to calculate the partition ratio which is monitored to insure a reproducible film thickness. The number of plates per meter is calculated and used to evaluate column efficiency. Selectivity is ensured by calculating the retention indices of acetylene and methyl acetylene.

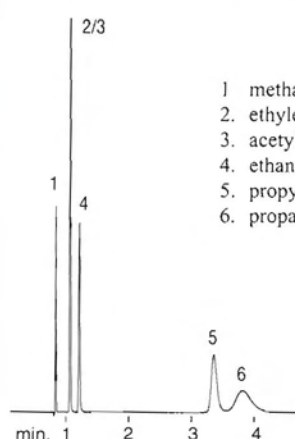
Molecular sieve PLOT columns are all tested with a mixture of permanent gases. In this test the peak height ratio of carbon monoxide is used to determine a uniform

film and to calculate the number of plates per meter.

Restek's expanding line of PLOT columns continue to bridge the gap from packed to capillary columns. Through continuing research Restek now offers unbreakable and particle free Porous Layer Open Tubular capillary columns for the analysis of permanent gases, light hydrocarbons and volatile chemicals.

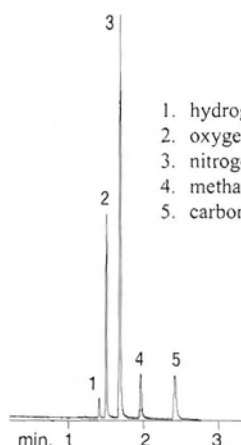


**Figure 2 - MXT® PLOT columns give fast, efficient separation of light hydrocarbons & permanent gases.**



- 1 methane
- 2 ethylene
- 3 acetylene
- 4 ethane
- 5 propylene
- 6 propane

30m, 0.53mm ID MXT-Q (cat.# 79716)  
100µl split inj. of a hydrocarbon mix.  
**Oven temp.:** 50°C isothermal  
**Inj. & det. temp.:** 200°C  
**Carrier gas:** hydrogen  
**Linear velocity:** 62.5cm/sec.  
**FID sensitivity:**  $1.28 \times 10^{-10}$  AFS  
**Split ratio:** 10:1



1. hydrogen
2. oxygen
3. nitrogen
4. methane
5. carbon monoxide

30m, 0.53mm ID MXT-Msieve 13X (cat.# 79706)  
20µl split inj. of permanent gases.  
**Oven temp.:** 40°C isothermal  
**Inj. & det. temp.:** 200°C  
**Carrier gas:** helium  
**Linear velocity:** 37cm/sec.  
**Detector:** TCD (212 mA)  
**Split ratio:** 15:1

## PLOT Columns

### Rt-S

(fused silica)

30m, 0.53mm ID	cat.# 19712
15m, 0.53mm ID	cat.# 19713
30m, 0.32mm ID	cat.# 19710
15m, 0.32mm ID	cat.# 19711

### Rt-Q

(fused silica)

30m, 0.53mm ID	cat.# 19716
15m, 0.53mm ID	cat.# 19715
30m, 0.32mm ID	cat.# 19718
15m, 0.32mm ID	cat.# 19717

### MXT®-Q

(Silcosteel®)

30m, 0.53mm ID	cat.# 79716
15m, 0.53mm ID	cat.# 79715

### Rt-Msieve 13X

(fused silica)

30m, 0.53mm ID	cat.# 19706
15m, 0.53mm ID	cat.# 19708
30m, 0.32mm ID	cat.# 19705
15m, 0.32mm ID	cat.# 19707

### MXT®-Msieve 13X

(Silcosteel®)

30m, 0.53mm ID	cat.# 79706
15m, 0.53mm ID	cat.# 79708

### Rt-Alumina

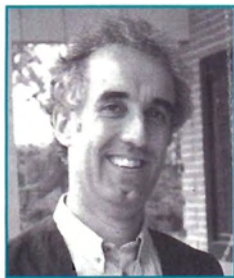
(fused silica)

30m, 0.53mm ID	cat.# 19700
50m, 0.53mm ID	cat.# 19701
30m, 0.32mm ID	cat.# 19702
60m, 0.32mm ID	cat.# 19703





# Why 5 cm syringe needles for capillary GC?



Dr. Konrad Grob

*GC is a complex technique. All too often the analyst stands in front of his instrument, surprised about a result, maybe annoyed about a problem, and at a loss for an explanation for what he observes. Often, not even his colleague is able to explain. Another of these GC mysteries? Probably he would have the knack of it if he knew the many details involved in the analytical process. We make numerous choices without being aware of them, overlook variables clinging to the illusion that they had been thoroughly investigated in the past and that an international committee has decided that this or that is the correct choice. The length of the syringe needle is one such frequently neglected detail and is an example of a parameter which has never received proper attention.*

Many years ago, the manufacturers of GC syringes looked upon their customers and noticed that there was no agreement on how long syringe needles should be for conventional vaporizing (split or splitless) injection. Some said 1.5 inch (the needle protruding 37 mm from the glass barrel), others 3 inch (71 mm), or even longer. So, father syringe producer decided to compromise and have it in between: 2 inch (51 mm). Whether or not he died in the mean time, that's how it still is. Some disagreed, but since it seems to be more important that GC is simple than that it is well optimized, the subject was commonly neglected. The subject of needle length seems not to be of sufficient scientific status to justify closer investigation.

As you can check by a few experiments, the length of the syringe needle and the depth by which a long needle is inserted into the injector often have an important impact on quantitative analysis. The reasons are explained below. It is concluded that they need to be adjusted to the situation. The length of the syringe needle de-

termines from which point inside the liner the sample expands during the evaporation process. It may, however, also influence vaporization itself.

## HEADSPACE ANALYSIS:

We start by looking at gas or headspace analysis, because the situation is particularly simple since no vaporization interferes. However, the same principals will also apply to liquid samples. We refer to (manual or automated) injection with a gas-tight syringe of 0.5-1 ml capacity.

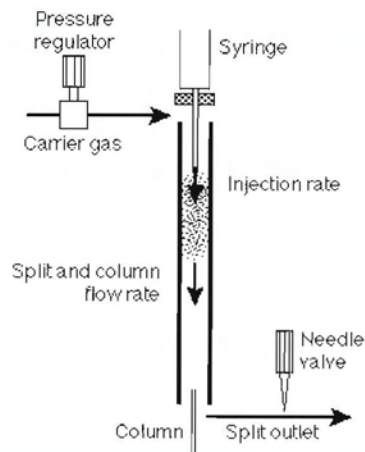
Usually an amount of gas phase is injected that approaches the internal volume of the vaporizing chamber. For instance, a 4 mm ID liner of 80 mm length has an internal volume of 1 ml. A 500  $\mu$ l sample mixes with carrier gas to form a vapor cloud of close to this volume (inlet pressure compresses the cloud, but increased temperature causes it to expand). Care must be taken to release the sample from the syringe needle in such a way that it ends being positioned inside the chamber.

Gas and headspace samples are usually injected in the split mode in order to achieve sharp initial bands. Depression of the plunger at normal speed introduces the sample at around 0.5-1 ml/s, i.e. 30-60 ml/min. If the sum of the split and the (comparably small) column flow rate corresponds to the rate of injection, expansion of the sample downwards replaces the gas

flow from the rear. Gas supply is stopped; the gas phase running off originates from the syringe (assumption of a pressure-regulation/needle valve system, Fig. 1). At higher split flow rates, the sample is diluted with additional carrier gas from the rear. Under these conditions, basically unlimited volumes of sample can be injected without overloading the injector. A short syringe needle merely entering the vaporizing chamber (2-3 cm) serves the purpose, but longer needles are no drawback.

Since headspace analysis is mostly trace analysis, the split flow rate is usually substantially below the 30-60 ml/min mentioned above. This leaves the choice of injecting at a correspondingly reduced rate or temporarily storing the vapor cloud inside the vaporizing chamber. The latter corresponds to common practice. If more sample is injected than gas runs off at the same time, carrier gas must be displaced within the injection system. Appropriately designed injectors with a pressure regulator at the rear and a needle valve in the split outlet have a relatively large internal volume in the gas supply and a small one in the split outlet, causing the sample to expand backwards (Fig. 2). Long syringe needles are required such that the sample expands from a point near the column entrance towards the rear. If the liner is 80 mm long, the column enters by 5 mm, and the injector head is some 12 mm high, the syringe needle should be around 80 mm

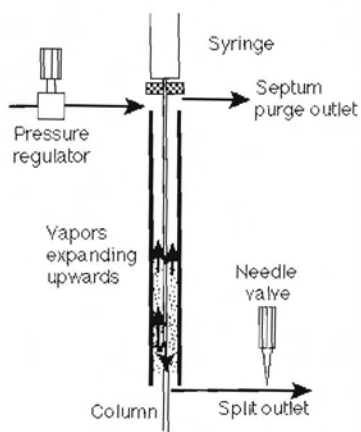
**Figure 1 - Injection at a rate equal to the flow rate of the gas passing through the liner: the flow from the carrier gas supply is substituted by that leaving the syringe needle.**







**Figure 2 - Headspace injection at a low split flow rate, using gas supply by the pressure regulator/needle valve system: the sample should expand from the column entrance backwards.**



long. The commonly used 5 cm needles enter the liner by less than 4 cm and merely exploit the upper half of the chamber. 500  $\mu$ l thus injected already overfill the injector liner, i.e. cause sample material to be expelled through the septum purge outlet or to penetrate the carrier gas supply line.

Systems with flow-regulated carrier gas supply and a back pressure regulator in the split outlet (e.g. Hewlett Packard) behave differently. Pressure increase by injection causes the back pressure regulator to open widely and increase the split flow rate. The sample cloud expands downwards (Fig. 3). As the volume of the injector can only be exploited by releasing the sample at the top of the chamber, the syringe needle should be no longer than 2-3 cm (or a longer needle should be introduced only partially).

A drawback of this type of gas supply is the split flow rate during the splitting process is rather ill defined.

### SPLIT INJECTION OF LIQUID SAMPLES

Split injection of liquids resembles gas/headspace injection except that the rate of vapor formation cannot be controlled. Injection must occur rapidly in order to avoid excessive evaporation inside the syringe needle. 2  $\mu$ l of a solution in a volatile solvent, such as dichloromethane, creates some 0.9 ml of vapor in maybe 0.5, i.e. vapors are formed at 1.8 ml/s (108 ml/min). With a split (and column) flow rate of 108 ml/min at least, the situation of Fig. 1 applies, i.e. the syringe needle should merely enter the vaporizing chamber. It leaves maximum room between the needle exit

and the column entrance for sample evaporation and mixing across the vaporizing chamber. If the split flow rate is lower, i.e. vapors are formed more rapidly than gas is discharged, a long or a short needle is best suited, depending on the carrier gas supply system involved (Fig. 2 or 3).

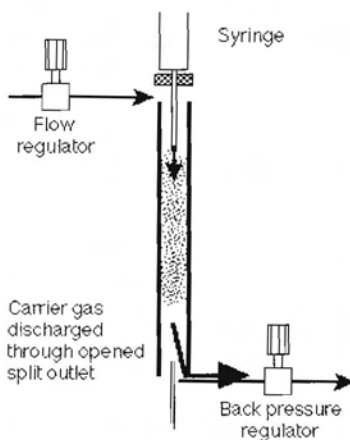
Samples with high boiling matrices, such as many undiluted liquids, evaporate slowly; discharge of the vapors is a problem only if the split flow rate is extremely low. Such liquids are easily transferred to the wall of the liner (no repulsion by vapors). If an empty liner is used (preferably of narrow bore, e.g. 2 mm), short syringe needles render such transfer more reliable as the risk of shooting the sample liquid by the column entrance becomes small.

### SPLITLESS INJECTION

In splitless injection, the sample vapors must be stored in the vaporizing chamber until they are transferred into the column, which may take over a minute. Before being diluted with carrier gas, 2  $\mu$ l of a solution in hexane produce around 500  $\mu$ l of vapor, in dichloromethane as much as 900  $\mu$ l, which shows that the internal volume of an 80 mm x 4 mm ID liner must be fully exploited.

As the split outlet is closed, there is only one way of filling the vaporizing chamber: from the bottom to the top, displacing the carrier gas backwards. The syringe needle must be adjusted to situate the center of sample evaporation slightly above the column entrance. The distance between the needle exit and the column entrance must account for the distance the droplets travel before evaporating, i.e. 1-2 cm. For the usual geometry of the injector this means using 3 inch (71 mm) needles (or rather the vaporizing chamber was designed such that standard 3 inch needles would fit). There is a second reason for depositing the sample close to the column entrance. As shown in Fig. 4 (on the following page), a 5 cm syringe needle leaves a distance of some 40 mm to the column entrance, representing a plug of some 400  $\mu$ l of carrier gas. Before substantial amounts of sample vapor reach the column, this gas must be discharged into the column, i.e. during 10-20s primarily carrier gas is "injected".

**Figure 3 - Sample expanding downwards in the instance of a system with flow regulation/back pressure regulation.**

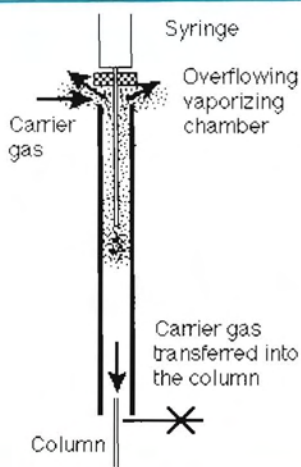






## KONI'S KORNER

- **Figure 4 - 5cm syringe needles are too short for splitless injection as the chamber is overfilled even with small sample volumes & some 400 $\mu$ l of carrier gas must be transferred into the column before sample vapors get there.**



evaporation in the gas phase of the injector (the most gentle vaporization process, since there are no contacts with packing materials adsorbing or degrading solutes). There is more vaporization inside long needles accentuating these advantages and disadvantages.

### CONCLUSIONS

The 5 cm needle for vaporizing GC injectors is a typical compromise: it is between the desirable long and the desirable short needle, but is hardly ever desirable as such. The following table suggests optimum needle lengths.

### Optimum Needle Lengths

Injection Technique	Gas Supply System	
	Pressure reg./ needle valve	Flow reg./ back pres. reg.
Splitless	71 mm	71 mm
Split (flow rate >100 ml/min.)	25 mm	25 mm
Split (flow rate <100 ml/min.)	71 mm	25 mm
Split, high boiling matrix	25 mm	25 mm

Knowing how difficult it is to achieve complete sample transfer in splitless injection, this is certainly not the kind of problem we need.

### SAMPLE EVAPORATION INSIDE THE NEEDLE

As if the subject were not of sufficient complexity yet - the length of the syringe needle also influences sample evaporation. Parts of the sample may be vaporized inside the needle during injection or when the needle content is eluted after the plunger is fully depressed. On the one hand, this often causes problems as more is injected than measured and preferential vaporization of volatile components discriminates against high boilers. On the other hand, it helps nebulizing the sample liquid at the needle exit, which is the prerequisite for sample

The length of the syringe needle is more critical than usually recognized. Try and see! Although this has been known for more than 15 years, only a few autosamplers give you the choice of varying the injection point. Presumably this is because too many customers ask more about the software for data handling than about the gas chromatograph when they buy a new instrument. Today much emphasis is given to quality assurance. Large amounts of time are invested into general QA procedures, the usefulness of which is not always obvious. Upon such efforts, easily more important optimization of technical aspects is neglected.

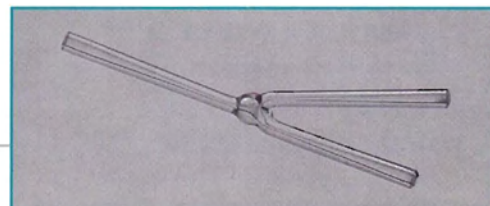


## PERFORMERS

- Alleviates column end connection strain.
- Inlet and outlet ends conform to the column radius.
- Perform confirmational analysis with a single injection.

### Universal Angled "Y" Press-Tight® Connector

cat.# 20403, each  
cat.# 20404, 3-pk.

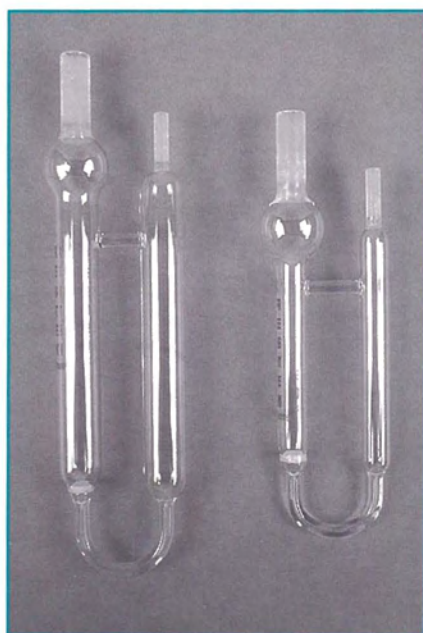






## Purge & Trap Spargers

- For Tekmar 2000, 3000 and ALS 2016/2032
- Available in 5 and 25ml sizes
- Uniform frits to ensure maximum purge efficiency



Restek now offers purge and trap fritted spargers for Tekmar concentrators. These spargers provide maximum purge efficiency for water samples. Each sparger is manufactured with tight tolerances to ensure a leak free seal. These spargers are not recommended for wastewater samples since the frit may become plugged.

### Spargers

5ml Fritted Sparger, 1/2" mount  
cat.# 21150, each

25ml Fritted Sparger, 1/2" mount  
cat.# 21151, each

## Restek's Thermal Gas Purifier

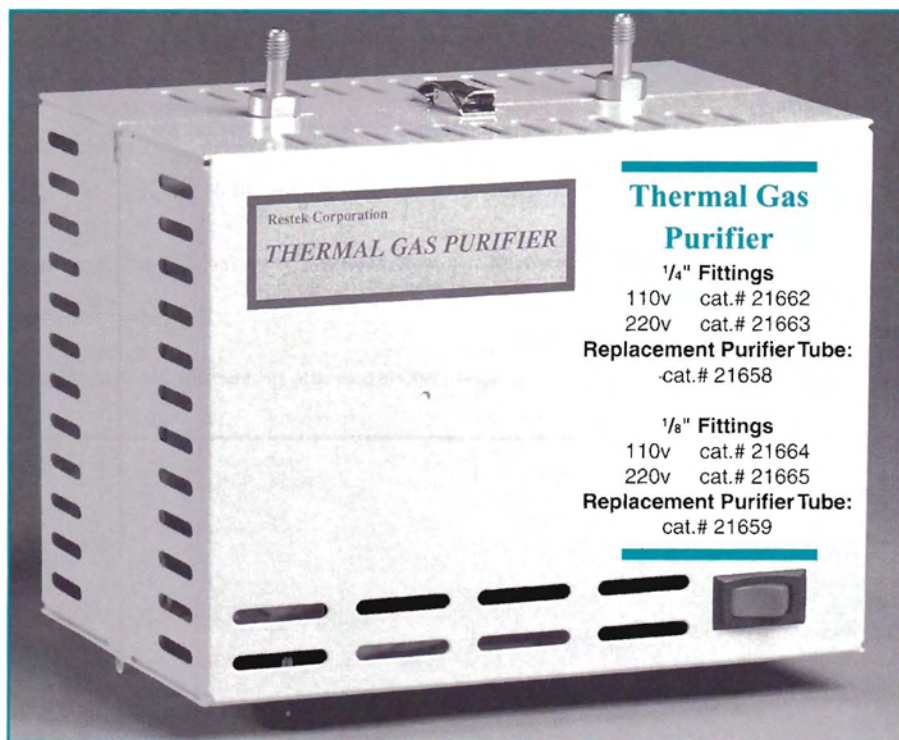
GC columns and detectors require pure carrier gases to operate at maximum sensitivity. With only 1% of trace contaminants in the carrier gas, column lifetime will be reduced. Restek has developed a new thermal gas purifier that removes trace contaminants from the carrier gas stream. This purifier removes oxygen, water, carbon monoxide and carbon dioxide by a high temperature reaction with purifier granules. compared to conventional adsorption traps. The constant high temperature reaction with the *getter* granules prevents contaminants from returning to the gas stream.

Each of Restek's purifier tubes are thermally cleaned prior to shipment and are

filled with N<sub>2</sub> and capped to ensure contamination free start-up. The purifier tubes have welded end fittings that eliminate leaks from occurring. Submicron frits are used in the end of each tube to prevent particles from entering and contaminating the carrier gas lines. One purifier removes 17 liters of oxygen and 42 liters of water vapor, allowing a single unit to service multiple instruments.

- Top mounted fittings ease installation & save bench space.
- Save money by scrubbing lower grade gases clean.
- Removes oxygen and water.
- Welded end fittings eliminate leaks.
- U-tube configuration eliminates channeling.
- UL certification (pending).

Restek's purifier design utilizes less bench space than comparable devices since all gas connections enter and exit the top of the unit. The "U-tube" configuration virtually eliminates channeling of the getter material ensuring maximum purification of the carrier gas and higher flow rates.



### Thermal Gas Purifier

#### 1/4" Fittings

110v cat.# 21662  
220v cat.# 21663

Replacement Purifier Tube:  
cat.# 21658

#### 1/8" Fittings

110v cat.# 21664  
220v cat.# 21665

Replacement Purifier Tube:  
cat.# 21659

*Note: Not recommended for use with flammable gases.*





" some" **promos / Products / Offers** in the **ADVNews**  
have been since been progressively superceded  
/ UPDATED OR Since Discontinued  
**CHECK** THE latest Restek ADVantage Newsletter, Restek ESSENTIALS  
... Or The Restek Catalog ... Or other Restek publications for updates  
**www.chromtech.net.au** or NEW site 2015 > **www.chromalytic.net.au**

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FAMEWAX™, PINNACLE™, Rtx®, MXT®, Integra-Guard™, Silcosteel®, Silcosleeve™, CarboFrit™, Silcoport™, Press-Tight®, Vu-Union™ and the Restek logo.

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# THE RESTEK

## ADVANTAGE



*Hot*

### **MXT®-500 Sim Dist** *New carborane stationary phase for High Temperature GC*

by Andy Schuyler

- MXT columns will not break like brittle Al-Clad columns.
- Safe for Hydrogen carrier gas.
- Low bleed and long life to 430°C.

Gas chromatographic analysis at temperatures above 400°C requires stationary phases and tubing that can withstand temperatures beyond the limits of most conventional polymers and tubing used in GC. By incorporating carborane into the backbone of the polymer chain, the thermal stability is increased (**Figure 1**). Because these slightly polar polymers are not pure dimethyl polysiloxanes like MXT-1, Restek uses the phase designation MXT-500 Sim Dist for this new stationary phase.

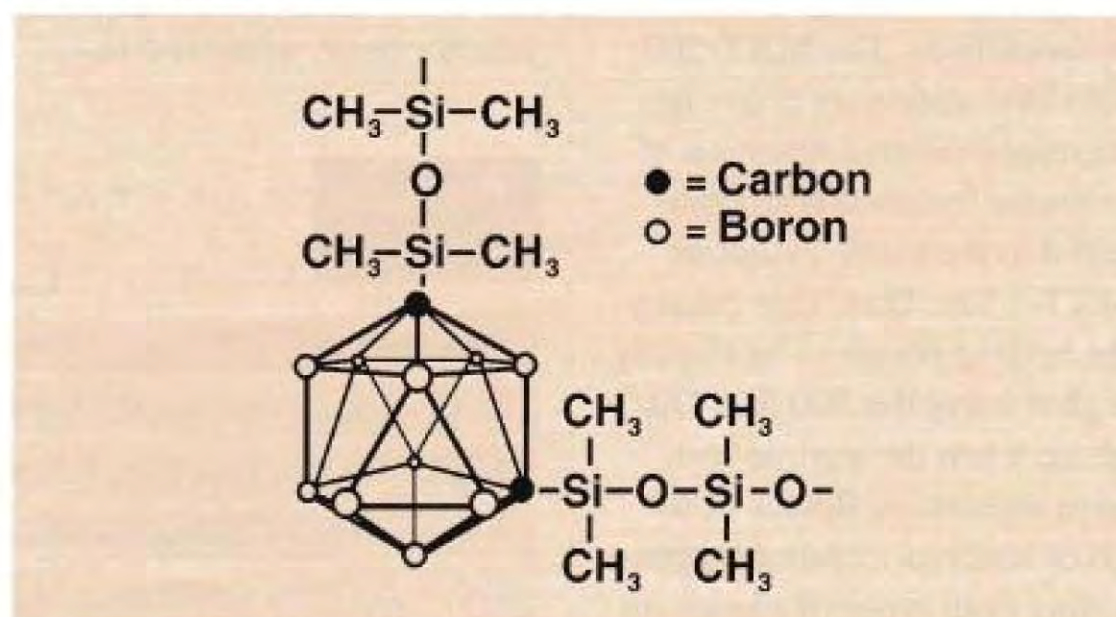
#### **Tubing Constraints**

An improved, high-temperature stationary phase is not enough; the tubing used is also an important consideration. The polyimide coating that keeps fused silica tubing flexible breaks down rapidly at oven temperatures above 360°C and is unsuitable for high temperature gas chromatography. Aluminum-clad tubing overcomes the problems with the polyimide, but has limitations. When repeatedly temperature programmed above 400°C or



**Figure 1:**

Carborane dimethyl polysiloxane—MXT-500 Sim Dist stationary phase.



Innovators of  
High Resolution  
Chromatography  
Products

### *in this issue*

MXT®-500 Sim Dist  
Column  
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**ECHNOLOGY** Pty Ltd

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FALL  
97



# MXT<sup>®</sup>-500 Sim Dist

Continued from page 1.

Tubing stable  
at ANY GC  
temperature!

Figure 2:

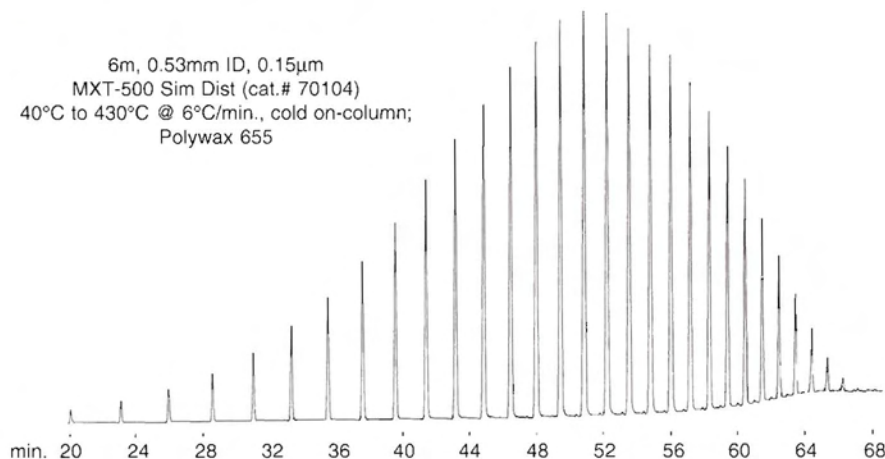
The MXT-500 Sim Dist column demonstrates low bleed and stable baseline to 430°C for high temperature Sim Dist calibration and analysis.

allowed to cool below 50°C, the aluminum sheath becomes brittle and eventually breaks. The most durable capillary columns available are Restek's MXT columns, which are manufactured using Silcosteel<sup>®</sup> tubing (metal tubing with the inertness of fused silica and the robustness of metal).

## MXT columns are designed for High Temperature Simulated Distillation

Simulated distillation (Sim Dist), one of the most common high temperature GC applications, is a good demonstration of the durability of MXT columns. Simulated distillation is a technique in which the GC is calibrated using the retention times of hydrocarbons that have published boiling points. The analysis of a high molecular weight petroleum sample, such as lubricating oil, is compared to the calibration by a special software program and the boiling range distribution is determined. Simulated distillation requires stable retention times under temperature programmed conditions and a baseline with low bleed that is repeatable with multiple temperature programmed analyses. **Figure 2** illustrates low and stable column bleed, excellent peak symmetry, and good recovery of the high molecular weight hydrocarbons in Polywax 655 on an MXT-500 Sim Dist column. Retention time and baseline stability are excellent indications that the polymer is stable. This column has been operated at 430°C isothermal for over 100 hours

6m, 0.53mm ID, 0.15µm  
MXT-500 Sim Dist (cat.# 70104)  
40°C to 430°C @ 6°C/min., cold on-column;  
Polywax 655



without significant retention time shift or baseline increase. And, of course, the MXT tubing will never become brittle!

## Restek offers MXT-1 and MXT-500 Sim Dist stationary phases for high temperature Sim Dist

Although the carborane 500 Sim Dist stationary phase is the most stable phase available for Sim Dist, many analysts prefer to use a true methyl silicone column for this analysis. Differences in polarity of the stationary phases cause a shift in the calculated boiling range distribution for petroleum samples containing aromatic hydrocarbons. The MXT-500 Sim Dist stationary phase has increased relative retention of aromatic hydrocarbons compared to the methyl silicone MXT-1 Sim Dist. This causes the boiling points to be slightly higher using the 500 Sim Dist phase when the sample contains aromatics. Restek now gives analysts a choice by providing both types of phases on

high temperature MXT columns. While both columns can be operated to 430°C, the MXT-500 Sim Dist column has lower bleed and longer life-time, while the MXT-1 offers methyl silicone polarity that matches many laboratories' historical data.

## Durable Silcosteel<sup>®</sup> tubing and stable stationary phases for High Temperature GC

High temperature GC challenges the limits of existing column and stationary phase technology. Restek's MXT tubing is ideally suited to the task when compared to fused silica or aluminum clad tubing, which cannot withstand re-

peated temperature programmed operation to 430°C. These temperatures also push GC polymers to the limit of thermal decomposition. But Restek's new MXT-500 and MXT-1 Sim Dist columns hold up well under these extreme conditions. When properly conditioned to 430°C, these columns give stable baselines with low bleed and repeatable retention times needed for high temp Sim Dist and other HTGC analyses.

## Product Listing:

### Columns

Description	cat.#	price
MXT-1 Sim Dist (6m, 0.53mm ID, 0.15µm)	70101	\$300
MXT-500 Sim Dist (6m, 0.53mm ID, 0.15µm)	70104	\$300
Polywax (655 Calibration Material 1 gm)	36225	\$10
Polywax (1000 Calibration Material 1 gm)	36227	\$10

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SCC~35C~10SS



# The Rtx<sup>®</sup>-OPPesticides Column

## Fast, efficient analysis of Organophosphorus Pesticides in EPA Method 8141A

by David Smith

- **Efficient analysis of 55 components in EPA Method 8141A. Includes: 49 organophosphorous pesticides, 4 internal standards, and 2 triazines.**
- **Quick analysis—less than 20 minute run time for 55 compounds.**
- **Flexible column configuration—0.32mm ID allows for direct injection with FPD or NPD detection and GC/MS confirmation.**
- **Maximum Temperature of 300°C.**

In contract analytical labs, time is money when analyzing client samples. The efficient throughput of samples in shorter peri-

ods of time results in higher revenue. Using the latest in method and phase development technologies, the chemists at

Restek have designed a unique phase for the efficient analysis of organophosphorus (OP) pesticides. The Rtx-OPPesticides can analyze the 55 components in EPA Method 8141A in under 20 minutes, which is 50% faster than other published analysis times.<sup>1</sup>

When the Rtx-OPPesticides column is run under the specified conditions, it exhibits four coelutions involving nine compounds. The coelutions will not affect identification and quantitation of GC/MS analysis. For more element-specific detectors, such as the NPD or

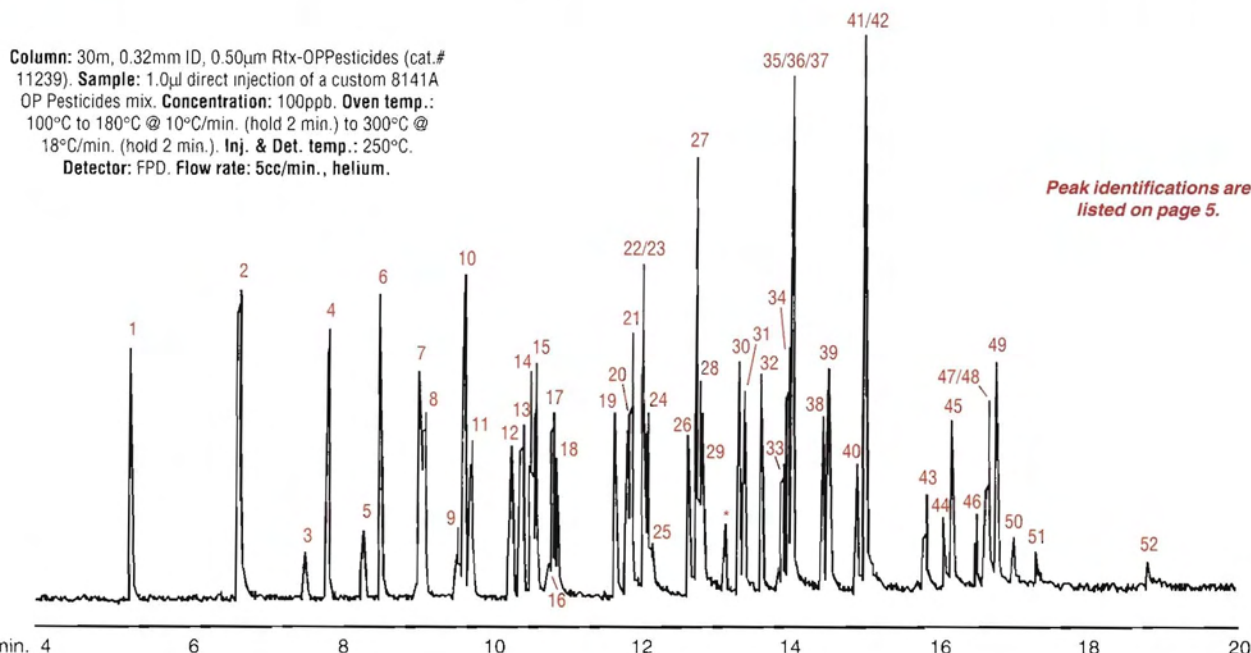
FPD, these coelutions may be of concern if the full compound list is analyzed, although the 27 compounds used for method 8141A validation are all resolved. In order to present a complete method for organophosphorus pesticides, the Rtx-35 was researched and chosen as a confirmational column (see Figure 3 on page 4).

An illustration of the organophosphorus pesticide separations achieved with the 30m, 0.32mm ID, 0.5µm Rtx-OPPesticides is shown in **Figure 1**. The concentration of the standard is 100ppb, which is

*Continued on page 4.*

**Figure 1:**

The Rtx<sup>®</sup>-OPPesticides column separates 43 organophosphorus pesticides at 100ppb in less than 20 minutes by FPD.





# The Rtx<sup>®</sup>-OPPesticides Column

Continued from page 3.

**Figure 2:**

The 0.32mm ID Rtx<sup>®</sup>-OPPesticides allows GC/MS separation of OP pesticides and triazines.

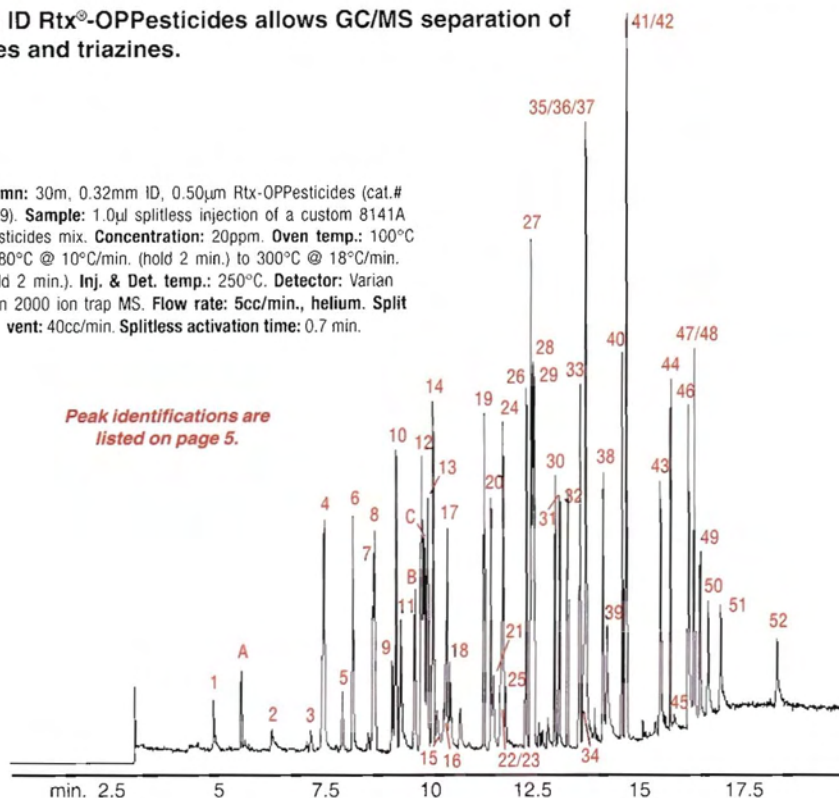
near the lowest concentration used in the development of calibration curves. The 100ppb standard shows the inertness of the column when analyzing low levels of organophosphorus pesticides. With this column, it is possible to analyze the OP pesticides in Method 8141A using a direct injection with run times under 20 minutes. The two triazine herbicides listed in Method 8141A, atrazine and simazine, are not detected using an FPD, but are resolved as shown in **Figure 2**.

The ion trap chromatogram shown in **Figure 2** exemplifies the flexibility of the Rtx-OPPesticides. The same dimension column used to

Continued on page 5.

**Column:** 30m, 0.32mm ID, 0.50µm Rtx-OPPesticides (cat.# 11239). **Sample:** 1.0µl splitless injection of a custom 8141A OP Pesticides mix. **Concentration:** 20ppm. **Oven temp.:** 100°C to 180°C @ 10°C/min. (hold 2 min.) to 300°C @ 18°C/min. (hold 2 min.). **Inj. & Det. temp.:** 250°C. **Detector:** Varian Saturn 2000 ion trap MS. **Flow rate:** 5cc/min., helium. **Split vent:** 40cc/min. **Splitless activation time:** 0.7 min.

Peak identifications are listed on page 5.

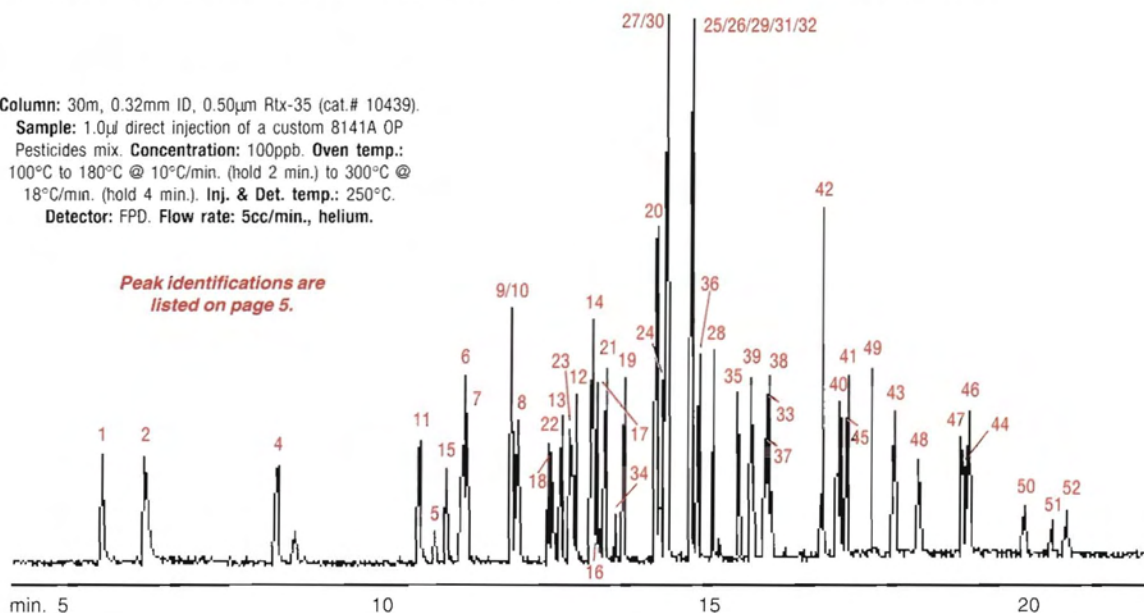


**Figure 3:**

An analysis time of less than 21 minutes makes the Rtx<sup>®</sup>-35 an excellent confirmational column.

**Column:** 30m, 0.32mm ID, 0.50µm Rtx-35 (cat.# 10439). **Sample:** 1.0µl direct injection of a custom 8141A OP Pesticides mix. **Concentration:** 100ppb. **Oven temp.:** 100°C to 180°C @ 10°C/min. (hold 2 min.) to 300°C @ 18°C/min. (hold 4 min.). **Inj. & Det. temp.:** 250°C. **Detector:** FPD. **Flow rate:** 5cc/min., helium.

Peak identifications are listed on page 5.







achieve fast separation of OP pesticides with element-specific detection is used to gain valuable structural information with mass spectrometer detection.

An outstanding confirmational column for OP pesticide analysis is a standard Rtx-35 of the same Rtx-OPPesticides dimension (30m, 0.32mm, 0.5µm). Using the identical temperature and flow conditions shown in **Figures 1 and 2**, the Rtx-35 chromatogram in **Figure 3** has a run time of just over 20 minutes, and resolves all coeluting

compounds shown in the Rtx-OPPesticides chromatogram.

The Rtx-OPPesticides is the latest in GC stationary phase innovation. This phase gives an efficient analysis of 55 organophosphorus pesticides in under 20 minutes. And one column can do it all—direct injection on FPD or NPD for high sensitivity and splitless injection on a GC/MS for structural identity. Only from Restek!

<sup>1</sup>C. George, *Separation Times*, 11, 1 (1997) 8-10.

#### Peak List for Figures 1-3

- |                                  |   |
|----------------------------------|---|
| 1. dichlorvos                    | 31. malathion                                 |
| 2. hexamethylphosphoramide       | 32. fenitrothion                              |
| 3. trichlofon                    | 33. tokuthion                                 |
| 4. mevinphos                     | 34. phosphomidon                              |
| 5. demeton-S                     | 35. chlofenvinphos                            |
| 6. zinophos                      | 36. parathion                                 |
| 7. ethoprop                      | 37. merphos oxone (merphos breakdown product) |
| 8. phorate                       | 38. stirophos                                 |
| 9. naled                         | 39. crotoxyphos                               |
| 10. sulfotepp                    | 40. bolstar                                   |
| 11. tributylphosphate (standard) | 41. carbophenthion                            |
| 12. diazinon                     | 42. ethion                                    |
| 13. terbufos                     | 43. triphenylphosphate (standard)             |
| 14. fonofos                      | 44. leptophos                                 |
| 15. TEPP                         | 45. fensulfothion                             |
| 16. dioxation                    | 46. tri-o-cresyl phosphate                    |
| 17. disulfoton                   | 47. phosmet                                   |
| 18. demeton-O                    | 48. EPN                                       |
| 19. dichlofenthion               | 49. famfur                                    |
| 20. chlorpyrifos methyl          | 50. azinphos methyl                           |
| 21. dimethoate                   | 51. azinphos ethyl                            |
| 22. dicrotophos                  | 52. coumaphos                                 |
| 23. monocrotophos                |   |
| 24. ronnel                       |   |
| 25. merphos                      | * phosphomidon breakdown product              |
| 26. chlorpyrifos                 |   |
| 27. aspon                        |   |
| 28. fenthion                     | Nitrogen-containing compounds                 |
| 29. trichloronate                | A. 1-bromo-2-nitrobenzene                     |
| 30. methyl parathion             | B. simazine                                   |
|                                  | C. atrazine                                   |

#### Product Listing:

##### 30m, 0.32mm ID, 0.50µm Columns

Rtx®-OPPesticides: Cat.# 11239, \$475

Rtx®-35: Cat.# 10439, \$415

#### Analytical Reference Materials

##### 8140/8141 OP Pesticide Calibration Mix A

azinphos methyl	fenthion
bolstar (sulprofos)	merphos
chlorpyrifos	methyl parathion
coumaphos	mevinphos
demeton, O and S	naled
diazinon	phorate
dichlorvos	ronnel
disulfoton	stirofos
ethoprop	tokuthion (prothiofos)
fensulfothion	trichloronate

200µg/ml ea. in hexane/acetone (95%/5%), 1ml/ampul

	Each	5-pk.	10-pk.
	32277 \$90	32277-510 \$405	
w/data pack	32277-500 \$100	32277-520 \$450	32377 \$810

##### 8141 OP Pesticide Calibration Mix B

dimethoate	parathion
EPN	sulfotepp
malathion	TEPP
monocrotophos	

200µg/ml ea. in hexane/acetone (95%/5%), 1ml/ampul

	Each	5-pk.	10-pk.
	32278 \$55	32278-510 \$247.50	
w/data pack	32278-500 \$65	32278-520 \$275	32378 \$495

#### 8140/8141 Internal Standards & Surrogates

1000µg/ml in acetone, 1ml/ampul

##### 1-bromo-2-nitrobenzene Standard

(Recommended 8141A NPD Internal Standard)

	Each	5-pk.	10-pk.
	32279 \$20	32279-510 \$90	
w/data pack	32279-500 \$30	32279-520 \$100	32379 \$180

##### tributylphosphate Standard

(Recommended 8141A FPD Surrogate)

	Each	5-pk.	10-pk.
	32280 \$20	32280-510 \$90	
w/data pack	32280-500 \$30	32280-520 \$100	32380 \$180

##### triphenylphosphate Standard

(Recommended 8141A FPD Surrogate)

	Each	5-pk.	10-pk.
	32281 \$20	32281-510 \$90	
w/data pack	32281-500 \$30	32281-520 \$100	32381 \$180

##### 4-chloro-3-nitrobenzotrifluoride Standard

(Recommended 8141A NPD Surrogate)

	Each	5-pk.	10-pk.
	32282 \$20	32282-510 \$90	
w/data pack	32282-500 \$30	32282-520 \$100	32382 \$180



# SilcoCan™—The Ideal Canister for Sulfur Compound Storage

by Dave Shelow

7-Day  
Stability at  
Concentrations as  
Low as 1 ppm

Sulfur compounds are emitted from a variety of sources including petrochemical processes, land fills, and stack emissions. Because of their odor, these compounds are a nuisance. They frequently require air monitoring and analysis.

Collection of air samples containing trace levels of sulfur compounds is difficult because they readily react with stainless steel sampling vessels such as Summa® Canisters. Because of this reactivity with stainless steel, Tedlar® bags have been used for collection of sulfur compounds. However, the stability of these compounds in Tedlar® bags is limited to 24-48 hours.

Restek's Silcosteel®-lined SilcoCan™ canister is the ultimate solution for long term storage of air samples containing sulfur compounds. Silcosteel is a unique process that chemically bonds a layer of fused silica material to the stainless steel surface, reducing adsorption and breakdown of active compounds. The SilcoCan air sampling canister has been shown to maintain the stability of trace level sulfur compounds up to seven days with little or no degradation.

A stability study of six common sulfur compounds was recently conducted by the Bay Area Air Quality Management District. These compounds were spiked at two concentration levels into SilcoCan air

sampling canisters and measured at time intervals of 1, 2, 3, 4, and 7 days. The results of this study are shown in Figures 2 and 3. The data clearly shows that even after seven days of storage in a SilcoCan canister, over 90% of these six sulfur compounds were successfully recovered.

Since any stainless steel surfaces that come into contact with sulfur compounds will cause adsorption, a SilcoCan canister with a Silcosteel-treated valve is recommended. Figure 4 shows a Silcosteel-treated diaphragm valve. All internal parts that come into contact with the sample have been Silcosteel-treated. Also, any portion of the sampling pathway, such as the flow controller or tubing, should also be Silcosteel-treated. For more information about Restek's Silcosteel process, please contact our Technical Service team or your local Restek representative.

Collection and storage of highly adsorptive sulfur compounds is no longer a problem with Restek's SilcoCan canister. Silcosteel technology reduces the adsorptive character-

Figure 1:

The Silcosteel® lining in the SilcoCan™ canister reduces adsorption of sulfur compounds.



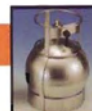
For more information,  
request a free  
copy of Restek's  
Air Monitoring  
Products Guide.

Restek Corporation

• 6 •

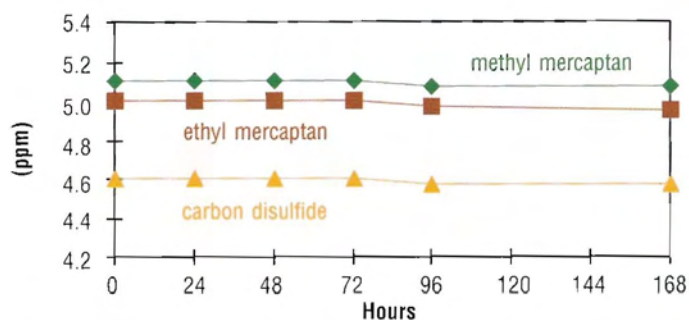
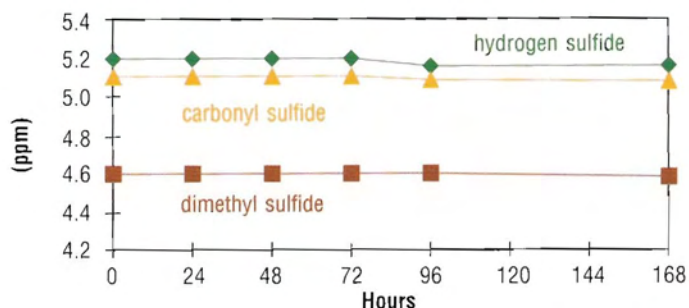
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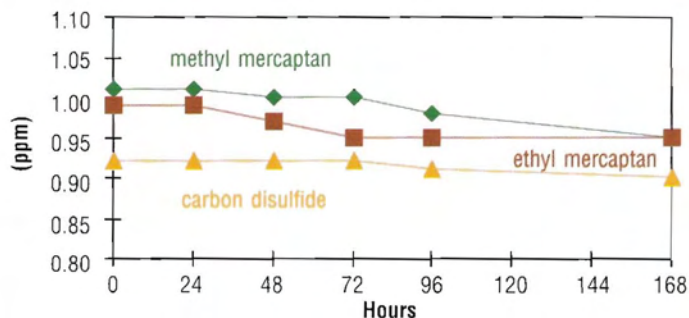
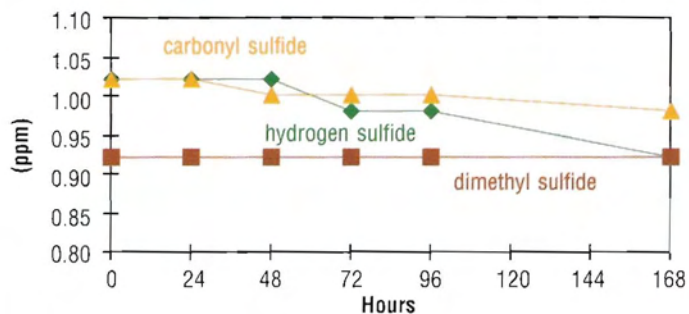
**Figure 2:**

No significant loss of sulfur compounds when stored in a SilcoCan™ for up to 7 days.



**Figure 3:**

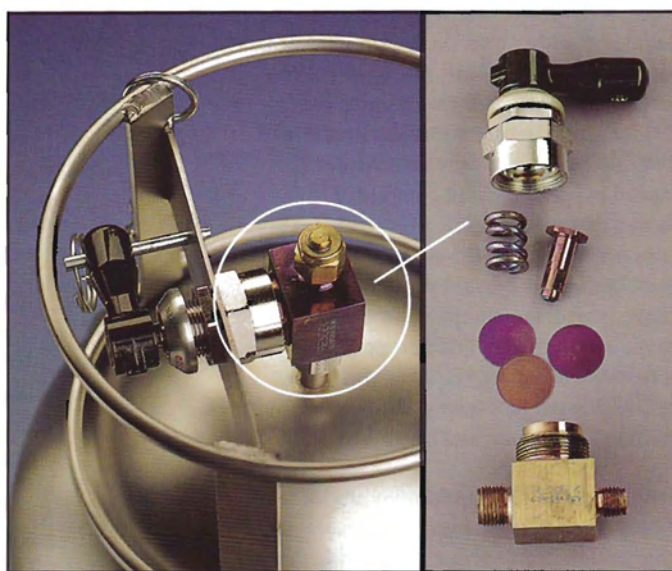
Even 1ppm of sulfur compounds is recovered from a SilcoCan™ canister after 7 days.



Since any stainless steel surfaces that come into contact with sulfur compounds will cause adsorption, a SilcoCan canister with a Silcosteel-treated valve is recommended.

**Figure 4:**

A Silcosteel®-treated diaphragm valve insures a completely inert sample pathway.



**Product Listing:**

**SilcoCan™ Canisters with Silcosteel®-treated Valves**

Sizes	Cat.#	Price
1.0 Liter	24201-650	\$510
1.8 Liter	24202-650	\$520
3.0 Liter	24203-650	\$530
6.0 Liter	24200-650	\$550
15.0 Liter	24204-650	\$850

Silcosteel® Replacement Diaphragm Valve: cat.# 24221, \$305



# Organo Tin Analy

by Frank Dorman

Figure 1/2:

500 pg Organo Tin Compounds on the Rtx®-5 and Rtx®-35 Columns by GC-FPD.

Tributyl tin was commonly used as an antifouling agent in marine paint, as well as a pesticide and fungicide before its use was discontinued in the 1980's. Tributyl tin has since been found to bioaccumulate and cause a number of health-related problems, and has been recently added to the growing list of possible endocrine disrupting compounds.<sup>1</sup> As awareness of endocrine disruptors grows, and shipyards are remediated, many environmental laboratories are faced with requests for the analysis of tributyl tin and its breakdown products. Unfortunately, there is no "EPA accepted" method for this, so most laboratories have decided to either pass on these requests, or subcontract the analysis to one of the few laboratories that perform this test. Generally, laboratories are under the impression that this analysis requires a considerable capital expenditure and complex techniques that would result in high cost. This does not have to be the case, however, and most laboratories could perform this analysis with the equipment they already have. The purpose of this proposed method is to make this analysis "available" to an environmental laboratory at low cost using common glassware and instrumentation.

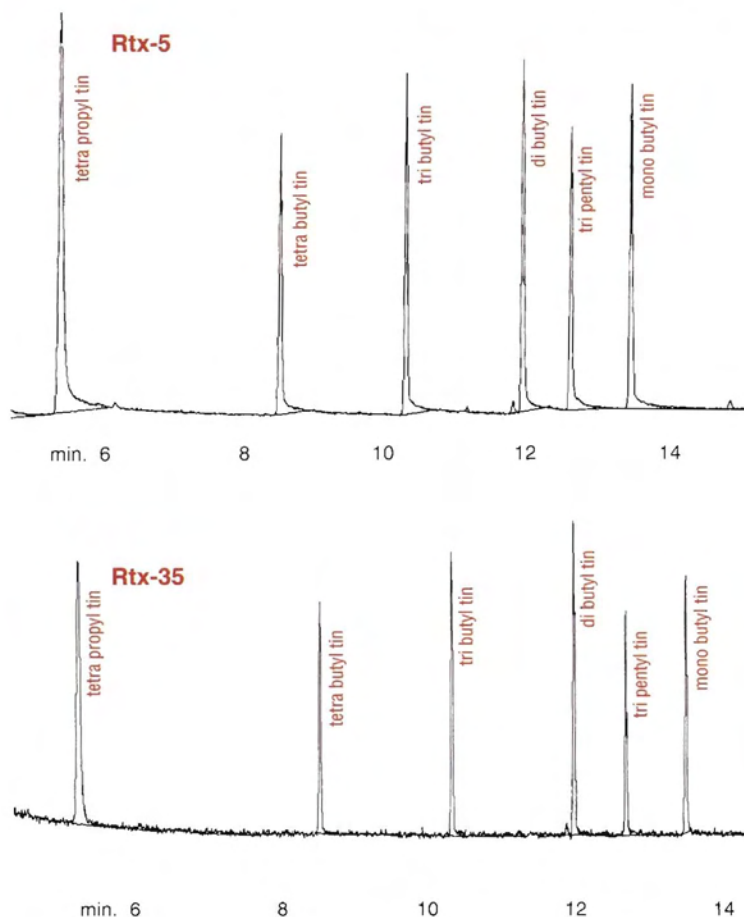
Tributyl tin and its breakdown products of dibutyl tin and monobutyl tin present a preparation problem due to the wide range of polarity. These compounds usually exist as chlorides, and it is difficult to completely extract all of them quantitatively from the sample matrix, although there has been some promising data from open-vessel microwave extraction techniques. What is possible, however, is to quantitatively extract the tetra, tri, and di butyl tin, and achieve reasonable and reproducible extraction of the monobutyl tin.

For this method it is very important to remove as much of the potential interferences as possible through a thorough extract cleanup. The primary interference is from sulfur-containing compounds, and

these can be at relatively high concentration compared to the organo tin compounds. The 16 gram Florisil and 5 gram silica gel method<sup>2</sup> has a large capacity and works well for all three sample matrices, water, soil, and biota. The cleanup column can be made in glass prep-scale chromatography columns, or purchased as SPE cartridges from Restek (cat.# 53305). In either case, the extract is applied to a hexane-wetted column, and eluted using 100 mls of hexane. The extract is again collected and the internal standard, tetra-n-

propyl tin, is added before final concentration.

There are many reported methods of analysis in the literature, but since the goal of this method was to be adaptable to an environmental laboratory, gas chromatography (GC) with flame photometric detection (FPD) was chosen. The FPD must be operated under fuel-rich conditions for efficient conversion of the alkyl tin compounds into tin hydrides. The only other necessary modification is to use a



30m, 0.32mm ID, 1.0µm Rtx-5 and Rtx-35 columns (cat.# 10254 and 10454)  
3µl direct injection. Concentration: ~500pg on-column. Head pressure: 15 psi, constant.  
Oven temp.: 100°C (hold 1 min.) to 285°C @ 10°C/min. hold 10 min., Inj. & det. temp.: 250°C; Carrier gas: He

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• 8 •

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# by Capillary GC

New Method  
from RESTEK!



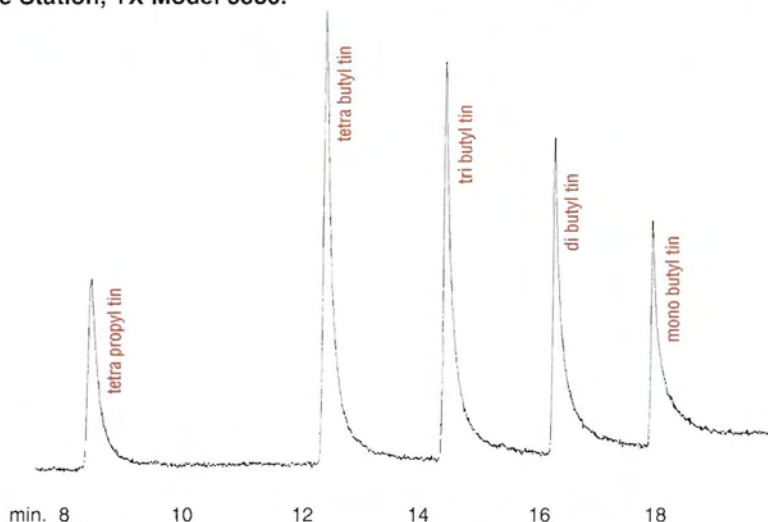
Figure 3:

Commercial Laboratory Results using the Proposed Method from Restek.

Compound	Water Extraction Recovery (%)	Soil Extraction Recovery (%)	Restek SPE-Cleanup Recovery (%)	MDL Liquid (ng/L)	MDL Soil (µg/Kg)
tetrabutyl tin	83	86	92	29.9	0.45
tributyl tin	110	96	99	20.9	0.39
dibutyl tin	75	66	96	15.7	0.46
tripentyl tin (SSTD)	NA	NA	101	NA	NA
monobutyl tin	38	36	118	19.6	0.14

Figure 4:

Organo Tin compounds using Pulsed FPD. Detector courtesy of O.I. Analytical, College Station, TX Model 5380.



30m, 0.32mm ID, 1.0µm Rtx-35 (cat.# 10454). Concentration: 5pg on-column. Head pressure: 15 psi, constant. Oven temp.: 100°C (hold 1 min.) to 285°C @ 10°C/min. hold 10 min., Detector: PFPD from O.I. Analytical Corp.

610 nm wavelength filter to collect the molecular emission from the tin hydride. Tin chlorides are analyzed as hexyl derivatives which are formed by a Grignard reaction using n-hexyl magnesium bromide. The surrogate tri-pentyl-tin chloride is added to the sample prior to extraction and tetra-n-propyl-tin is the recommended internal standard. The calibration compounds, surrogate and internal standard solutions are available from Restek as custom reference materials.

Figures 1 and 2 show the resulting chromatograms from the mid-point calibration standard on the Rtx-5 and the Rtx-35 columns by GC-FPD. Figure 3 shows the method performance obtained by a commercial laboratory using this method.<sup>3</sup>

The method presented easily meets the requirements of 50 ng/L. In order to meet a possible proposed detection limit of 1ng/L, some method modification will be necessary. The easiest modification would

be to switch from using a regular FPD to a pulsed FPD (PFPD) detector. This detector gives a sensitivity enhancement of 10 to 100 times over the standard FPD for the organo tin compounds. Figure 4 shows the chromatogram obtained for 5 pg of each tin compound on the Rtx-35 using PFPD detection. Comparing this chromatogram to Figure 1 and 2 it is observed that a similar signal to noise ratio is obtained with 100 times less material. This demonstrates roughly an increase of 100 in sensitivity using the PFPD with the same preparation method, resulting in the ability to meet the proposed 1 ng/L detection limit being considered by the EPA.

In summary, this method allows laboratories to perform organo tin analysis with minimal start up and implementation costs. It is reliable, rugged, and utilizes equipment that most laboratories already have. To meet current and proposed detection limits, it is not necessary to use tandem MS or GC-AED which have high purchase and operating costs, and are typically not found in most laboratories. Finally, Restek can provide the technical training, and supplies required to perform this method so that literature and vendor research is not required.

<sup>1</sup> Special Report on Environmental Endocrine Disruption: An Effects Assessment and Analysis, EPA/630/R-96/012, Feb. 1997.

<sup>2</sup> Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Vol. IV, NOAA Technical Memorandum, NOS ORCA 71

<sup>3</sup> ITS - Environmental, 55 South Park Drive, Colchester, VT 05446.

## Product Listing:

### Columns and Accessories

30m, 0.32mm ID, 0.50µm Rtx-5:  
cat.# 10239, \$415

30m, 0.32mm ID, 0.50µm Rtx-35:  
cat.# 10439, \$415

Florisil/silica gel SPE cartridge:  
cat.# 24049, \$120 16-pack

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800-356-1688



# Koni's Korner

## Carrier Gases for GC

by Dr. Konrad Grob

Probably more than 90% of the present GC instruments run with helium as carrier gas. Some people use hydrogen or nitrogen, maybe because the first ones are hidden pyromaniacs (some GC ovens actually exploded) and the second still have nitrogen mounted on the instrument from the times they worked with packed columns. These gases serve to produce wind through the column to move our solutes forward. The solute molecules evaporate from the stationary phase surface, i.e. enter the open space of the capillary column, are hit by a carrier gas molecule and start traveling down the tube. After a short distance, however, they touch the sticky surface of the stationary phase and go through another partitioning process. Does the choice of the carrier gas interfere with this? Yes, it does, through its diffusivity and viscosity. You want to know why hydrogen is the best carrier gas?

### Diffusivity

Diffusivity provides a measurement for the diffusion speed of a solute vapor in a given gas. For helium and hydrogen, diffusivities are similar, but that of nitrogen is about four times lower (see Table I).

The diffusion speed of the solute in the carrier gas determines the speed of chromatography. A solute molecule evaporating from the

stationary phase surface into the gas stream should be given enough time to diffuse back to the stationary phase (Figure 1) before having gone far in order to undergo another partitioning process - it is these contacts which differentiate between different substances, and a

large number of contacts are needed to obtain the best separation. We get more of them if the solute diffuses more rapidly and/or when we give it more time, i.e. reduce the gas velocity. However, there is a limit: giving it more time for the diffusion towards the

stationary phase (radial diffusion) also provides more time for spreading within the open bore of the column, i.e. for band broadening through longitudinal diffusion. This is why there is an optimum gas velocity: it provides a maximum number of contacts with the stationary phase with a minimum of band broadening in the gas phase.

This kind of logic applies to all gases. In fact, all carrier gases provide similar separation efficiencies - provided conditions are adjusted correspondingly. The time needed is different: since diffusion in hydrogen and helium is much faster than in nitrogen—for (wanted) radial as well as (unavoidable) longitudinal diffusion—GC is 2-3 times faster with the former. If we users of hydrogen wait for one hour, users of nitrogen should wait for 2-3 hours to get the same performance. Nitrogen is for those who own a comfortable arm chair in the lab or who are afraid of the result. Usually users of nitrogen are not really that patient and run their chromatography at similar speed as others using hydrogen and helium. Table II shows what they get. It compares separation efficiencies measured in terms of Trennzahl (TZ) indicating the number of peaks which could be fully separated between two components to be defined, in this case, the alkanes C<sub>13</sub> and C<sub>14</sub>. At the gas velocities most commonly used with hydrogen (40-60 cm/s), nitrogen produced hardly more than half as many peaks. When using

Table I:

Relevant characteristics of carrier gases<sup>1</sup>

Carrier gas	Viscosity at 50°C [kg/s m]	Diffusivity (butane, 100°C [m <sup>2</sup> s])
Hydrogen	9.4	6 · 10 <sup>-6</sup>
Helium	20.8	5.5 · 10 <sup>-6</sup>
Nitrogen	18.8	1.5 · 10 <sup>-6</sup>

Figure 1:

Diffusion of a molecule in the gas phase of the column.

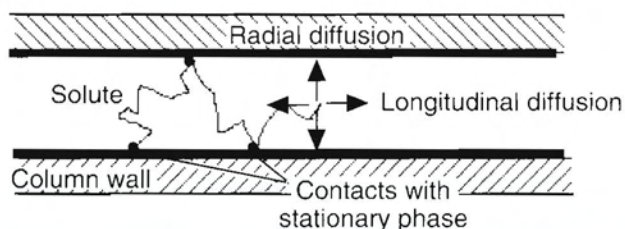


Table II:

Separation efficiencies in terms of separation numbers (Trennzahl, TZ) for the n-alkanes C<sub>13</sub> and C<sub>14</sub> and a 12m, 0.25mm ID column coated with a methyl silicone.

Gas velocity	Hydrogen	Nitrogen
50 cm/s	24	13
40 cm/s	25	15
30 cm/s	23	17
20 cm/s	20	23





hydrogen, the same result could have been obtained from a column roughly 3 times shorter in a third of the time. To give an impression of how the chromatograms look like, an example is shown in Figure 2. At halved velocity, nitrogen provided good performance also.

In this application, nitrogen just requires extra time. However, long retention times also produce low peaks, i.e. poor sensitivity (see Figure 2). Additionally, do not try to run triglycerides or other labile compounds with nitrogen as carrier gas: they are largely

degraded during the long run time required.

### Viscosity

The other difference between the carrier gases concerns the viscosity that determines the inlet pressure required for a given gas velocity. High inlet pressures strongly compress the gas in the column inlet, which causes the problems shortly outlined below. This explains why hydrogen is preferable to helium. You have certainly seen the  $h/u$  curves, also called van Deemter curves, plotting HETP (plate height) against gas velocity. Their peculiarity: the

best is at the bottom, i.e. the optimum gas velocity is at the lowest point of the curve; the larger the plate heights, the worse the separation. The curves say that separation is poor when the gas velocity is below the optimum velocity (left of the optimum in Figure 3, the result of excessive longitudinal diffusion) and that it worsens again beyond that optimum (the curve rising at the right, the result of insufficient radial diffusion).

For columns of a given diameter, the optimum velocity is highest when the column is short. This is because inlet pressure is low. For hydrogen or helium, with about the same diffusivity, the optimum is almost the same, i.e. around

40-50 cm/s. Further, the losses in performance upon speeding, i.e. using excessive gas velocity, are relatively small. The longer the column, the higher is the inlet pressure required. This shifts the optimum gas speed to lower values and, as if there were a strict educator behind the chromatographer, speeding is punished more strongly when the velocity must be low anyway. Hence, using a column of doubled length requires more than twice as much run time, because the gas velocity must be lower. In this respect, helium is worse than hydrogen because its viscosity is about twice as high: the higher inlet pressure requires a lower gas velocity and if you do not obey, the punishment is harder.

Figure 2:

Separation of a kerosene fraction using hydrogen or nitrogen as carrier gas at the same average gas velocity (40 cm/s).

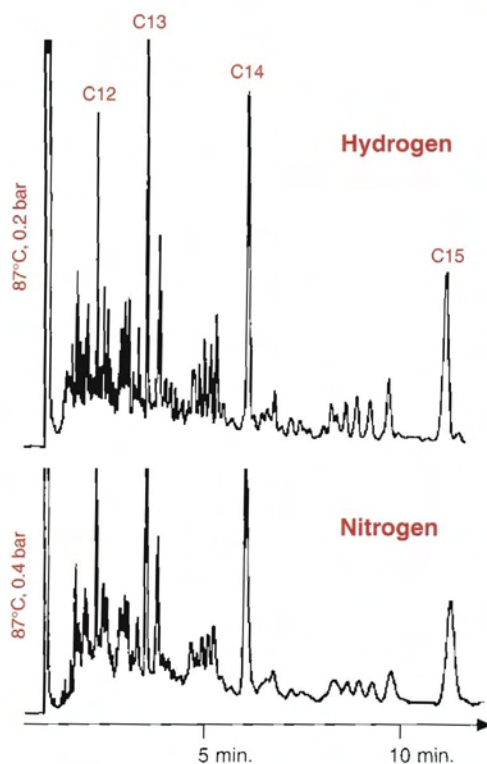
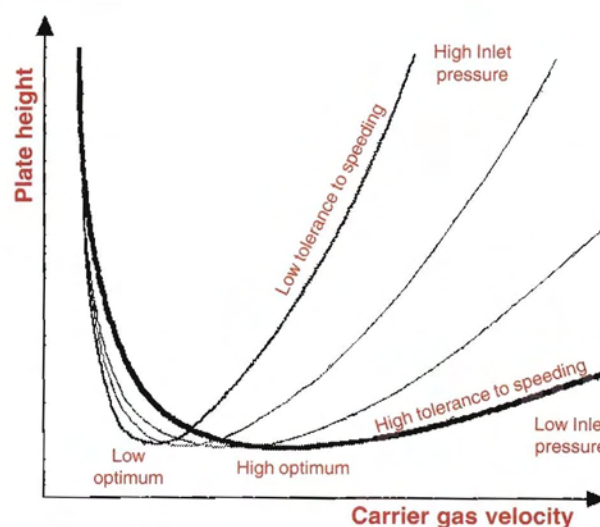


Figure 3:

High inlet pressures cause the optimum gas velocity to be low and the loss in separation efficiency when exceeding this optimum to be high.







## Koni's Korner

Continued from page 11.

What is the reason for this? If the column head pressure is, e.g., 1 bar, corresponding to 2 bar absolute pressure, the carrier gas in the inlet is compressed to half the volume compared to the column outlet (assuming the latter is at ambient pressure, 1 bar absolute, **Figure 4**). Hence the plug corresponding to 2 ml in the outlet is only 1 ml and is half as long. To displace 1 ml, half the velocity is required compared to displacing 2 ml at the outlet. Hence optimization must compromise between a low velocity in the inlet and a higher one at the outlet.

Conclusions are against intuition. From short columns we know that 40-50 cm/s are best. In the last, e.g., 15 m of a long column, pressure conditions are the same as in a short column, i.e. the optimum gas velocity and tolerance to

speeding must be the same. The problem resulting from the compressibility of the gas is obviously in the inlet of the long column. We are tempted to assume that it is related to the fact that the gas velocity is 20-25 cm/s only and would conclude that a compromise should be chosen between maybe 30 cm/s in the inlet and 70 cm/s in the outlet in order to result in some 50 cm/s as an average. Experiments show that this is wrong: the best average velocity is only 20-25 cm/s. Hence the system wants an even lower velocity in the inlet: about 10 cm/s. And it insists in that: it forces to choose a velocity at the outlet lower than found to be optimum, and if you do not obey to the 10 cm/s in the inlet, punishment is hard. A rapid glance into the above h/u curve shows that 10 cm/s would provide extremely poor performance at the column outlet. Thus the correct

conclusion is that optimum velocities are far lower in a compressed gas. This is not really new: GC with vacuum at the outlet, e.g. with GC-MS, is even faster.

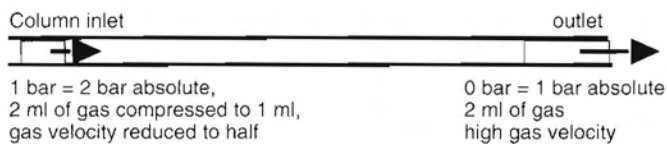
Nitrogen has only drawbacks and is not suitable for capillary GC. Helium is as good as hydrogen if inlet pressures are below about 50 kPa, but

requires slower GC at higher inlet pressures (for longer columns), the difference being roughly a factor of two when 150-200 kPa must be applied for helium.

The most important argument against the use of hydrogen concerns safety. The next "Korner" will report on how our lab solved that problem.

**Figure 4:**

**Compressibility of the carrier gas causes the gas velocity in the inlet to be lower than in the outlet.**



*from Rohrschneider, Ullmanns Enzyklopädie der technischen Chemie, Vol. 5.*

**I welcome your feedback. Reach me by e-mail at**  
**Koni@grob.org.**

## Capillary GC Reference Books

by Dr. Konrad Grob

### **On-Column Injection in Capillary Gas Chromatography, 2nd Edition**

*Basic Technique; Retention Gaps; Solvent Effects (Konrad Grob)*

On-column injections minimize detrimental adsorption and non-linearity problems associated with split/splitless techniques. Grob's text is a *must-read* treatise for the novice as well as for the experienced chromatographer. Basic technique is explained clearly with excellent schematics.

Huethig Publishing, Ltd., 1987 • 591pp.  
 cat.# 20453, \$130.75 ea.

### **Split and Splitless Injection in Capillary GC, 3rd Edition**

*(Konrad Grob)*

Represents one of the most comprehensive, single-volume treatment of all aspects of split and splitless injection. The book is divided into four sections: split injection, splitless injection, problems arising from the heated syringe needle in vaporizing injection, and Programmed Temperature Vaporizing (PTV) injection.

Huethig Publishing, Ltd., 1993 • 547pp.  
 cat.# 20451, \$98.50 ea.



# Inertsil™ HPLC Columns

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by Matt Piserchio

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- Efficient, reproducible packing.

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Figure 1:

## Sulfa Drugs

1. sulfathiazole
2. sulfamethizole
3. sulfadimethoxine
4. sulfaquinoxaline

Inertsil ODS-2  
5µm, 150 x 4.6mm ID  
(cat.# 9000006). Eluent: 5mM  
1-heptanesulfonate Na in 28%  
CH<sub>3</sub>OH. Flow rate: 1.2 mL/min.  
Detector: 254nm. Col. temp.: RT.

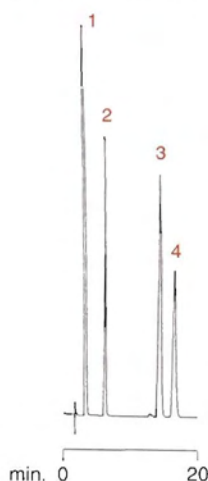
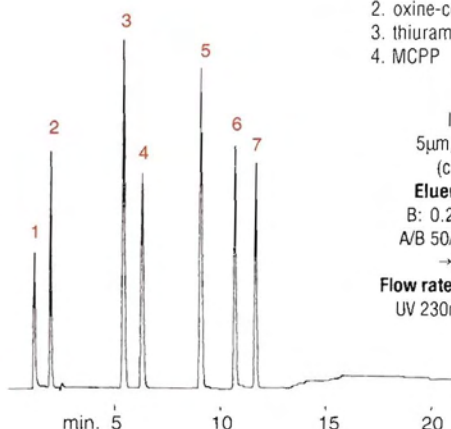


Figure 2:

## Golf Course Pesticides

1. asulam
2. oxine-copper
3. thiuram
4. MCPP
5. iprodione
6. bensulide
7. penicuron

Inertsil ODS-3  
5µm, 150 x 4.6mm ID  
(cat.# 9000001).  
Eluent: A: 0.2% H<sub>3</sub>PO<sub>4</sub>  
B: 0.2% H<sub>3</sub>PO<sub>4</sub> in CH<sub>3</sub>CN  
A/B 50/50 → (10min) → 10/90  
→ (10min) → 10/90  
Flow rate: 1.0 mL/min. Detector:  
UV 230nm. Col. temp.: 40°C.



Each lot of Inertsil reverse-phase bonded phases must pass several critical tests for inertness and stability prior to release for sale. First, packings are evaluated for inertness by observing the elution order and peak symmetry of pyridine in a mixture with phenol, methylbenzoate, and 3-methylindole. Then, a mixture of oxine copper and caffeine is injected and evaluated for peak shape and reproducibility to check for residual metals that can irreversibly adsorb certain compounds. After a check for selectivity, all bonded phases are subjected to both pH 2 and pH 9 to determine their chemical stability. Finally, columns are packed using proprietary techniques developed to produce highly efficient, reproducible HPLC columns.

Each Inertsil HPLC column is backed by the same expert, friendly, technical support and customer service that you have come to expect from Restek. Call us today at 800-356-1688, ext. 2157, for more information about Inertsil or any other Restek HPLC product.

## Product Listing:

### Inertsil Columns

Phase	Particle Size	Dimensions	Cat.#	Price
ODS-3	5µm	150 x 4.6mm	9000001	\$360
	5µm	250 x 4.6mm	9000002	\$395
ODS-2	5µm	150 x 4.6mm	9000006	\$360
	5µm	250 x 4.6mm	9000007	\$395
C8	5µm	150 x 4.6mm	9000010	\$360
	5µm	250 x 4.6mm	9000011	\$395
C4	5µm	150 x 4.6mm	9000014	\$360
	5µm	250 x 4.6mm	9000015	\$395
Phenyl	5µm	150 x 4.6mm	9000018	\$360
	5µm	250 x 4.6mm	9000019	\$395
Silica	5µm	150 x 4.6mm	9000022	\$360
	5µm	250 x 4.6mm	9000023	\$395

### Inertsil Guards

Phase	Particle Size	1cm Cartridges (Pack of 2)	Guard Set (Holder + Guards)
ODS-3	5µm	9000004 \$160	9000005 \$250
ODS-2	5µm	9000008 \$160	9000009 \$250
C8	5µm	9000012 \$160	9000013 \$250
C4	5µm	9000016 \$160	9000017 \$250
Phenyl	5µm	9000020 \$160	9000021 \$250
Silica	5µm	9000024 \$160	9000025 \$250

Cartridge Guard Holder: cat.# 9000003, \$110

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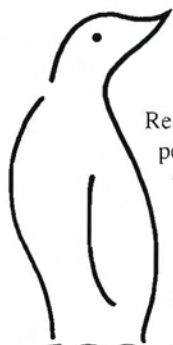


# Peak Performers

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#### IceBlue™ Septa

Septa Size	50-Pack Cat.#/price	100-Pack Cat.#/price
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10mm	22390 \$35	22391 \$65
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12.5mm	22394 \$35	22395 \$65
17mm	22396 \$35	22397 \$65
Shimadzu Plug	22398 \$35	22399 \$65

\*Add suffix #247 to the appropriate cat.# for a free 3-pack sample.

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Whether you require a 10.0 or 10.6 eV potential, the Model 108-10.0/10.6 will be right for your application needs. The Model 108 lamp has a 0.781" diameter base and is used in Tracor, OIC and Baseline instruments. The Model 103 lamp has a 1.375" base diameter and is used in HNU and SRI detectors. A Lamp Cleaning Kit is available that contains aluminum oxide cleaning compound, swabs and instructions.

Description	Cat. #	Price
PID Lamp, Model 103 C	20676	\$390
PID Lamp, Model 108-10.0/10.6	20675	\$479
PID Lamp Cleaning Kit	20674	\$15

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**Please direct comments & suggestions on this publication to my attention:** Sherry Wenrick, Newsletter Editor, Ext. 2113, or e-mail to [sherw@restekcorp.com](mailto:sherw@restekcorp.com)

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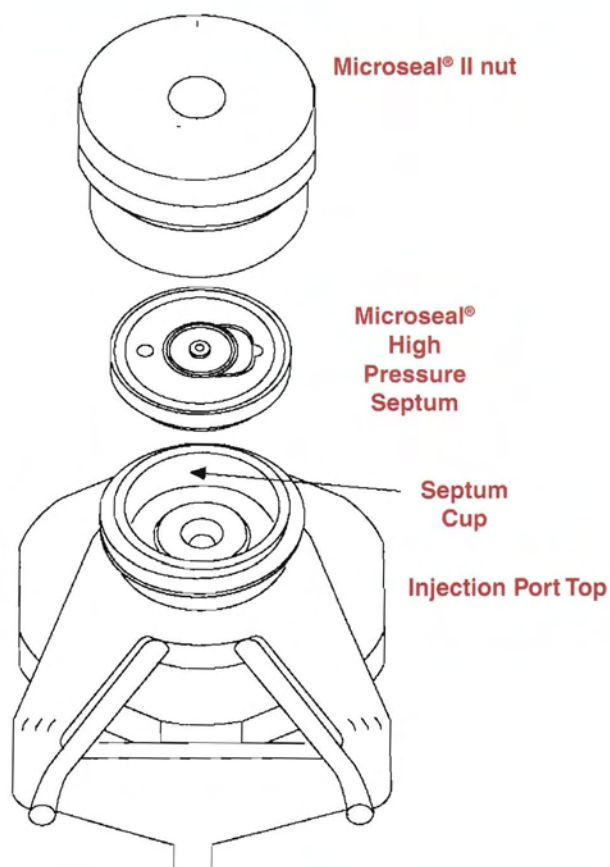
## Merlin Microseal® High Pressure Septum *New*

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### INSTALLATION



### Product Listing:

#### Merlin Microseal® High Pressure Septa

Description	Cat.#	Price	Merlin#	Similar to HP#
Nut Kit (1 Nut, fits 300 & 400 series septa):	22809	\$100	403	5182-3445
Standard Kit (Nut, 2 High Pressure Septa):	22810	\$340	404	Not offered
Starter Kit (Nut, 1 High Pressure Septum):	22811	\$240	405	5182-3442
High Pressure Replacement Septa (1-septum):	22812	\$140	410	5182-3444

#### Merlin Microseal® Septa

Description	Cat.#	Price	Merlin#	Similar to HP#
Standard Kit (Nut, 2 Septa):	22813	\$250	304	5181-8833
Starter Kit (Nut, 1 Septum):	22814	\$190	305	5181-8816
Microseal Replacement Septa (1-septum):	22815	\$90	310	5181-8815
Replacement PTFE Washers (2-pack):	22808	\$5	311	5181-0853

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# RESTEK

## Behind the Scenes

### Welcome Jingzhen Xu and Doug Elliott!

• **ASTM E-19 Meeting...** Oct. 12-17, San Diego, CA.

• **Northeast Regional Chromatography Discussion Group (NERCDG)...** Oct. 15, Rochester, NY.

• **Northeastern Association of Forensic Scientists...** Oct. 16-18, White Plains, NY.

• **FACSS '97...** Oct. 25-31, Providence, RI.

• **California Association of Toxicologists...** Nov. 3-7, Las Vegas, NV.

• **Eastern Analytical Symposium...** Nov. 16-21, Somerset, New Jersey.

• **Chiral GC Seminar...** Nov. 21, E. Brunswick, New Jersey.

Restek is pleased to have Jingzhen Xu join its dynamic research and development team. Jingzhen received his bachelor in chemical engineering from Tsinghua University, Master in chemistry from Beijing Polytechnic University and Ph.D. from Southern Illinois University at Carbondale.

Jingzhen has extensive experience in chromatography, especially multidimensional gas chromatography. His knowledge in hydrocarbon analysis and instrumentation will help him develop new products and applications. He is now working on PLOT columns. If you would like to discuss your PLOT column applications, don't hesitate to call Jingzhen at extension 2158.



During 10 years in analytical Services at Air Products, Doug was involved in

various disciplines including gas chromatography and instrumental microanalysis. He left to join the instrument and supplies side of analytical chemistry as Manager of Sales and Marketing at Control Equipment Corp. and then Accessories Marketing Manager at Supelco. Doug's experience has provided him with the skills to develop new products and the personal contacts in our industry to bring the best commercial products to you. Doug is here to support you with your questions to optimize the performance of your chromatography accessories as well as to develop products that better meet your needs. Call him at extension 2159.



### Restek On The Road

**Comprehensive Capillary Gas Chromatography Seminar...** Sept. 22-25, California & surrounding area.

**SOFT '97 Meeting...** Oct. 5-9, Snowbird, UT.

**Comprehensive Capillary Gas Chromatography Seminar...** Oct. 7-9, Houston, TX & surrounding area.

**ISA '97...** Oct. 7-9, Anaheim, CA.

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# RESTEK

## The Advantage

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### Fast GC Using Microbore Capillary Columns

by Kristi Sellers

Reducing  
instrument &  
operator time for gas  
chromatographic  
analyses has  
become an important  
consideration for  
many laboratories.

The use of microbore (0.10mm ID) columns can significantly reduce analysis time without sacrificing resolution. The extremely high efficiency of microbore columns (~7000 plates/meter) can provide resolution of complex mixtures while using shorter lengths. Shorter columns are less expensive and reduce analysis times, resulting in a cost savings for the lab.

Some instrument companies have been promoting the benefits of fast screening columns, but the sacrifices required aren't always evident from their literature. The reduction of analysis time at the expense of resolution, sample capacity, and ease of use is not always an acceptable alternative. This article will discuss and demonstrate the benefits and limitations of 0.10mm ID columns.

#### Speed and Resolution

**Table I** compares the characteristics of microbore columns to conventional columns. This data holds the key to whether microbore columns are right for your analysis. The most striking difference of microbore columns is their high efficiency (plates/meter) compared to other diameters. **Table I** indicates that a 0.10mm ID column is 160% more efficient than a 0.25mm ID column. This high effi-

ciency allows shorter columns to maintain excellent resolution and increase the speed of analysis. However, some of the other parameters in **Table I** illustrate limitations that may negate the usefulness of microbore columns in your laboratory. The effect of low flow rates, low sample capacity, and high operating pressures on your sample requirements will ultimately determine if microbore columns are an improvement for your laboratory.

#### Flow Rates

The low flow rates for microbore columns can be either an advantage or a limitation. Low flow rates are beneficial for GC/MS users because the flow rates are well within the pumping capacity of most systems. In addition, the microbore prevents "pumping out the column" or operation below atmospheric pressure. This provides more efficiency

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**FIA Fuel Testing with the  
Rtx®-5 Column**  
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**Peak Performers**  
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**Pro ezGC™ for Windows®**  
...pg. 15

**Restek Behind the  
Scenes**  
...pg. 16

Table I:

#### Column Characteristics

Column ID	0.10mm	0.18mm	0.25mm	0.32mm	0.53mm
Theoretical plates/m	8,600	5,300	3,300	2,700	1,600
Effective plates/m	6,700	3,900	2,500	2,100	1,200
He flow @ 20cm/sec	0.1cc/min.	0.3cc/min.	0.7cc/min.	1.0cc/min.	2.6cc/min.
H <sub>2</sub> flow @ 40cm/sec	0.2cc/min.	0.6cc/min.	1.4cc/min.	2.0cc/min.	5.2cc/min.
Sample Capacity	5-10ng	10-20ng	50-100ng	400-500ng	1000-2000ng
Operating Pressures	40.0psig	21.0psig	12.5psig	7.5psig	3.0psig

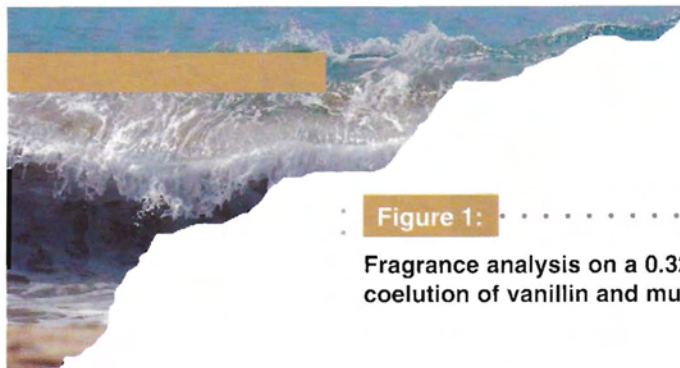
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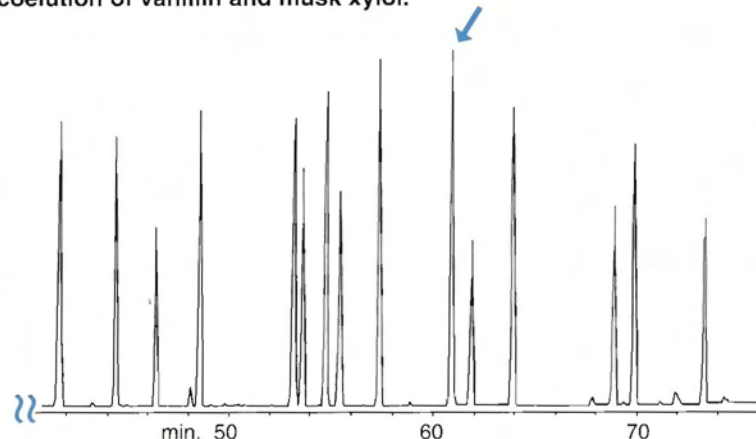
Summer  
97





**Figure 1:**

Fragrance analysis on a 0.32mm ID Rtx®-Wax column takes 75 minutes with complete coelution of vanillin and musk xylol.



30m, 0.32mm ID, 0.25µm Rtx-WAX (cat.# 12424). Splitless injection, 50mls/min. **Oven temp.:** 75°C to 225°C @ 70°C/min. (hold 15 min.). **Inj. & det. temp.:** 225°C. **Carrier gas:** helium, 30cm/sec. set @ 75°C.

for the end user. However, low flow rates also translate into more flow path problems for the chromatographer. Unswept dead volume has disastrous consequences on the column performance.

### Operating Pressures

**Table I** also shows that microbore columns require higher operating pressures which results in more ferrule leaks, septum leaks, and sample blow back through leaking syringe plungers. Connections need to be monitored for leaks more often. The pneumatic systems for older GCs are designed to operate at only 30psig and may need to be modified to handle higher pressures required for narrow bores. Operating microbore columns below optimum pressures will translate into poor resolution and poor performance.

### Sample Capacity

A limiting factor of a microbore column is the amount of sample that can be injected onto the column.

**Table I** indicates that the sample capacity of a microbore column is ten times less than a 0.25mm ID column. Therefore, the on-column injection should be at least ten times lower for a microbore column.

Sample cleanliness is another important factor to take into consideration when using microbore columns. Because the surface area of the 0.10mm ID columns is much lower than a conventional column,

contamination will occur more rapidly when dirty samples are injected. This means that 0.25 or 0.32 mm ID columns will be more rugged and require less maintenance for dirty samples than microbore columns. Whenever possible, samples containing non-volatile residue should be avoided. If dirty samples are a must, extensive column and injection port maintenance is required. Otherwise, loss of resolution,

ghost peaks, and a high background signal will result.

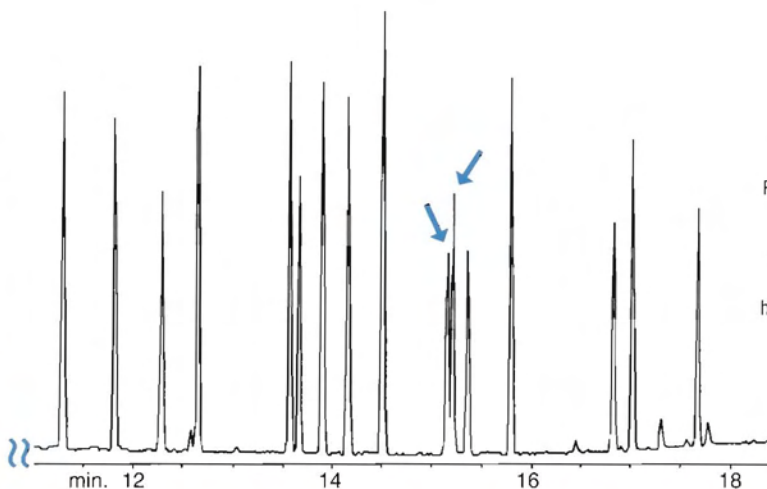
### Injector Considerations

Direct and on-column injection modes are not recommended due to the required low flow rates and small bore size of these columns. Therefore, trace analyses are difficult to perform with microbore columns. Split and splitless injections are the best alternatives. However, since

microbore columns require low flow rates, speed of sample transfer through the liner to the column is a concern. Due to the high dead volume, poor peak shape, and response, loss of resolution will occur when 2 or 4mm ID liners are used in conjunction with microbore columns. Thus, 1mm ID inlet liners are a must for sharp, well resolved, and recovered peaks. Not only is the inlet liner a consideration when

**Figure 2:**

Fragrance analysis on the Rtx®-Wax microbore column reduces run times by 75% with increased resolution between vanillin and musk xylol.



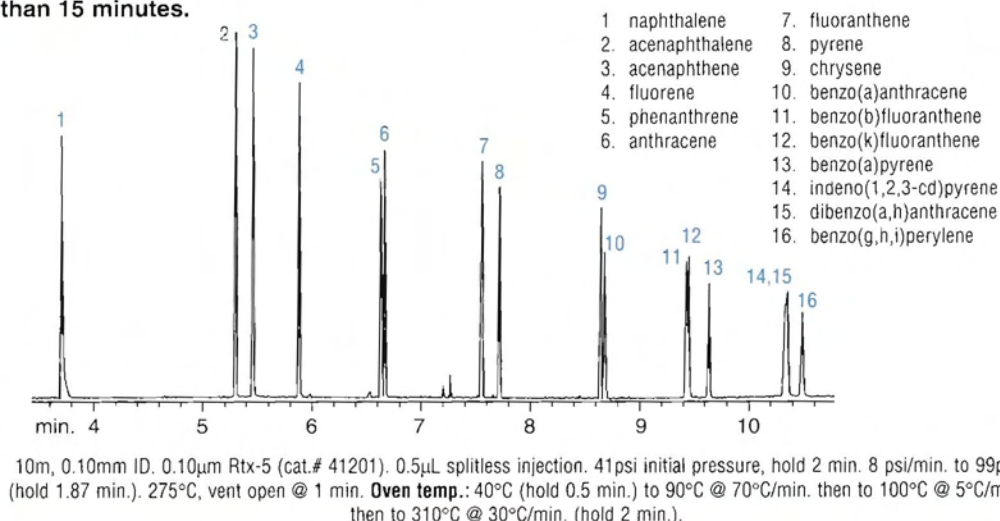
10m, 0.10mm ID, 0.20µm Rtx-WAX (cat.# 41603). Splitless injection, 250mls/min. **Oven temp.:** 55°C (hold 1 min.) to 225°C @ 10°C/min. **Inj. & det. temp.:** 225°C. **Carrier gas:** hydrogen, 70cm/sec. set @ 55°C.





**Figure 3:**

**Polynuclear aromatic hydrocarbons on an Rtx®-5 microbore column are analyzed in less than 15 minutes.**



using microbore columns for split or splitless injections, but other parameters specific to the type of injection method must also be optimized.

In a split injection, the choice of inlet liner and initial temperature will affect peak shape, response, and resolution the most. **Figure 1** shows part of a typical fragrance analysis on a conventional column (0.32mm ID). Under optimal conditions (4mm ID inlet liner and initial temperature of 75°C), the analysis time is more than 70 minutes and the separation of vanillin and musk xylol could not be achieved. By switching to a microbore column and optimizing run conditions (1mm ID inlet liner and initial temperature of 55°C), we were able to reduce the analysis time to 18 minutes and attain 80% resolution of the vanillin and musk xylol as shown in **Figure 2**. The 1mm ID inlet liner improved the recovery and peak shape of the early

eluting compounds.

**Figure 3** illustrates a splitless PAH analysis on a 10m, 0.10mm ID, 0.10µm Rtx®-5 using an optimized inlet liner and inlet pressure. When a 2mm ID inlet liner was used, high molecular weight discrimination occurred. By changing to a 1mm ID inlet liner, high molecular weight discrimination was eliminated. However, this change caused peak splitting of the early eluting compounds. The peak splitting was eliminated completely when pressure programming was applied in place of constant pressure.

#### Detector Considerations

Detector design and flows must be optimized when using microbore columns. Make up gas flows may need to be increased to minimize detector dead volume and compensate for the lower column flow rates. Since peak widths are approximately half compared to conventional columns (< 1

second), fast integrator and detector electrometers must be used. Integrator sampling rates must be increased over rates used for 0.25mm ID columns since the peaks are much narrower with microbores. If

the sampling rate is too slow, then poor integration and non-reproducible peak areas will result. Check with your instrument company and data system manufacturer to be sure your system is capable of handling microbore sampling rates.

Microbore columns can produce shorter analysis times, equivalent resolution, and provide cost savings. But remember, converting your conventional system to a microbore system isn't as easy as changing columns. Column capacity, sample purity, and injector and detector conditions must be considered and optimized for a successful analysis. Keep in mind that when switching from conventional capillaries to microbore columns, there may be the need to optimize inlet temperatures, liners, and GC run conditions.

#### Product Listing:

Microbore Capillary Columns					
0.10mm ID, 0.10µm					
Column	temp. limits	10-meter	20-meter	10-meter	20-meter
Rtx®-1	-60 to 330/350°C	41101 \$250	41102 \$415		
Rtx®-5	-60 to 330/350°C	41201 \$250	41202 \$415		
Rtx®-Wax	20 to 250°C	41601 \$250	41602 \$415		
0.10mm ID, 0.20µm					
Column	temp. limits	10-meter	20-meter	10-meter	20-meter
Rtx®-Wax	20 to 250°C	41603 \$250	41604 \$415		
0.10mm ID, 0.40µm					
Column	temp. limits	10-meter	20-meter	10-meter	20-meter
Rtx®-1	-60 to 320/340°C	41103 \$250	41104 \$415		
Rtx®-5	-60 to 320/340°C	41203 \$250	41204 \$415		

**Contact Restek's GC experts to discuss the suitability of Microbore or other GC columns for your specific application.**



# Rtx<sup>®</sup>-CLPesticides Column

## The Complete Solution for Chlorinated Pesticides Analysis

by Frank Dorman

- Baseline resolution of the 22 pesticides in EPA 8081.
- Analysis time under 25 minutes.
- Exceptional inertness for endrin, DDT, & methoxychlor.
- Temperature stability > 300°C.
- Exceeds performance criteria for EPA 8081, 608, and CLP.

For years, environmental laboratories have struggled with various chlorinated pesticide analytical methods. They must keep track of resolution requirements and breakdown performance criteria while analyzing extracts which usually contain high-boiling contaminants. Historically, many laboratories have used cyanopropyl capillary column stationary phases (DB-1701, Rtx-1701) which very often provide the best resolution between target

compounds, but have several limitations. The new Rtx<sup>®</sup>-CLPesticides column has been specially designed to overcome the resolution, breakdown, and temperature limitations of these other phases.

The Rtx<sup>®</sup>-CLPesticides column is capable of baseline resolution of the 22 common chlorinated pesticides (see **Figure 1**) listed in USEPA Methods 8081 and 608 and in the EPA's Contract Lab

Figure 1:

The Rtx<sup>®</sup>-CLPesticides column provides baseline resolution of 22 chlorinated pesticides in less than 25 minutes.

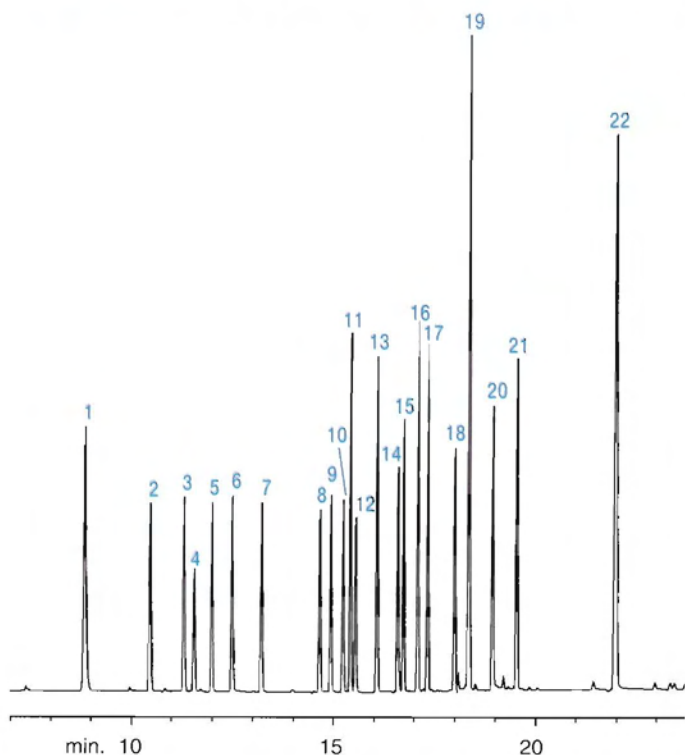
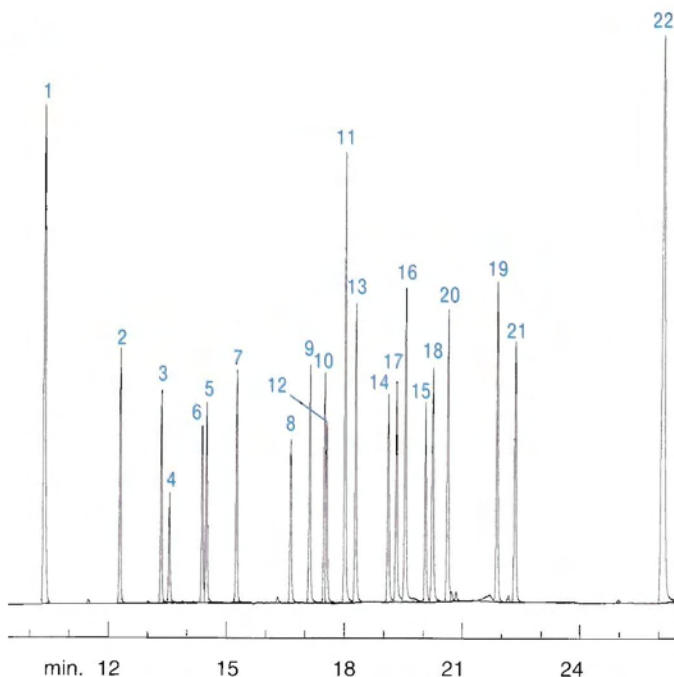


Figure 2:

The Rtx<sup>®</sup>-35 column provides elution order changes for 8 of the 20 chlorinated pesticides and is currently the best confirmational column for chlorinated pesticides.







Program (CLP) protocol. This column is available in 0.25, 0.32, and 0.53mm IDs, and has been optimized for ECD analysis resulting in very low bleed after conditioning. Analysis time has not been sacrificed in order to obtain baseline resolution. In less than 25 minutes, your analysis is complete with the Rtx®-CLPesticides column. This results in the laboratory's ability to make at least two injections per hour—an important factor for increasing sample throughput. In addition to its separating power, the Rtx®-CLPesticides column has a maximum temperature of 330°C, allowing you to bake out high molecular weight contaminants.

Although the Rtx®-CLPesticides column is clearly the column of choice for this analysis, most laboratories require a second column of different polarity for confirmation. To fulfill this requirement, Restek recommends using the Rtx®-35 column. It provides elution order changes

for several compounds, while exhibiting high thermal stability and inertness. The Rtx®-35 only has two compounds that elute closely: endosulfan I and  $\alpha$ -chlordane. Under the same temperature and flow conditions as the Rtx®-CLPesticides column, the Rtx®-35 column is an excellent choice for a confirmation column, see **Figure 2**.

The combination of the Rtx®-CLPesticides and Rtx®-35 columns provides unsurpassed performance for the analysis of chlorinated pesticides. They both exhibit the necessary resolution, low bleed, and thermal stability for this demanding analysis, as well as exceptional inertness for methoxychlor, endrin, and DDT. Both columns provide different elution orders for confirmation. If you are involved with the analysis of chlorinated pesticides and want to improve your resolution and increase your throughput, try the Rtx®-CLPesticides column.

### Peak List & Conditions for Figures 1 & 2

- |                                 |                        |
|---------------------------------|------------------------|
| 1. 2,4,5,6-tetrachloro-m-xylene | 12. endosulfan I       |
| 2. $\alpha$ -BHC                | 13. dieldrin           |
| 3. $\gamma$ -BHC (lindane)      | 14. endrin             |
| 4. $\beta$ -BHC                 | 15. 4,4'-DDT           |
| 5. $\delta$ -BHC                | 16. endosulfan II      |
| 6. heptachlor                   | 17. 4,4'-DDD           |
| 7. aldrin                       | 18. endrin aldehyde    |
| 8. heptachlor epoxide           | 19. methoxychlor       |
| 9. $\gamma$ -chlordane          | 20. endosulfan sulfate |
| 10. $\alpha$ -chlordane         | 21. endrin ketone      |
| 11. 4,4'-DDE                    | 22. decachlorobiphenyl |

30m, 0.32mm ID, 0.50 $\mu$ m Rtx®-CLPesticides (cat.# 11139)  
30m, 0.32mm ID, 0.50 $\mu$ m Rtx®-35 (cat.# 10439)

**Oven temp.:** 120°C (hold 1 min.) to 285°C @ 8.5°C/min. (hold 6 min.).

**Inj. port:** Direct injection using a Uniliner® (cat.# 20335) at 200°C.

**Detector:** ECD at 300°C with Anode Purge

**Flow rate:** helium @ 2.1ml/min. set @ 120°C

### Product Listing:

#### Rtx®-CLPesticides Columns

	Cat.#	Price
30m, 0.25mm ID, 0.25 $\mu$ m	11123	\$445
30m, 0.32mm ID, 0.50 $\mu$ m	11139	\$475
30m, 0.53mm ID, 0.50 $\mu$ m	11140	\$525

#### Rtx®-35 Columns

	Cat.#	Price
30m, 0.25mm ID, 0.25 $\mu$ m	10423	\$385
30m, 0.32mm ID, 0.50 $\mu$ m	10439	\$415
30m, 0.53mm ID, 0.50 $\mu$ m	10440	\$465

### New CLP Pesticide Standards

#### Organochlorine Pesticide Mix AB #1

aldrin	dieldrin
$\alpha$ -BHC	endosulfan I
$\beta$ -BHC	endosulfan II
$\delta$ -BHC	endosulfan sulfate
$\gamma$ -BHC (lindane)	endrin
$\alpha$ -chlordane	endrin aldehyde
$\gamma$ -chlordane	endrin ketone
4,4'-DDD	heptachlor
4,4'-DDE	heptachlor epoxide (B)
4,4'-DDT	methoxychlor

200 $\mu$ g/ml ea. in hexane/toluene (1:1), 1ml/ampul

	Each	5-pk.	10-pk.
	32291 \$35	32291-510 \$157.50	—
w/data pack	32291-500 \$45	32291-520 \$175	32391 \$315

#### Organochlorine Pesticide Mix AB #2

aldrin	8 $\mu$ g/ml	dieldrin	16 $\mu$ g/ml
$\alpha$ -BHC	8	endosulfan I	8
$\beta$ -BHC	8	endosulfan II	16
$\delta$ -BHC	8	endosulfan sulfate	16
$\gamma$ -BHC (lindane)	8	endrin	16
$\alpha$ -chlordane	8	endrin aldehyde	16
$\gamma$ -chlordane	8	endrin ketone	16
4,4'-DDD	16	heptachlor	8
4,4'-DDE	16	heptachlor epoxide (B)	8
4,4'-DDT	16	methoxychlor	80

At concentration listed in hexane/toluene (1:1), 1ml/ampul

	Each	5-pk.	10-pk.
	32292 \$25	32292-510 \$112.50	—
w/data pack	32292-500 \$35	32292-520 \$125	32392 \$225



# Optimizing Linear Velocity and Temperature Program for Capillary Chiral Analyses

by Sherry Sponsler

Chiral chromatography can be a useful tool for determining purity and authenticity in flavor, fragrance, and drug applications. As with all GC analyses, certain parameters should be considered to attain maximum enantiomeric separation and chiral capillary column performance. GC conditions such as linear velocity and temperature program must be optimized to ensure maximum chiral resolution.

## Linear Velocity "Faster is Better"

In many instances, the resolution between enantiomers can be improved with linear velocities that are faster than those considered optimum by the Van Deemter curve. This has been demonstrated for five out of six racemic compounds that were evaluated on a 30-meter, 0.32mm ID, 0.25 $\mu$ m Rt- $\beta$ DEXsa column using different linear velocity conditions. Although optimum linear velocity can be different for each chiral compound and column, the typical optimum linear velocity for maximum enantiomeric separation is ~80 cm/sec. with hydrogen carrier gas (Figure 1). This is twice the expected linear velocity required to achieve maximum column efficiency, as indicated by the Trennzahl values in Figure 2. Therefore, conditions providing maximum chromatographic peak efficiency do not always result in optimum chiral resolution.

Not all chiral components achieve maximum enantiomeric resolution at the same linear velocity, so this must be adjusted for specific target analytes. For instance, Grob<sup>1</sup> demonstrated optimum chiral resolution of  $\gamma$ -lactones at 50 cm/sec.<sup>1</sup> However, 1-octen-3-ol was an exception since Figure 1 shows no increase in chiral resolution from 40 cm/sec. to 80 cm/sec.

## Temperature Program "Slow Temperature Ramp Rates Are Better"

Several different temperature ramp rates were evaluated to determine the optimum linear velocity for enantiomeric resolution of 6 chiral compounds. The best chiral separations were achieved with temperature program rates between 1 and 2°C/min. (Figure 3). Unlike linear velocity, Trennzahl values increase along with enantiomeric resolution at these temperature program rates (Figure 4). Lower elution temperatures can provide increased enantiomeric resolution for chiral compounds. This suggests that selectivity of cyclodextrin columns improves with decreased temperature during the separation process.<sup>1</sup>

Flow rates that provide maximum separation efficiency do not always result in optimum enantiomeric selectivity. Higher linear velocities and slow temperature ramp rates promote lower elution temperatures, which

can provide better chiral separation. Optimizing these conditions can enhance

cyclodextrin column performance and the overall quality of chiral chromatography.

Figure 1:

Higher linear velocities provide maximum resolution of chiral pairs.

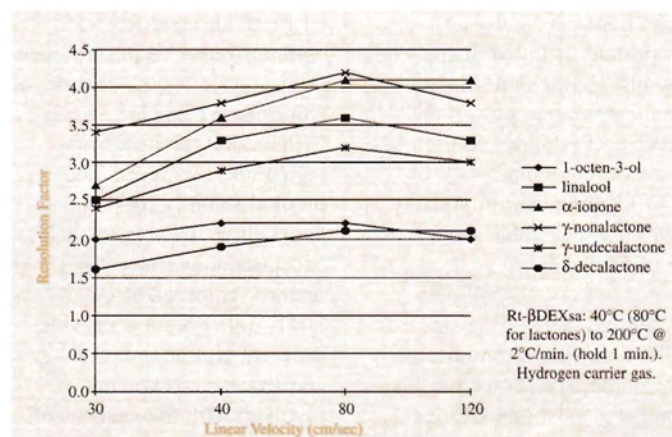
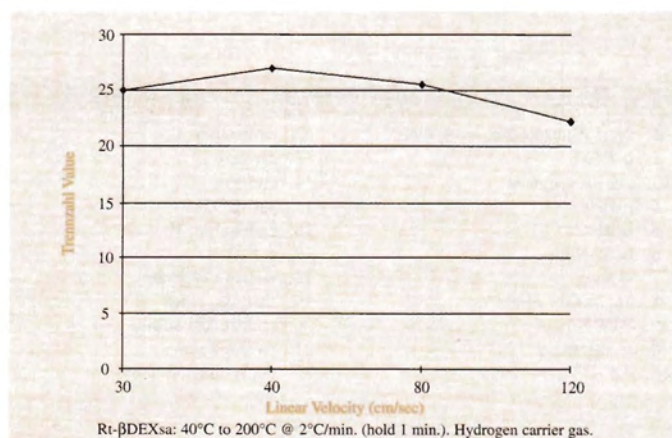


Figure 2:

Higher trennzahl values do not correspond with optimum enantiomeric separation.



## What about helium?

The observed optimum linear velocity for helium carrier gas is about 60 cm/sec for many of these chiral separations.



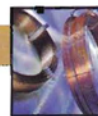


Figure 3:

Temperature program rates between 1 and 2°C/min. provide optimum enantiomeric separation.

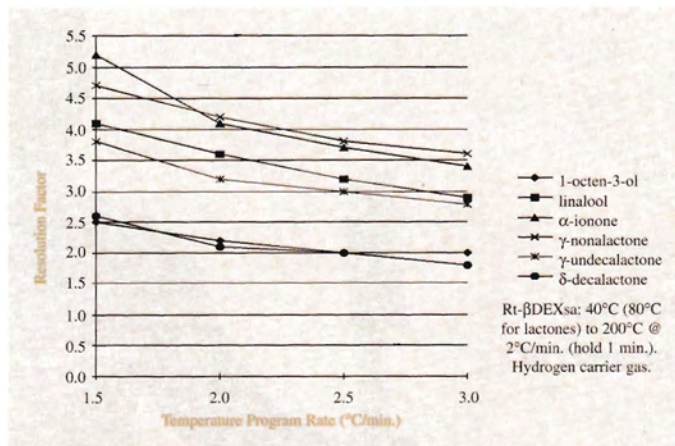
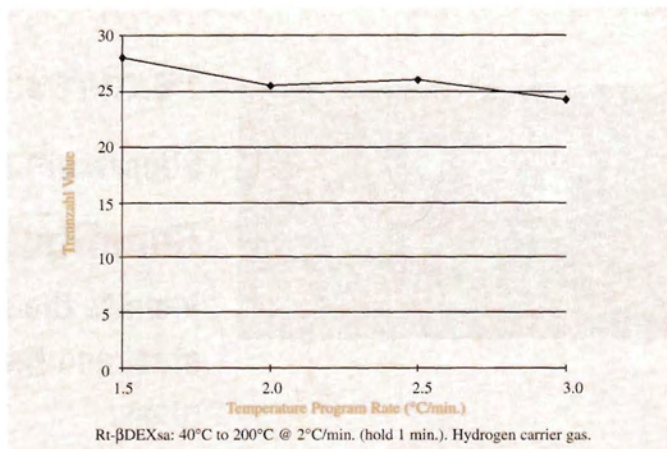


Figure 4:

Trennzahl values increase along with enantiomeric resolution at these slow temperature ramp rates.



#### References

<sup>1</sup> K. Grob, H. Neukom, H. Schmarr and Armin Mosandl, *Journal of High Resolution Chromatography* **13**, 433-436 (1990).

## RESTEK IS THE LONG-LIFE LEADER. . .

### Just Ask Chris Nelson and Richard Johnson at PPD Pharmaco!

Chris Nelson and Richard Johnson, chemists at PPD Pharmaco in Madison, Wisconsin, truly believe that Restek is the **Long Life Leader**! Chris was using a Gamma-cyclodextrin trifluoro-acetyl column for the analysis of dexfenfluramine and fenfluramine. Unfortunately, the columns he had been using for the analysis had so many problems, they really made his job difficult. They gave inconsistent performance, had poor resolution, were extremely expensive, were difficult to obtain because of very long delivery times, and once the seal was broken they could not be returned. But most importantly, the columns did not last very long. Chris only averaged about 150 injections before the column failed!

When he switched to Restek's Rt-βDEXcst columns, Chris was amazed. How could a column that costs less last ten times longer? On top of being less expensive and lasting longer, the columns were delivered quickly and showed more column-to-column consistency. According to Chris, "Every column we pull out of the box works".

With Restek's Rt-βDEXcst columns, Chris averages 1,000 to 1,100 injections before the column's performance degrades, unlike the 150 injections from the other columns he was previously

using. One Restek column actually performed for up to 2,000 injections!

Thanks Chris for sharing your information with everyone and showing that once again Restek is the **Long-Life Leader**!



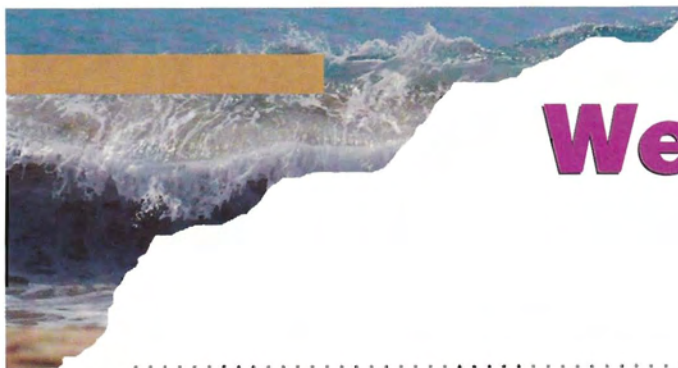
Richard Johnson and Chris Nelson have made 2,000 injections on Restek's Rt-βDEXcst column before replacement was necessary.

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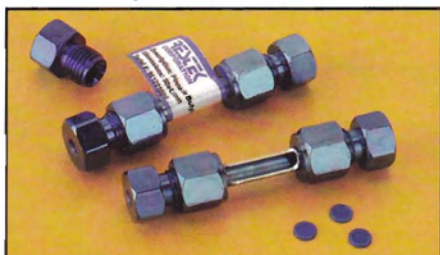
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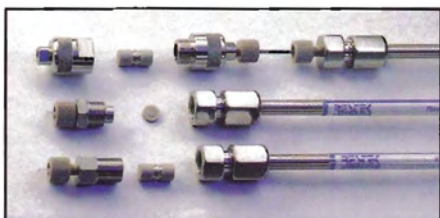


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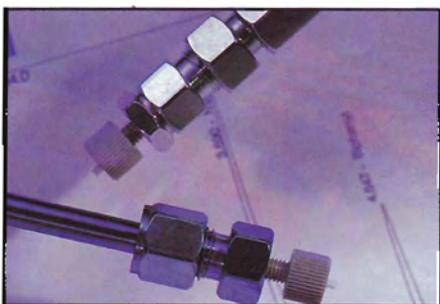


## Features:

**Trident™ Integral Guard System**

## Benefits:

Reliable column protection without the hassle of connecting tubing.

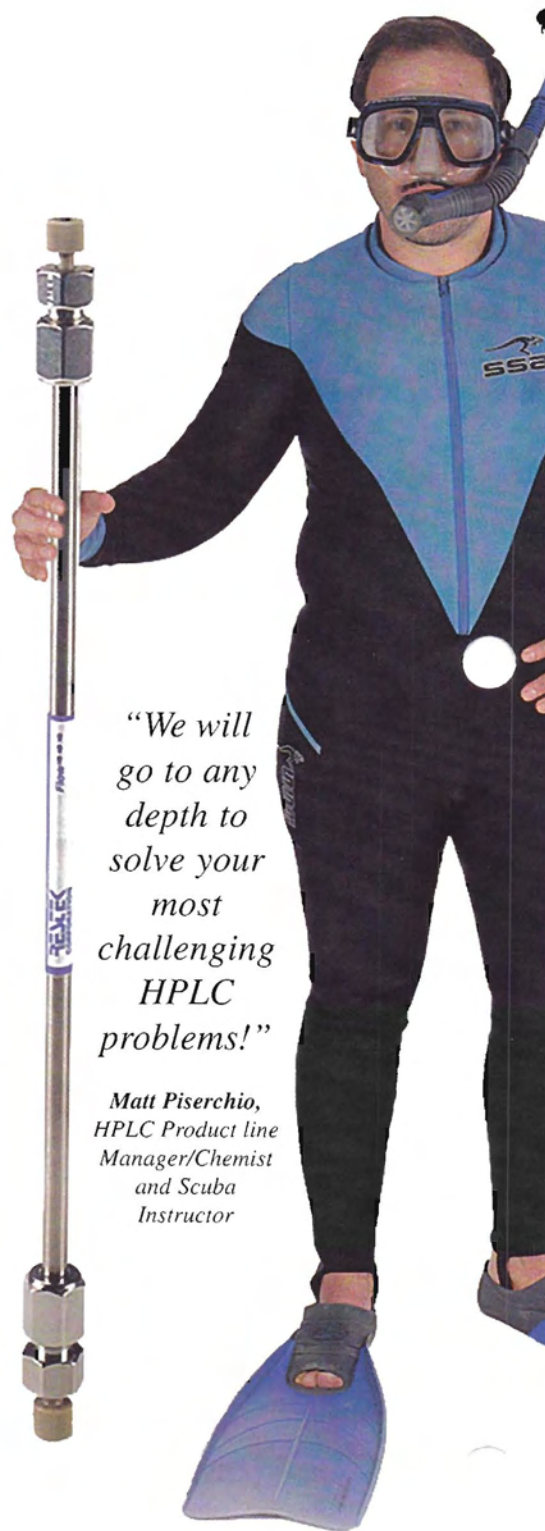


## Features:

**Stringent QA Specifications**

## Benefits:

Guarantee of High Performance



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HPLC Product line  
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# HPLC Columns...



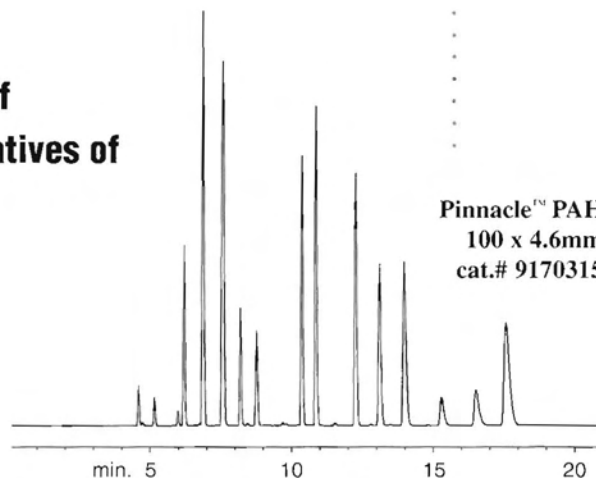
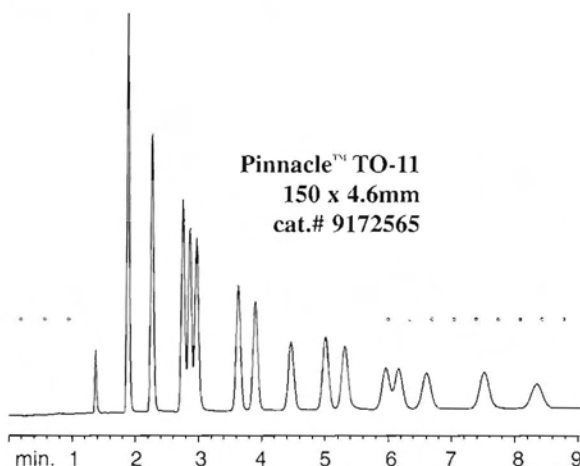
## Features:

Application Specific Columns:

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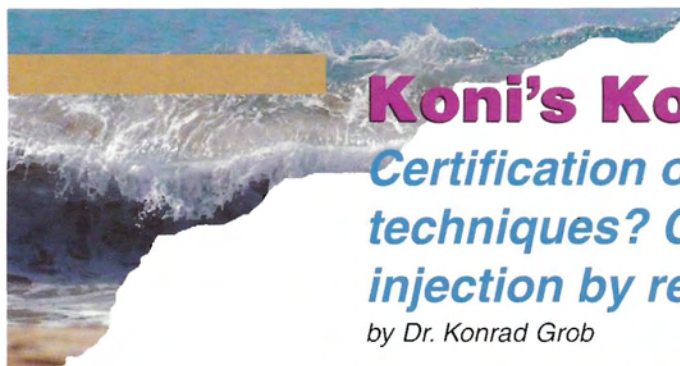
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## Koni's Korner

### Certification of injectors and injection techniques? Comments on splitless injection by readers.

by Dr. Konrad Grob

In the spring and summer 1996 issues of *The Restek Advantage*, I posed the question of whether the most frequently used injection technique in capillary GC, splitless injection, is as mature as one tends to think. I traced its history to show that there has never been the systematic optimization and testing many think should have happened. Nobody felt responsible: Users assumed that instrument manufacturers provide exhaustively tested injectors and working instructions, whereas instrument companies just produce what they think "science" wants. But who is "science"?

Optimization of a technique as complex as splitless injection is work of such a volume that it cannot be accomplished on a single Friday afternoon, when the work of the week is completed. One of the open questions concerned sample evaporation in splitless injection. Should the liner be empty or packed? Should it have a constriction at the bottom? I was hoping for contributions by those routine users who must have found an answer in one way or another, but only received more general comments, three of which I want to bring up here.

#### Who introduced splitless injection?

Leslie Ettre was upset by my saying that my father introduced/invented splitless injection. Indeed, non-splitting injection was used from the very

beginning of capillary GC, in particular before splitting was invented. I want to apologize for not having mentioned this. In response to him, my definition of "splitless" injection is not any non-splitting injection technique, but that of using an injector with a split outlet which is closed during the splitless period. At least in Europe, "direct" injection has always been distinguished from "splitless" injection.

#### Accelerated transfer through increased flow

E.H. Foerster, from Southwestern Institute of Forensic Science in Dallas, Texas, found that the analysis of low concentrations of certain active drugs (he named alprazolam, trazodone, and quinidine) was possible by split, but not by splitless injection (4 mm i.d. liner with glass wool). He could improve the results from splitless injection approximately four fold by increasing the carrier gas inlet pressure (gas flow rate) during a 1 min. transfer period after injection. He explained this by the reduced residence time in the injector during split or accelerated splitless injection. The same argument was used by Hewlett-Packard in favor of what they termed "pressure pulse".

An increase of the flow rate by a factor of four is possible only if initial inlet pressures are modest and, nevertheless, does not seem overwhelming: it reduces reaction time by a factor of four. However, the effect

could be more than proportional, since the sample liquid deposited onto the packing initially forms an island cooled to the solvent boiling point. A high flow rate might remove the solute material from these surfaces before they have reached the injector temperature again. If evaporation occurred in the gas phase, the fog of the non-evaporated solute material could have been transferred into the column before it settled onto the packing material. Unfortunately, Mr. Foerster did not compare the performance of the packed liner with that of the empty liner, because gas phase evaporation is usually still most gentle (but not always complete).

#### Injector overloading

Gary Kellog, from the Springfield, Missouri Public Health Department, drastically illustrated the effect of overloading too small vaporizer chambers. "Last February we received a new GC/MS system, including a Varian 1078 temperature programmable split/splitless injector. At about the same time, I received my first copy of *The Restek Advantage* including your article on injector design and sample introduction. I had never used a split/

splitless injector before. The old instrument was set up with a flash vaporization injector with a 0.53 mm ID column, and it didn't take long to realize that the old operating parameters would not work on the new system. When I began to calculate the vapor volumes and the liner volumes (54 mm x 0.8 mm ID with 9 mm column installed height, methanol as solvent), it was obvious that a lot of my sample was going into places other than the column. Due to the limited size of the 1078's liners (54 mm long), I chose the largest ID liner offered (3.4 mm), added a 1 cm plug of deactivated fused silica wool placed above the installed column height, and began to experiment with the temperature programming on the injector. I also switched to a lower vapor volume solvent, with a higher boiling point to take advantage of solvent effects (toluene)."

Gary Kellog used a mixture of pesticides to compare the peak areas obtained by the old conditions (0.8 mm i.d. liner, 250°C) with those he introduced recently (3.4 mm i.d. liner, injector programmed from 200 to 300°C). The detector, column, injection volume, and other conditions

Peak area x 10 <sup>6</sup>			
Compound	0.8 mm i.d.	3.4 mm i.d.	Difference
alpha HCH	0.63	4.16	7.85
diazinon	0.69	6.34	9.19
heptachlor	0.49	3.98	8.12
endrin	0.41	2.32	5.66
p,p'-DDT	0.72	5.00	6.94
coumaphos	0.69	2.08	3.01





were identical. Results were obtained with toluene as the solvent, which must have improved them substantially. From a long list of results, I just want to cite a few.

The results show drastic (66-89 %) losses of solute material with the small vaporizer chamber, but also that losses are different for each component. This was no surprise. The usable volume in this vaporizing chamber was 23 $\mu$ l. 1 $\mu$ l of methanol (which he usually used) must have produced 600-700 $\mu$ l of vapor (@ 250°C injector temperature & intermediate inlet pressure). Even if the needle was only partially emptied, the injector was overloaded more than 40 times. 1 $\mu$ l of the toluene actually used produces only about 200 $\mu$ l of vapor. Losses of solute material are usually smaller than those of the solvent, because solutes may be deposited onto surfaces cooled by the evaporating solvent—but the process is poorly controlled. It is as if an analyst would spill more than 90 % of the solution during titration and then be surprised that results are poorly reproducible. There is no pool of liquid running out of the GC instrument, which in turn explains why so many people “spill” in the GC inlet without noticing it.

Gary Kellogg's new injection technique might perform correctly, although it involves unusual conditions. He introduced his solution in toluene (b.p. 110°C) into the PTV at 200°C. Standard working rules would require an injector temperature at, or below, the pressure-corrected solvent boiling point, in order to prevent rapid expansion of the vapors. He calculated that the chamber has a usable internal volume of

354 $\mu$ l, which should be sufficient to store the vapors even when considering that they will mix with the carrier gas present in the injector. A 2 $\mu$ l volume (or 1 $\mu$ l of a solvent producing more vapor), however, would again be too much. Further, he applied some glass wool, which might retain the solutes when solvent vapors expand out of the injector chamber.

### Confusing injection conditions

Gary Kellogg plans to carefully test his injection conditions, maybe by comparison with on-column injection. However, does it really make sense that every gas chromatographer develop his own conditions to get his sample into the column?

The comment by Gary Kellogg demonstrates how chaotic injection in capillary GC still is. In HPLC, injection just requires filling of a sample loop without air bubbles and that the sample solvent is not too strong an eluent. It is standardized and essentially the same for all instruments. It is totally different in GC. Every instrument manufacturer seems to be proud of producing something different than the others and giving their injector another name. Did you ever count the names given to temperature-programmable injectors? Manuals do not provide sufficiently clear and safe rules on how to operate the device and warnings on where the limitations are. Confusion among the non-specialists is inevitable.

Why didn't anybody tell Gary Kellogg that his old injector cannot be used in the way he used it—and how many others continue to do the same? Why didn't he know that with his

new injector he can inject up to about 50 $\mu$ l (quite regardless of the vapor volume formed), provided he keeps the chamber below the solvent boiling point for the time of solvent evaporation?

### Why are injectors and injection techniques not validated?

Today, splitless injection is frequently performed with too small vaporizing chambers, too short syringe needles, poorly suited carrier gas supply systems, excessively large samples, by the cool instead of the hot needle technique (or vice versa), by slow instead of rapid injection, with too low carrier gas flow rates, wrong column temperature during the sample transfer, too short splitless periods, packings in the liner at the wrong site, and without information on what all the critical parameters are. Properly written methods should specify all these conditions in at least as much detail as they specify sample preparation by saying that the flask must be rinsed twice and the solvent combined.

Analytical methods are validated in order to demonstrate the reliability of results. Chemicals, balances and pipettes are usually of certified quality and performance. Users check them ever so often. GCs are also checked. Oven temperatures are measured—as if this would be a critical parameter. Methods describe all steps of sample preparation in great detail, but when they reach the injection of the sample into GC, they become extremely short. Their authors would say that they cannot write as many versions as there are instrument manufacturers. True. But many users would badly need instructions, especially if their

instruments work properly at best under special conditions.

The quality management people might not have realized the potential of the errors occurring during injection, as shown by the above example, it is many times larger than that of a balance. How can they validate methods if one of the principal sources of error remains out of control? Maybe they did realize the problem, but felt unable to make valid suggestions. Methods cannot be validated for all the different injectors on the market, nor can they require the use of an injector from a particular manufacturer. They must assume a properly working injection system and the application of validated working rules for that system. These rules do not exist. At least for the time being, the concept of validation reaches its limit at this point. It underlines that capillary GC is not a simple technique and it relies a great deal on the expertise of the operator.

### 3 Final Points

- 1) Does it really make sense that every gas chromatographer finds his own way to get his sample into the column?
- 2) How can methods be validated if one of the principal sources of error, injection, remains out of control?
- 3) Methods cannot be written in as many versions as there are instrument manufacturers.

**I welcome your feedback.**  
**Reach me by e-mail at**  
**Konrad.grob@attach.ch.**



# Attention Heated Purifier Users!

## Restek Improves a Laboratory Classic

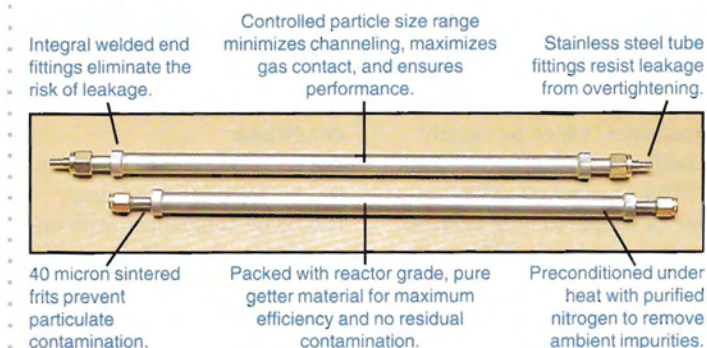
by Doug Elliott and Brad Rightnour

For decades, heated purifiers have provided unequalled service for contamination capacity, efficiency, and versatility. That is because nothing has been found to be more effective overall than heated non-evaporable getter materials for removing critical reactive contaminants from gas streams... *until now.*

Restek has taken current technology a step farther. Besides removing the same quantity of oxygen and water (14L and 35L) and purifying the same number of gas tanks at the same high 1100 ml/min. maximum flow rate as the original Supelco heated purifier tube, our attention to design detail eliminates leak sources, our control of material specifications optimizes performance, and our production processes remove potential contamination; essentially preconditioning the tube. This provides the analyst with hassle-free installation.

*The next time you need a replacement tube, choose Restek's Straight Replacement Purifier Tube and appreciate the differences. You'll be glad you did! As a special offer, SAVE 10% off the regular catalog price now through August 1997!*

**Restek's replacement Purifier Tube has improved fittings, getter material, and manufacturing processes for optimum carrier gas purification.**



### Replacement Straight Purifier Tube

1/8-inch	cat.# 21661, <del>\$90</del> <b>\$81</b>	Similar to Supelco #2-2396
1/4-inch	cat.# 21660, <del>\$90</del> <b>\$81</b>	Similar to Supelco #2-2398

## Restek Designs a Thermal Gas Purifier Furnace and U-Shaped Getter Tube

If you are not one of the analysts currently using heated getter technology to purify your carrier gas, try the Restek Thermal Gas Purifier. *It's everything you'd expect in a heated purifier plus more!* The unit:

- Completely removes up to 14 L of O<sub>2</sub> and 35 L of H<sub>2</sub>O vapor.
- Purifies up to 50 cylinders at 50 ppm impurity at flow rates up to 1100 ml/min.
- Removes O<sub>2</sub>, H<sub>2</sub>O, CO, CO<sub>2</sub>, sulfur compounds, and hydrocarbons.
- Saves you money by using less expensive grades of carrier gas.

### Restek Thermal Gas Purifier

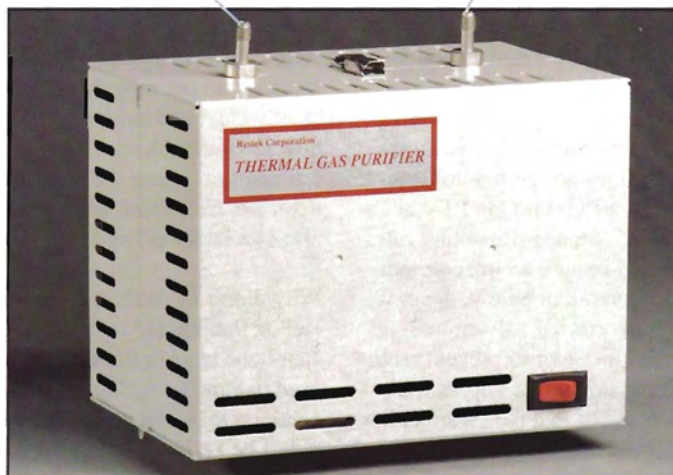
Fittings	110v	220v
1/8-inch	cat.# 21664, \$400	cat.# 21665, \$420
1/4-inch	cat.# 21662, \$400	cat.# 21663, \$420

### Restek Purifier Replacement U-Tube

1/8-inch	1/4-inch
cat.# 21659, \$90	cat.# 21658, \$90

**Restek's Thermal Gas Purifier combines the benefits of U-tube getter configuration with improved material handling to provide optimum carrier gas purification performance.**

- Top mounted fittings for easy installation and enhanced space utilization.
- U-tube configuration: vertical beds eliminate channeling.



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# Rtx<sup>®</sup>-5

## the Column of Choice for FIA Fuel Testing

by Lisa Serfass

Dr. Peter Tibbetts of M-Scan Ltd (Ascot, England) attends every Formula 1 Grand Prix Race. Although an avid racing fan, he doesn't actually get much time to enjoy the race. Dr. Tibbetts is the Director of Environmental Services at M-Scan Ltd and also serves as a Fuels Consultant for the FIA (Federation Internationale De l'Automobile). It is his responsibility to test fuel samples from the Formula 1 race cars in the FIA's Mobile Fuel Testing Lab.

The FIA is the governing body that sets the regulations that all Formula 1 competitors must comply with. It is the FIA's responsibility to ensure that each race car meets all technical regulations for safety as well as performance. Fuel testing is only one part of the process. The cars undergo intense scrutiny to ensure they meet all criteria set by the FIA. In addition to being measured and weighed, each car is checked for the proper engine capacity, aerodynamics, control systems, tires, and refueling systems.

Capillary gas chromatography is used to analyze the fuel and determine the exact pattern of components contained within the sample. Since race car fuel is composed of a complex mixture of hydrocarbons and other volatile organic compounds, each sample exhibits different chromatographic patterns. These patterns are known as fingerprints, since no two fuels are exactly alike.

The fuel specifications process begins with each racing team submitting a sample of the race fuel they wish to use for approval. If the sample is approved, its fingerprint is placed into a databank. The information is held for comparison with fuel samples at race time. Three replicate 250ml fuel samples are taken at the race. They can be taken at any time, usually during qualifying or just before or after the race. The containers are then sealed and witnessed by the team concerned to ensure there is no tampering with the samples. One of the samples is tested and compared with the fingerprint in the databank. The sample must match the fingerprint. If it does not, the race stewards are informed of the discrepancy and the sample is then sent on to the UK laboratory for a full mass spectral analysis. The second sample is sent to an independent laboratory for testing and the third sample is returned to the racing team so they can have their own analysis performed.

The fuel samples are analyzed using a 30 meter, 0.32 mm ID, 3.0µm Rtx<sup>®</sup>-5 capillary column. The Rtx<sup>®</sup>-5 column was chosen for this analysis because race car fuel is a highly complex mixture of very volatile compounds. The high separation efficiency of this column combined with the thick coating of stationary phase provides the necessary resolution needed to produce the unique fingerprints. The Rtx<sup>®</sup>-5 polymer was also chosen for its stability which results in extended column

lifetime and very low background bleed level.

In addition to the GC analysis, additional testing is done to measure the density of the sample. The sample is injected into a PAAR DMA48 Density Meter and held at a constant temperature (15°C) in a glass U-tube. This is vibrated by a piezo-actuator and the natural frequency is measured. The natural frequency is directly proportional to the mass of the U-tube containing the fuel sample. Since the volume is

known, the density can be calculated. Calibration is checked by injecting dodecane as a reference standard.

Usually, a minimum of three cars are randomly chosen from each race for testing. Each fuel analysis takes approximately 60 minutes and all testing must be completed before the race results can be confirmed. With all that is required for fuel testing, the drivers and cars aren't the only ones in a race. Dr. Tibbetts is in his own race—a race of time.

Figure 1:

Restek's high performance Rtx<sup>®</sup>-5 column is used to characterize fuel samples from Formula 1 racecars in a mobile fuel testing lab.



Rtx<sup>®</sup>-5

30-meter, 0.32mm ID, 3.0µm: cat.# 10284 \$415

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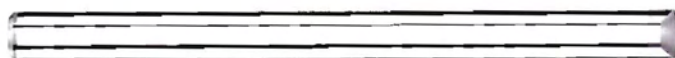


# Peak Performers

**New**

## Inlet Liners for Fisons 8000 & Shimadzu 17A Injectors

- Deactivated & tested for guaranteed inertness.
- Low internal volume makes them ideal for purge & trap or Fast GC analysis.
- Guaranteed to meet original equipment specifications.



**Fisons 8000 1mm Split Sleeve**

8.0mm OD x 1.0mm ID x 105mm

Each	5-pack	25-pack
20916 \$42	20917 \$168	—



**Shimadzu 17A 1mm Split Sleeve**

5.0mm OD x 1.0mm ID x 94mm

Each	5-pack	25-pack
20976 \$32	20977 \$128	20978 \$512

## Stainless Steel Capillary Nuts for HP 5890 GCs



**Stainless Steel Capillary Nut (inlet)**

For use with Standard Ferrules	For use with Compact "HP-type" Ferrules
20883 \$25/2-pack	21884 \$25/2-pack

## **New** Compact Vespe<sup>®</sup>/Graphite Ferrules for HP GCs

Restek now offers the compact Vespe<sup>®</sup>/graphite (60%/40%) ferrules for HP GC capillary injection ports. The Vespe<sup>®</sup>/graphite ferrules are designed to seal with minimal torque, are reusable, and are stable to 400°C. They are available in economical 10 & 50 packs and meet original equipment specifications.

**Compact Vespe<sup>®</sup>/Graphite Ferrules for HP GCs**

Ferrule ID	Fits Column ID	V/G 10-pack	V/G 50-pack	Similar to HP part #
0.4mm	0.25mm	20238 \$45	20239 \$180	5062-3516
0.5mm	0.32mm	20248 \$45	20249 \$180	5062-3514
0.8mm	0.53mm	20263 \$45	20264 \$180	5062-3512

**Compact Graphite Ferrules for HP GCs**

Ferrule ID	Fits Column ID	Graphite 10-pack	Graphite 50-pack	Similar to HP part #
0.4/0.5mm	0.25/0.32mm	20250 \$25	20251 \$100	HP0100-8853
0.8mm	0.53mm	20252 \$25	20253 \$100	HP0100-1042

**Graphite Capillary Ferrules**

Ferrule ID	Fits Column ID	Graphite 10-pack	Graphite 50-pack
0.4mm	0.25mm	20200 \$25	20227 \$100
0.5mm	0.32mm	20201 \$25	20228 \$100
0.8mm	0.53mm	20202 \$25	20224 \$100

**Vespe<sup>®</sup>/Graphite Capillary Ferrules**

Ferrule ID	Fits Column ID	V/G 10-pack	V/G 50-pack
0.4mm	0.25mm	20211 \$30	20229 \$120
0.5mm	0.32mm	20212 \$30	20231 \$120
0.8mm	0.53mm	20213 \$30	20230 \$120

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Please direct comments & suggestions on this publication to my attention: Sherry Wenrick, Newsletter Editor, Ext. 2113, or e-mail to [sherry@restekcorp.com](mailto:sherry@restekcorp.com)

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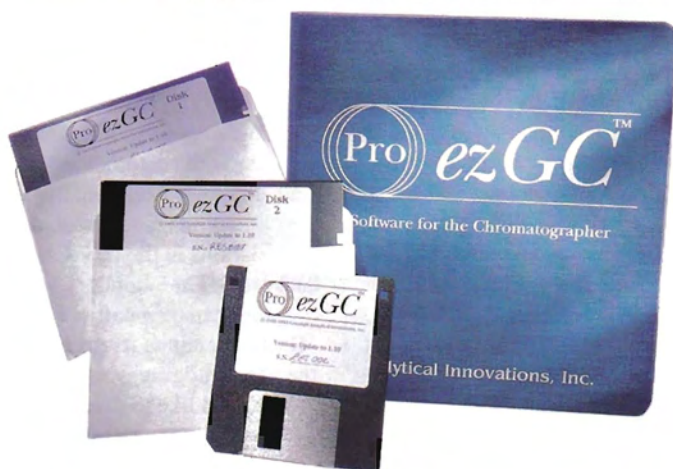
# Pro ezGC™ Software (Ver. 2.0) for Windows®.



*The most powerful GC optimization software has new features!*

by Mike Feeney

- Optimize temperature and flow programs from a single analysis.
- Model retention gap and guard column applications including Restek's Integra-Guard™ columns.
- Model columns in series for process GC and multi-dimensional GC analyses.
- Software routines expanded for Fast GC and Windows® 95.



Pro ezGC™ software is a powerful optimization software and retention index database for gas chromatography. This program accurately predicts GC separations on any capillary column and is useful for selecting the best column and conditions for your GC analysis. Pro ezGC™ software utilizes either your chromatographic retention data or you can use the extensive library containing thousands of commonly analyzed compounds. From this data, it is possible to automatically evaluate thousands of combinations of column dimensions, oven temperature program, and carrier gas pressure program to determine

the best separation with the fastest analysis time. This saves days of method development time for new analyses and can decrease analyses times of existing analyses by more than 50%. Several new features incorporated into version 2.0 make Pro ezGC™ software for Windows® even more powerful and easy to use.

## Optimize separation and analysis time from a single GC run

In order to accurately simulate separations and analysis time, the software requires either selecting a compound from an existing library or entering retention times from a GC analysis. This new version of

Pro ezGC™ software for Windows® can calculate the thermodynamic retention indices from a single chromatographic run. This analysis can be your current method, retention data from a scientific journal, or retention times from publications such as Restek's chromatography products catalog. This makes it possible to select a GC column for your analysis and estimate the separation quickly and easily.

## Model GC separation including guard columns and capillary restrictors

The analysis of contaminated samples often requires the use of a guard column to minimize contamination of the analytical column. Cool on-column injection often utilizes retention gaps to improve peak widths and to facilitate injection using standard gauge syringes. These "precolumns" can either be connected using a low-volume union or built-in as with Restek's Integra-Guard™ columns. In GC/MS applications, a length of deactivated tubing is sometimes added to the end of the analytical column to either increase the GC inlet pressure or to decrease background bleed from the transfer line tube. Pro ezGC™ software for Windows® now permits accurate modeling when these types of columns are used, even when the diameters are not identical to the analytical column.

## Predict the optimum column for dual columns connected in series or parallel

Many times, resolution of all components in a sample is not possible using a single column. This is where the power of Pro ezGC™ software becomes especially obvious because the program can either model two columns connected in parallel to two separate detectors or two columns connected in series. With series connections, the lengths of the two columns are often cut to give the best combination. This approach would be costly and time consuming without Pro ezGC™ software. Now, any laboratory can quickly evaluate whether serially coupled columns will give the separation without cutting up valuable capillary columns.

Take the guesswork out of selecting the best column and conditions for your GC analysis with Pro ezGC™ software for Windows®. Increase the productivity of your existing gas chromatographs by improving separations and shortening analysis time with optimized temperature and carrier gas programs. For more information, download a copy of the demo software directly from Restek's website at <http://www.restekcorp.com> or call Technical Service at (800) 356-1688, ext. 4, and request a demo disk of Pro ezGC™ software for Windows®.

**Pro ezGC™ for Windows®  
version 2.0**

cat.# 21487 \$795

**Upgrade from Pro ezGC™ for  
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Windows®**

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# RESTEK

## Behind the Scenes

### Welcome, Barbara Lyke and Chris Lope!

**Restek On the Road Seminar...** June 13,  
St. Louis, MO

**Air & Waste Management Association's 90th Annual Meeting and Exposition...** June 8-13,  
Toronto, Canada.

### Restek On The Road

**Restek On the Road Seminar...** June 9,  
Minneapolis, MN

**Restek On the Road Seminar...** June 10,  
Detroit, MI

**Restek On the Road Seminar...** June 11,  
Rockford, IL

**Restek On the Road Seminar...** June 12,  
Chicago, IL

**IFT Food Expo...** June 14-18,  
Orlando, FL.

**NERM '97...** June 23-25,  
Saratoga, NY.

**Pesticide Residue Workshop...** July 13-16,  
St. Pete Beach, FL.

**111th AOAC International Annual Meeting and Exposition...** September 8-10,  
San Diego, CA.

**Gulf Coast Conference...** September 9-11,  
Galveston Island, TX.



**Barbara Lyke** is our new Manager of Information Services. Her background in IS has

been diverse and includes positions held at The Pennsylvania State University, Office of Business Services; Murata-Erie North America, Inc.; and International Business Machines, T.J. Watson Research Center. Barb is a graduate of Marymount College, Tarrytown, NY, with a B.S. in Business and has continued her education in computer science and business logistics at Pace and Penn State Universities. Barb will be leading Restek's IS group into the next century. No Y2K bugs here!



In his new role as Director of Sales and Domestic Distribution, **Chris Lope** will be responsible

for Restek's growing Technical Sales Force and its outstanding Customer Response Team, along with customer service and shipping. Additionally, he will manage distributor relations in the U.S. Chris' background in laboratory sales and management will help Restek continue its growth in the chromatography market. If you would like to discuss any issues relating to the sales and service of Restek products, please contact Chris at 800-356-1688, ext. 2175.

### Team Pennsylvania Comes to Restek

Team Pennsylvania is comprised of a 32-member board made up of top corporate executives and Governor Ridge's cabinet members that form a central business resource center. The purpose of this "team" is to visit businesses throughout the state, listen to their needs, and coordinate efforts by people across Pennsylvania to improve the business climate. It is intended to allow businesses to be treated as customers and to get the attention of state business leaders and elected officials. Restek, first on the list of businesses to visit, was selected because of the company's environmental track record, recent expansion, growth rate, and overall success.

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Bellefonte, PA 16823

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"some" **promos / Products / Offers** in the **ADVNews** have been since been progressively superceded / UPDATED OR Since Discontinued

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# RESTEK

## The Advantage

### Allure C18 HPLC Column

#### Specialized Columns for LC-MS

by Keith Duff & Dave Bell

Innovators of  
High Resolution  
Chromatography  
Products

#### in this issue

The development of High Performance Liquid Chromatography/Mass Spectrometry (HPLC-MS) has grown into one of the most powerful analytical techniques currently available. Among its advantages, HPLC-MS technology provides a high level of sensitivity previously not available, especially for weak or non-ultraviolet (UV)-absorbing compounds. The level of sensitivity is related to the percent organic content in the mobile phase solvent. As the mobile phase organic concentration increases (relative to water) the signal-to-noise ratio increases. Most commercial C18 phases only contain 10-15% carbon, which means that more water (less organic) must be used in the mobile phase to retain solutes. These phases result in low signal-to-noise ratios, making them less desirable for HPLC-MS applications.

The Allure™ C18 HPLC column is a high-carbon (27%), densely-bonded C18 phase that gives added retention for neutral to slightly polar solutes. Higher mobile phase organic concentrations can be used on

- Higher LC-MS sensitivity.
- Maximum retention of neutral to slightly polar solutes.
- Symmetrical peak shape for basic compounds.
- High reproducibility.
- Separation of all EPA Method 8330 explosives compounds on one column.
- Excellent stability towards hydrolysis.

this phase because of its extremely high retention. As a result, better HPLC-MS sensitivity is achieved.

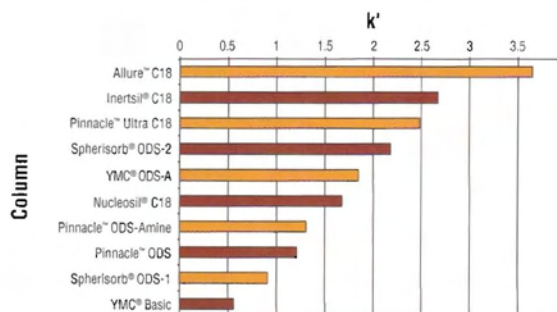
Table I shows increased retention for neutral compounds obtained on the Allure™ C18 phase, as compared to other

C18 phases. Slightly polar solutes, such as some steroids, also are retained longer on the Allure™ C18 phase than on other C18 phases (Figure 1). Note: The more neutral steroids are retained longer.

Continued on page 2.

Table I:

Relative Capacity Factors (k') Show Maximum Retention on the Allure™ C18 Column for Neutral Compounds



Column: Allure™ C18 (cat. # 9164565); Compound: biphenyl; Mobile Phase: water:methanol (15:85); Flow rate: 1.0mL/min; Wavelength: 254nm; Temperature: ambient.

- Allure™ C18 HPLC Columns ...pg. 1
- MXT® PLOT Columns ...pg. 3
- Optimizing VPH Analysis ...pg. 4
- Analysis of Endocrine Disruptors ...pg. 6
- Koni's Korner ...pg. 7
- Mass Spectrometry Columns ...pg. 8
- Restek & Hypersil Announce Partnership ...pg. 10
- Sample Cylinders & Valves ...pg. 12
- Advances in GC Septa ...pg. 13
- New Resprep™ Tubes, Disks, & Accessories ...pg. 14
- Peak Performers ...pg. 15
- Erik Steindl Scholarship ...pg. 16

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98

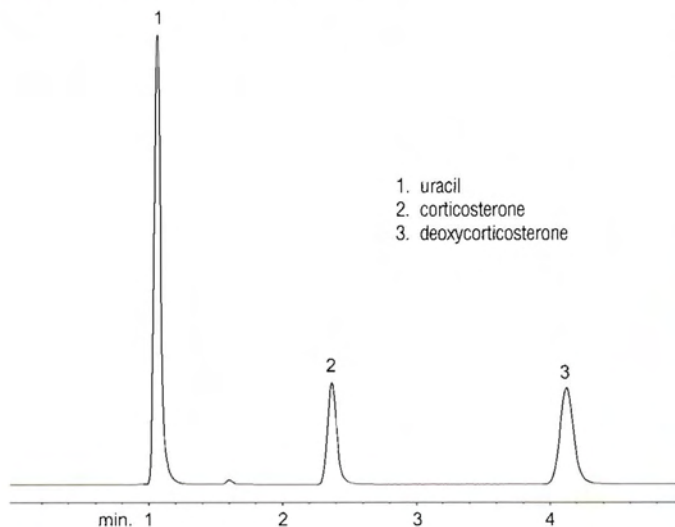


# Allure™ C18 HPLC Column

Continued from page 1.

Figure 1:

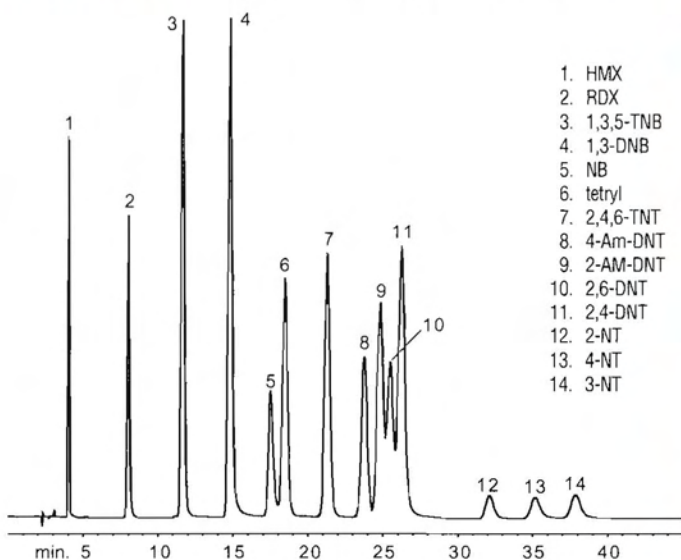
Corticosteroids on Allure™ C18 Column Show Higher Retention of Less Polar Compounds



Allure™ C18 (cat.# 9164565). Dimensions: 150 x 4.6mm ID, 5µm particles (60Å); Mobile phase: water:acetonitrile (40:60), v/v; Flow rate: 1ml/min.; Wavelength: 254nm.

Figure 2:

All EPA-8330 Explosives are Separated on One Column



Allure™ C18 (cat.# 9164575). Dimensions: 250 x 4.6mm ID, 5µm particles (60Å); Conc.: 5µl, 1mg/ml ea.; Mobile phase: water:methanol (50:50); Flow rate: 1mL/min.; Wavelength: 254nm.

Additionally, this column shows improved selectivities for explosive mixture separations. The Allure™ C18 column separates all of the EPA Method 8330 components by itself (Figure 2), whereas the method suggests using two columns (a lower coverage C18 and a cyano phase). The dense bonding in the Allure™ C18 phase also shields surface silanols, thereby providing resistance to hydrolysis and undesirable solute-silanol interactions. This means re-

duced peak tailing of basic solutes, even at neutral pH mobile phase conditions.

The Allure™ C18 column is an ideal HPLC-MS column for neutral to slightly polar solutes. It is highly stable, reproducible, and often more selective than ordinary C18 phases.

For more information on the Allure™ C18 column, call 800-356-1688, ext. 2302, to speak with Keith Duff, HPLC R&D group leader, or email him at keithd@restekcorp.com.

## Product Listing:

### Allure™ C18 HPLC Columns

5µm particles, 60Å pore size

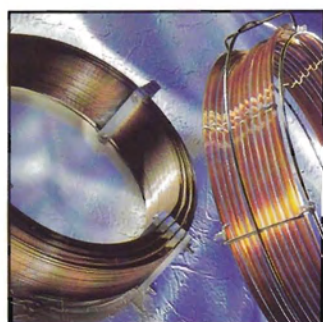
Description	cat. #	price
Bulk Silica, 5µm, 60Å	91645	\$35.00/g
<b>2mm ID</b>		
Length	cat.#	price
30mm	9164532	\$299.00
50mm	9164552	\$305.00
100mm	9164512	\$315.00
150mm	9164562	\$320.00
200mm	9164522	\$325.00
250mm	9164572	\$335.00
<b>3mm ID</b>		
30mm	9164533	\$305.00
50mm	9164553	\$315.00
100mm	9164513	\$320.00
150mm	9164563	\$330.00
200mm	9164523	\$340.00
250mm	9164573	\$355.00
<b>4.6mm ID</b>		
30mm	9164535	\$305.00
50mm	9164555	\$310.00
100mm	9164515	\$335.00
150mm	9164565	\$355.00
200mm	9164525	\$380.00
250mm	9164575	\$399.00
<b>Guard Columns</b>		
10mm, 2mm ID	916450212	\$125.00, 3-pk.
10mm, 4mm ID	916450210	\$125.00, 3-pk.
20mm, 4mm ID	916450220	\$125.00, 2-pk.



# 100% Bonded, Unbreakable MXT®-PLOT Columns

by Andy Schuyler

- No need for expensive particle traps.
- Same inertness and passivity as fused silica columns.
- Easy handling and installation.
- Fully interchangeable with fused silica columns.
- Expanded line of MXT® PLOT phases.



**Restek has engineered PLOT column technology to introduce a new line of unbreakable metal MXT®-PLOT columns.**

Our exclusive Silcosteel® treatment ensures that MXT®-PLOT columns are highly inert. They have been designed with the same dimensions as fused silica columns and are fully interchangeable in your GC. Additionally, the durable, flexible stainless steel tubing is ther-

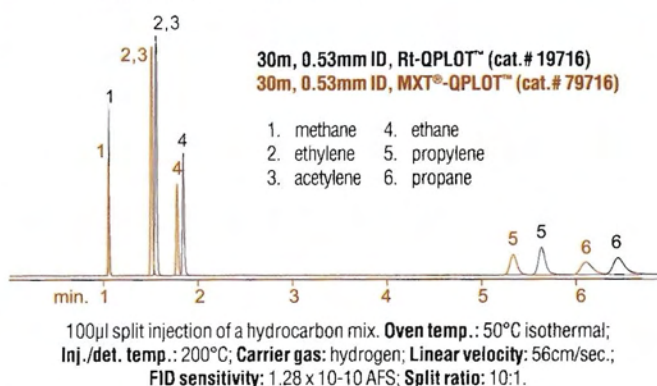
mally stable, easy to handle, and will not break.

In the past, PLOT column particle generation has forced chromatographers to install expensive particle traps to avoid signal spiking and instrument damage. The new MXT®-PLOT phases are 100% bonded, eliminating the need for specialized particle traps, regardless of repeated valve switching applications. Restek uses advanced technology for coating and bonding the stationary phase, which produces strong, uniform particle adherence to the inside of the capillary tube. (See **Table I** for a list of PLOT column phases.)

MXT®-PLOT columns offer a similar degree of inertness as fused silica PLOT columns. The MXT®-PLOT columns are constructed from stainless steel tubing that has been passivated using Restek's Silcosteel® treat-

**Figure 1:**

**Metal MXT® and Fused Silica Rt-PLOT Columns Show Similar Selectivity and Efficiency.**



ment, a process that bonds a layer of inert material to the interior wall of the tubing. Once the tubing is fully deactivated, the same porous polymers used to make Restek's fused silica PLOT columns are bonded onto the Silcosteel® surface to create MXT®-PLOT columns.

These columns have the same inner and outer dimensions as 0.53mm ID fused silica PLOT columns, and require no special ferrules, making them easily interchangeable with existing fused silica PLOT columns. No additional hardware is necessary to install MXT®-PLOT columns, and they can be cut using the same ceramic scoring wafers.

umns, as shown in **Figure 1**, with the added advantage of resisting abrasion, scratches, and spontaneous breakage.

MXT®-PLOT columns can be repeatedly programmed to high temperatures without fear of breakage or degradation, making them ideal for high-temperature analysis (see **Table I**). They are practical for use in small ovens, portable gas chromatographs, and process analyzers because they are flexible and can be coiled into smaller diameters (3.5"). In addition, the unbreakable stainless steel construction is appropriate for GCs transported in off-road vehicles to hazardous sites.

MXT®-PLOT columns provide similar resolution and retention times as fused silica PLOT col-

For more column-specific application information, request our new *Bonded PLOT Column* product guide, lit. cat. #59970.

**Table I:**

## Ideal Uses of MXT®-PLOT Columns.

MXT®-Msieve 5A (up to 350°C)	Argon/oxygen separation and gas purity analysis, e.g., semiconductor industry.
MXT®-Msieve 13X (up to 350°C)	H <sub>2</sub> , O <sub>2</sub> , N <sub>2</sub> , CH <sub>4</sub> and CO in natural and refinery gases.
MXT®-QPLOT (up to 250°C)	Light hydrocarbons and volatile polar organics such as solvents and alcohols.
MXT®-SPLOT (up to 250°C)	Light gases in ethylene and propylene; ketones and esters.
MXT®-UPLOT (up to 190°C)	Polar volatiles, nitriles, nitro compounds, alcohols, aldehydes, ethane/ethylene

## Product List

	15m, 0.53mm ID		30m, 0.53mm ID	
MXT®-Msieve 5A	79721	\$325	79723	\$500
MXT®-Msieve 13X	79708	\$310	79706	\$445
MXT®-QPLOT	79715	\$310	79716	\$445
MXT®-SPLOT	79713	\$290	79712	\$425
MXT®-UPLOT	79727	\$310	79726	\$445

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800~356~1688



# Optimizing Massachusetts Volatile Petroleum Hydrocarbon GC Analysis

by Christopher English and Eric Steindl

**Total Petroleum Hydrocarbons (TPH)** analysis includes the characterization of dozens of commercially available petroleum products, which are the most prevalent environmental pollutants. The two fractions of TPH—volatile gasoline range organics (GRO), also called petroleum volatile organic compounds (PVOC), and the semi-volatile diesel range organics (DRO)—are analyzed differently due to their different boiling points.

Typical methods for the identification of gasoline use early and late eluting compounds to determine an analytical window for total gasoline quantitation. Then, GC analysis uses pattern recognition, the specific ratio of peaks that make up a particular compound, to identify a fuel. If a pattern falls within this window, it may be reported as gaso-

line and quantified. Difficult matrices can result in misidentification or poor quantitation of the sample, and environmental degradation (i.e., weathering) further complicates this analysis.

On January 1, 1998, the Massachusetts Department of Environmental Protection (MADEP) promulgated a new method, known as Volatile Petroleum Hydrocarbons (VPH) to better quantify gasolines. This method identifies and evaluates PVOCs by differentiating and characterizing the aromatic and aliphatic fractions of gasoline using a photo ionization detector (PID) and a flame ionization detector (FID) in series. The data generated from this method will aid in evaluating human health hazards that may result from exposure to PVOCs. Other states in the US and provinces in Canada have adopted the VPH method

for use in remediation, site characterization, and toxicity data (standards for other methods are listed in the UST Product Listing, lit. cat. #59617).

## Difficulties with the Analysis

A large proportion of VPH samples are soil. The soil is weighed in the field and an equal amount of methanol is added at the time of sampling. 100ul of methanol extract is added to 4.9ml of water, to be purged. The problem is that most purge-and-trap concentrators were not designed to have large amounts of methanol purged onto their absorbent beds. The VOCARB™ 3000 & VOCARB™ 4000 traps both have difficulty retaining n-pentane and 2-methylpentane after repeated exposure to methanol. This causes poor linearity of n-pentane and 2-methylpentane. We suggest using a BTEX trap

because of its non-polar properties. You will experience a slight decrease in response for methyl-tert-butyl-ether, but it will not compromise your detection limit.

## Column Selection

All purge-and-trap methods for this analysis result in broad, early eluting peaks. Therefore, choosing the right chromatographic column can prevent coelutions and poor quantitation. Many columns may drastically change the quantitation of aliphatic and aromatic compounds, or may suffer from poor resolution of methanol and methyl-tert-butyl-ether, or poor separation of pentane and 2-methylpentane. For optimized separation of light hydrocarbons and light gas additives, use the Rtx™-502.2 column specified in the VPH method.

Figure 1:

For VPH Analysis on an Rtx®-502.2 Column, Use PID for Aromatic Compounds and FID for Aliphatic Compounds.

Figure 1A: PID

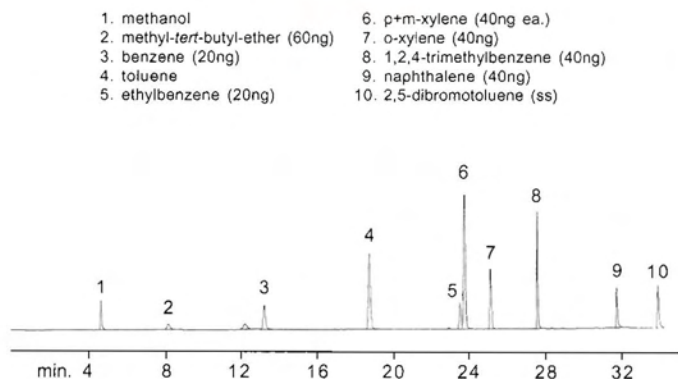
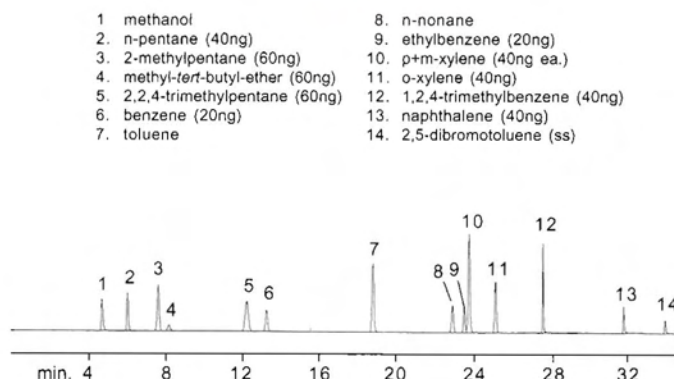


Figure 1B: FID



105m, 0.53mm ID, 3.0µm (cat.# 10910). **Concentration:** on-column at levels listed; **Oven temp:** 45°C to 90°C @ 3°C/min., to 140° @ 5°C/min., to 230°C @ 45°C/min. (hold 8 min.); **Carrier gas:** helium @ 15ml/min.; Tekmar® Model LSC 2000; **Trap:** BTEX; **Purge:** helium @ 40ml/min. for 11 min.; **Dry purge:** 2 min.; **Desorb preheat:** 245°C; **Desorb:** 2 min. @ 250°C; **Bake:** 6 min. @ 260°C. Chromatograms courtesy of Severn Trent Laboratories, Burlington, VT.



Figure 1 is an example of VPH compounds separated on an Rtx™-502.2 column, using a PID (Figure 1A) and an FID (Figure 1B). The first peak in the chromatogram is methanol. Notice that it is clearly separated from the methyl-*tert*-butyl-ether and n-pentane.

The PID is used to identify target VPH analytes, defined as benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, naphthalene, and methyl-*tert*-butyl-ether. The VPH method identifies all of the aromatic compounds from just after o-xylene to just before naphthalene. The reason for the distinction of aromatic from aliphatic compounds rests on current data, which suggests aromatic compounds are more toxic.

The FID detector is used only for reporting aliphatic compounds. This detector responds to all hydrocarbons, thereby necessitating the subtraction of aromatic and target compounds. The latter analytes are subsequently reported on the PID

detector. There are two aliphatic ranges: C5 to C8, which elute from n-pentane to just before n-nonane; and C9 to C12, which elute from n-nonane to just before naphthalene.

#### Standards

VPH standards are a mixture of 13 components, provided in three different concentrations. These 13 compounds allow for the determination of three groups of analytes in the sample: total gasoline, aliphatic and aromatic hydrocarbons, and individual target compounds. Restek offers these standards complete with QA documentation and a Certificate of Analysis that can be used for audits and internal QA departments. Our VPH standards are made in the appropriate concentrations for spiking, ensuring accurate identification and quantitation of environmental samples in your laboratory. Restek also offers high concentration mixes that can be diluted down to a spiking concentration, useful for cost savings.

#### Product Listing:

##### Rtx®-502.2 Columns (Fused Silica)

ID	df (µm)	30m	60m	105m
0.25mm	1.40	10915 \$415	10916 \$660	
0.32mm	1.80	10919 \$440	10920 \$720	10921 \$900
0.53mm	3.00	10908 \$495	10909 \$770	10910 \$995
ID	df (µm)	20m	40m	
0.18mm	1.00	40914 \$350	40915 \$625	

##### MXT®-502.2 Columns (Silcosteel®)

ID	df (µm)	30m	60m	105m
0.28mm	1.60	70919 \$440	70920 \$720	70921 \$900
0.53mm	3.00	70908 \$495	70909 \$770	70910 \$995

**For a complete product listing of TPH and EPH standards, call to request lit. cat.# 59617.**

#### Analytical Reference Materials

##### MA VPH Standard

benzene	500µg/ml	n-pentane	1000µg/ml
ethylbenzene	500	toluene	1500
isooctane	1500	1,2,4-trimethylbenzene	1000
2-methylpentane	1500	m-xylene	1000
methyl- <i>tert</i> -butyl-ether	1500	o-xylene	1000
naphthalene	1000	p-xylene	1000
n-nonane	1000		

in P&T methanol, 1ml/ampul

	Each	5-pk.	10-pk.
	30434 \$25	30434-510 \$112.50	
w/data pack	30434-500 \$35	30434-520 \$125	30534 \$225

##### MA VPH Standard with Surrogate

benzene	500µg/ml	n-pentane	1000µg/ml
ethylbenzene	500	toluene	1500
isooctane	1500	1,2,4-trimethylbenzene	1000
2-methylpentane	1500	m-xylene	1000
methyl- <i>tert</i> -butyl-ether	1500	o-xylene	1000
naphthalene	1000	p-xylene	1000
n-nonane	1000	2,5-dibromotoluene*	1000

in P&T methanol, 1ml/ampul

\*surrogate

	Each	5-pk.	10-pk.
	30452 \$30	30452-510 \$135	
w/data pack	30452-500 \$40	30452-520 \$150	30552 \$270

##### MA VPH Surrogate Standard

2,5-dibromotoluene

1000µg/ml in P&T methanol, 1ml/ampul

	Each	5-pk.	10-pk.
	30435 \$20	30435-510 \$90	
w/data pack	30435-500 \$30	30435-520 \$100	30535 \$180

10,000µg/ml in P&T methanol, 1ml/ampul

	Each	5-pk.	10-pk.
	30453 \$20	30453-510 \$90	
w/data pack	30453-500 \$30	30453-520 \$100	30553 \$180

##### MA VPH Matrix Spike Mix with Surrogate

benzene	n-pentane
ethylbenzene	toluene
isooctane	1,2,4-trimethylbenzene
2-methylpentane	m-xylene
methyl- <i>tert</i> -butyl-ether	o-xylene
naphthalene	p-xylene
n-nonane	2,5-dibromotoluene (surrogate)

2500µg/ml each in P&T methanol, 1ml/ampul

	Each	5-pk.	10-pk.
	30454 \$30	30454-510 \$135	
w/data pack	30454-500 \$40	30454-520 \$150	30554 \$270

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800-356-1688



# GC-MS Analysis of Alkyl Phenols and Bisphenol A

## Endocrine Disrupters\*

by Frank Dorman and Chris Cox

\*Part of a series on endocrine disrupters. See Fall 1997 Restek Advantage for analysis of organotin compounds.

Recently, there has been considerable interest in the analysis of endocrine disrupting chemicals. Several countries are actively investigating the effects of these compounds and beginning to research potential methods of preparation and analysis.<sup>1,2</sup> In addition, endocrine disrupters have become a focal point of several recent chemistry conventions, including the American Chemical Society (ACS) national meeting. Although many groups are very active in this research, there are few accepted or standard methods of analysis, and there is still considerable disagreement as to which compounds should be studied and how they should be prioritized.

Phenol compounds have long been used in the manufacture of plastics, and, until very recently, were not routinely monitored. Since the discovery that these compounds can exhibit estrogenic activity, they have become of great interest. Additionally, since these compounds often were not regulated, they typically can be found in the environment, which further intensifies public interest in them.<sup>3</sup>

Although no US EPA method exists for these specific compounds, it is possible to perform the sample preparation and analysis using an Rtx®-5MS column and the equipment commonly found in an environmental laboratory. The compounds can be extracted conventionally under acidic conditions following US EPA Methods 3510 (separatory funnel) or 3520 (liquid-liquid) for water samples,

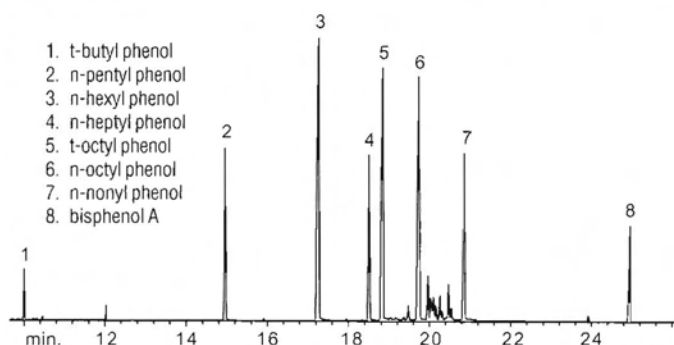
and US EPA Methods 3540 (soxhlet) or 3550 (sonication) for solid matrices. Alternatively, SPE, microwave, and pressurized fluid extraction also could be used. These methods produce a methylene chloride extract, which is then dried using granular sodium sulfate and concentrated to a final volume. For biota or soil samples with lipid or other high molecular weight contaminants, gel permeation chromatography (GPC) should be used to clean the extracts prior to final concentration.

Because most methods of endocrine disrupter analysis will require very low levels of detection, we suggest forming trimethylsilyl derivatives of the compounds of interest before analysis. The trimethylsilyl derivatives of phenols are best produced using the reagent N,O-bis(trimethylsilyl)acetamide (commonly sold neat, under the acronym of BSA). To prepare the derivatives, add 0.5ml of the methylene chloride extract to 0.5ml of pure BSA in a 1.5ml autosampler vial. Crimp the vial with a Teflon®-faced aluminum seal, shake well, and heat at 30°C for 1 hour. The sample is now ready to analyze. The excess reagent will elute before the compounds of interest. This derivatization method is best applied to extracts produced from an SPE-type extraction.

Alternatively, if beginning with larger volumes of solvent (e.g., sample extracts that will be concentrated by Kuderna-Danish evaporation), the following method is suggested. Add 1ml BSA and 1ml dimethyl

**Figure 1:**

**Separation of Alkyl Phenols and Bisphenol A on an Rtx®-5MS Column for Easy Identification of Endocrine Disrupters.**



30m, 0.25mm ID, 0.25µm Rtx®-5MS (cat.# 12623). **Concentration:** 5-10ng on-column; **Injection:** Splitless, purge on @ 1 min.; **Oven temp.:** 35°C (hold 1 min.) to 300°C @ 10°C/min. (hold 15 min.); **Inj./det. temp.:** 275°C/310°C; **Carrier gas:** He.

formamide for every 100mls of methylene chloride extract to be concentrated. The sample should remain heated for at least 30 minutes after exchange to dimethyl formamide/BSA.

**Figure 1** shows the GC-MS chromatogram obtained from the injection of 5 to 10ng of the phenolic compounds, using an Rtx®-5MS column. The smaller peaks eluting prior to the nonylphenol are branched chain isomers found in commercially available nonylphenol materials. These are found in nonylphenol obtained from any manufacturer, and need to be addressed in the quantitation of this compound.

This procedure provides a simple method for the analysis of phenolic endocrine disrupters in the environmental testing and plastics industries.

For additional information on this method, please contact Frank Dorman at 800-356-1688, ext. 2186. Restek also offers analytical standards for these compounds at custom concentrations to suit your application—call for details.

### References

1. *Special Report on Environmental Endocrine Disruption: An Effects Assessment and Analysis*, EPA/630/R-96/012, Feb. 1997.
2. *JAIMA '98*, Tokyo, Japan.
3. *The Boston Globe*, Vol. 255, #58, p. 1, 1998.

### Product Listing:

#### Rtx®-5MS Column

ID	df (µm)	temp. limits	15-meter	30-meter
0.25	0.25	-60 to 330/350	12620 \$260	12623 \$415

See page 9 for a complete listing of Restek's MS columns.

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# Koni's Korner

## Do We Need a Chromatography School?

by Dr. Konrad Grob

**Sometimes things  
need to be said**

**in a pointed way.**

**The following is  
one of those things.**

**If even less than 50%  
is applicable, then  
it is enough to  
sound an alarm.**

### *The Slow Sinking of Chromatography*

I grew up in the good old times when the world believed in chromatographers. We were proud of what we did, could work in our own way, but were also responsible for our results — we were our own maestros, playing our chromatographs with the best of melodies. But, then misery befell us. It was suspected our results were faked. Bureaucratic methods like Good Laboratory Practices (GLP) were invented and, ever since, many of us must document every move we make. We wanted to work in the lab, not do office jobs on an uncomfortable lab chair, didn't we?

Confidence in our results was lost almost completely. This has its origin in embarrassingly poor results delivered by some labs, but also in the difficulty of the public to understand that our results may have a substantial margin. They may think that if an instrument is expensive, the results must be absolutely accurate. At court, results are no longer questioned on their accuracy, but on the paperwork behind them: when was the balance checked last, and is there a certificate proving that the hexane was not water. Shouldn't

we feel offended by such general distrust? As a consequence, much of our work has become regulated in minute detail, with many ideas having a penetrating odor of stacks of paperwork and meeting rooms. Many methods grew to be more than 10 pages, half of which are taken up with titles and decimal numbering. They specify standard compounds in every detail, as well as simple manipulations like how to rinse a round flask. However, they all too often do not even mention the tasks that cause real problems, such as the details on how to perform injection techniques. Have we lost all of our competence or are some totally unskilled lab workers dictating our lab?

### *Lab Work Degrades*

In many labs, work has become dull. It has degraded to the execution of recipes — as mundane as making hamburgers, only having more frustrations and a lower success rate. Your results are accepted only if you have a validated method and a certified standard. Many methods could be greatly improved, saving large amounts of time and money, but changes presuppose such a large amount of rework that most people just keep quiet. It is performing analyses in chains. Many of the best chromatographers are no longer finding enjoyment in such work.

Commonly, lab supervisors are no longer in the lab. They are chemists who learned hardly more about chromatography than interpreting a van Deemter curve. For them, going into the lab might mean learning the basics from the supervised — it is easier to design Excel spread-

sheets and manage quality from a safe distance. Analytical refugees because of a lack of chromatography education?

It is sad to see such an exciting field declining. GC would not be the first chromatographic technique to degrade. Thin layer chromatography (TLC) is an excellent method for many purposes, much faster and cheaper than some other methods. However, where could I learn the art of TLC today?

### *Quality Management, a Make-shift Solution*

Much of quality management (QM) resembles a desperate support structure used to stop the decay of analytical chemistry; a sophisticated system to protect against a lack of competence. However, in reality, QM might even accelerate the decay as it chases away the good analysts by way of boredom and frustration. Many newcomers have no proper education and will hardly develop a passion for the work they encounter, and only look at peaks when asked to do so in **bold letters**. Many laboratories have lost their competence to create or modify methods. In the end, the pessimists are right: merely the most rigid and painstaking descriptions prevent people from doing the incredible things that many technical support services hear about every day. Analytical chemistry risks collapse, despite - or maybe because of - the rapidly growing QM systems.

### *A Need for Better Education*

Is there anything we can do to stop this decline? The key problem concerns competence of the analysts. Chromatography is demanding and requires profes-

sionally trained people. Analysts must be masters of their field, motivated to do their work well and react promptly if something peculiar is observed. They should feel responsible for their results, but also be recognized for performing a difficult job. Supervisors must be knowledgeable leaders in chromatography, guide with ideas and suggestions, understand problems, as well as pick up and support the good ideas of their people. Emphasis must be moved from paper-leaden QM systems towards ensuring the competence of the analysts.

Many of the modern technologies suffer from lacking education. In new fields, the originators are the natural teachers, informally passing on their knowledge. When they leave, classical education should take over. However, universities are unable to offer this service for all the emerging fields and there is not enough room for chromatography in the teaching of general chemistry. Hence, new models of education are needed. We might also need an internationally recognized qualification system, so that well-trained chromatographers are recognized as specialists in their field. The problem is serious and a great challenge for all who are willing to keep alive a field in which more than 500,000 persons are working.

**Dr. Konrad Grob can be  
reached by e-mail at  
koni@grob.org**



# Low-Bleed MS Columns for Capillary Gas Chromatography-Mass Spectrometry (GC-MS) Applications

Restek has recently developed new chemistry that allows us to manufacture even better low-bleed MS phases. These MS columns exhibit a much lower column bleed than was previously available.

Conventional capillary gas chromatography (GC) columns use liquid stationary phases, many of which are crossbonded to yield a higher working temperature. Even with crossbonding, however, the liquid stationary phase will slowly elute. This elution of the stationary phase, also termed column bleed, is more detectable at higher temperatures and is typically observed as an increasing baseline that follows the oven temperature program.

Depending on the method of detection, column bleed may not be an issue for certain separations. If the capillary column is connected to a sensitive detector like a mass spectrometer (MS), then column bleed can cause a number of problems—specifically misidentification of analytes, loss of sensitivity, and inaccurate quantitation.

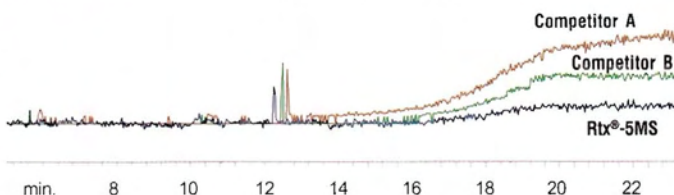
The level of column bleed affects the sensitivity of any MS, especially ion trap instruments, which utilize automatic gain control. As the level of column bleed increases, so does the signal—from bleed ions in the

mass spectra of analytes and unknowns—and the sensitivity (or detection limit) severely degrades. The contribution of bleed ions to the mass spectra can result in misidentification of compounds, requiring laboratory personnel to subtract these ions before performing library searches. Doing this can add considerable time to the analyses. Finally, because bleed ions contribute to the signal of the quantitation mass, quantitation of analytes and unknowns will be miscalculated. For these reasons, it is critical that analysts choose the lowest-bleed columns available for GC-MS applications.

Many manufacturers offer “MS” phases for applications requiring low bleed. In many cases, this represents nothing more than the reporting of the bleed signal when the column was tested at the manufacturer for a single analysis. Restek has recently developed new chemistry that allows us to manufacture true low-bleed MS phases. These columns exhibit a much lower column bleed than was previously available (Figure 1). We offer—in addition to the existing Rtx®-5MS column—Rtx®-1MS, Rtx®-200MS, and Rtx®-35MS columns designed specifically for GC-MS analyses. Even the trifluoropropylmethyl polysiloxane Rtx®-200MS column exhibits low bleed at 300°C as demonstrated in Figure 2. The addition of these columns gives GC-MS analysts a wide range of stationary phases to choose from, while guaranteeing low bleed.

Figure 1:

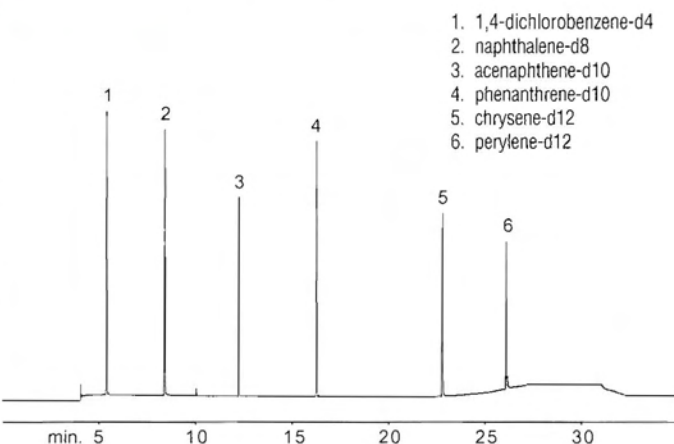
The Restek Rtx®-5MS Exhibits the Lowest Bleed.



30m, 0.25mm ID, 0.25µm Rtx®-5MS (cat.# 12623); Oven temp.: 50°C to 325°C @ 15°C/min. (hold 5 min.); Inj./det. temp.: 275°C/325°C; Detector: MSD; Flow rate: 35cm/sec. He @ 50°C; Scan parameters: TIC, low mass 50, high mass 550, threshold 200; Ion 207.00: (206.70 to 207.70).

Figure 2:

The Restek Rtx®-200MS Column Performs Highly Sensitive Analyses for Ion Trap GC-MS Applications.



30m, 0.25mm ID, 0.25µm Rtx®-200MS (cat.# 15623); 1.0µl split injection of a semi-volatile internal standard; On-column conc.: 5ng; Oven temp.: 40°C to 300°C @ 10°C/min. (hold 5 min.); Inj. program: 40°C to 280°C @ 50°C/min.; Det. type: Ion Trap GC/MS; Ionization: EI; Carrier gas: Helium; Scan range: 50-400m/z; Linear velocity: 27cm/sec. @ 40°C; Split ratio: 40:1.

In addition to Restek's new chemistry, our rigorous QA testing includes MS polymer batch testing and individual column testing for inertness, efficiency, and bleed using a

special test mix designed for environmental analysis. This combination ensures that each MS column exceeds requirements of the most sensitive mass spectrometers.



### Rtx®-1MS

*Best for GC/MS Analysis of Air Pollutants*

ID (mm)	df (µm)	temp. limits (°C)	length	
			15-meter	30-meter
0.25	0.10	-60 to 330/350	11605 \$270	11608 \$425
	0.25	-60 to 330/350	11620 \$260	11623 \$415
	0.50	-60 to 330/350	11635 \$260	11638 \$415
	1.00	-60 to 325/350	11650 \$260	11653 \$415
0.32	0.10	-60 to 330/350	11606 \$290	11609 \$455
	0.25	-60 to 330/350	11621 \$280	11624 \$445
	0.50	-60 to 330/350	11636 \$280	11639 \$445
	1.00	-60 to 325/350	11651 \$280	11654 \$445
0.53	0.50	-60 to 320/340	11637 \$285	11640 \$495
	1.00	-60 to 320/340	11652 \$285	11655 \$495
	1.50	-60 to 310/330	11667 \$285	11670 \$495

### Rtx®-5MS

*Ideal for Semi-Volatile Analyses*

ID (mm)	df (µm)	temp. limits (°C)	length	
			15-meter	30-meter
0.25	0.10	-60 to 330/350	12605 \$270	12608 \$425
	0.25	-60 to 330/350	12620 \$260	12623 \$415
	0.50	-60 to 330/350	12635 \$260	12638 \$415
	1.00	-60 to 325/350	12650 \$260	12653 \$415
0.32	0.10	-60 to 330/350	12606 \$290	12609 \$455
	0.25	-60 to 330/350	12621 \$280	12624 \$445
	0.50	-60 to 330/350	12636 \$280	12639 \$445
	1.00	-60 to 325/350	12651 \$280	12654 \$445
0.53	0.50	-60 to 320/340	12637 \$285	12640 \$495
	1.00	-60 to 320/340	12652 \$285	12655 \$495
	1.50	-60 to 310/330	12667 \$285	12670 \$495
ID (mm)	df (µm)	temp. limits (°C)	length	
			60-meter	
0.25	0.10	-60 to 330/350	12611	\$665
	0.25	-60 to 330/350	12626	\$665
	0.50	-60 to 330/350	12641	\$665
0.32	0.10	-60 to 330/350	12612	\$725
	0.25	-60 to 330/350°C	12627	\$725
	0.50	-60 to 330/350°C	12642	\$725

### Rtx®-35MS

*Excellent Confirmational Column for MS Analyses*

ID (mm)	df (µm)	temp. limits (°C)	length	
			15-meter	30-meter
0.25	0.10	-60 to 330/350	14605 \$270	14608 \$425
	0.25	-60 to 330/350	14620 \$260	14623 \$415
	0.50	-60 to 330/350	14635 \$260	14638 \$415
	1.00	-60 to 325/350	14650 \$260	14653 \$415
0.32	0.10	-60 to 330/350	14606 \$290	14609 \$455
	0.25	-60 to 330/350	14621 \$280	14624 \$445
	0.50	-60 to 330/350	14636 \$280	14639 \$445
	1.00	-60 to 325/350	14651 \$280	14654 \$445
0.53	0.50	-60 to 320/340	14637 \$285	14640 \$495
	1.00	-60 to 320/340	14652 \$285	14655 \$495
	1.50	-60 to 310/330	14667 \$285	14670 \$495

### Rtx®-200MS

*Preferred for Ion Trap Analyses*

ID (mm)	df (µm)	temp. limits (°C)	length	
			15-meter	30-meter
0.25	0.10	-20 to 320/340	15605 \$270	15608 \$425
	0.25	-20 to 320/340	15620 \$260	15623 \$415
	0.50	-20 to 310/330	15635 \$260	15638 \$415
	1.00	-20 to 290/310	15650 \$260	15653 \$415
0.32	0.10	-20 to 320/340	15606 \$290	15609 \$455
	0.25	-20 to 320/340	15621 \$280	15624 \$445
	0.50	-20 to 310/330	15636 \$280	15639 \$445
	1.00	-20 to 290/310	15651 \$280	15654 \$445
0.53	0.50	-20 to 300/320	15637 \$285	15640 \$495
	1.00	-20 to 290/310	15652 \$285	15655 \$495
	1.50	-20 to 280/300	15667 \$285	15670 \$495

**"We are confident that our MS columns will perform to the level of quality you've come to expect from Restek. Call Restek, or contact your local distributor, and ask about a 30-day free evaluation!"**

**Rick Crago**  
Fused Silica  
Product Line Manager



**Restek Trademarks:** Allure, Diskcover, IceBlue, Leak Detective, MXT, MXT-QPLOT, Pinnacle, RedLite, Resprep, Res-Sil, Rt-QPLOT, Rtx, Silcosteel, Stabilwax, Thermolite, and the Restek logo.

**Restek Service Marks:** Restek Analytical Services and The Future of Chromatography.

**Other Trademarks & Registered Trademarks:** BioRex (Bio-Rad Laboratories), Carbowax (Union Carbide Corp.), Durapak (Waters), Hyperbond (Hypersil), Hypersil (Hypersil), HyPurity (Hypersil), Inertsil (GL Sciences), KEL-F (3M Co., Chemical Div.), µBondapak (Waters), Microseal (Merlin Instrument Co.), Nucleosil (Macherey-Nagel), Porasil (Waters), Spherisorb (Phase Separations), Tekmar (Tekmar, Co.), VOCARB (Supelco, Inc.), and YMC (Waters).

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# Restek and Hypersil™ Announce Strategic Partnership

- Expanded column line gives Restek customers "one-stop shopping."
- Expanded technical support for Hypersil™ column purchasers.
- RAS cGMP method development and validation services.
- Laboratory and regulatory services.
- Continuous, aggressive R&D to develop new phases.

Restek Corporation is proud to formally announce a strategic partnership with Hypersil®. By forming this relationship, Restek can now offer the entire line of superior-quality Hypersil® columns. We are especially proud to offer the new HyPurity™ Elite columns, which combine the unrivaled chromatographic performance of a robust bonded phase on a high-purity silica.

## Hyperbond™ Columns

*Manufactured by Hypersil*

**Chromatographic Properties:** Since  $\mu$ Bondapak™ is only available from one manufacturer, a demand has grown for a more cost-effective column of equivalent performance from an alternative source. The Hyperbond™ C18 column provides equivalent performance and selectivity, at a lower cost.

**Physical Characteristics:** Particle size: 10 $\mu$ m, irregular; Not end-capped; Pore size: 150Å; pH Range: 2.5 to 7.5; Average carbon load: 10%

C18			
Particle size	Dimensions	cat.#	price
10 $\mu$ m	300 x 3.9mm	9260000301	\$328
Guard Column			
10 $\mu$ m	30 x 3.9mm	9260000307	\$210

## HyPurity™ Elite Columns

*Manufactured by Hypersil*

**Chromatographic Properties:** The HyPurity™ Elite C18 column is the first column to be based upon the new HyPurity™ silica\*. By bonding this unique, new HyPurity™ silica using a specialized procedure, universally applicable HyPurity™ Elite C18 columns are produced. These columns are then scrutinized using the extensive testing procedure established specifically for Hypersil® Elite columns. The HyPurity™ Elite column is the ultimate in chromatographic validation.

**Physical Characteristics:** Particle size: 5 $\mu$ m, spherical; Pore size: 180Å; pH Range: 0.9 to 9.0; Average carbon load: 13%

Elite C18			
Particle size	Dimensions	cat.#	price
5 $\mu$ m	30 x 4.6mm	9222105066	\$350
5 $\mu$ m	50 x 4.6mm	9222105065	\$350
5 $\mu$ m	50 x 2.1mm	9222105089	\$350
5 $\mu$ m	100 x 3.0mm	9222105078	\$350
5 $\mu$ m	100 x 4.6mm	9222105064	\$350
5 $\mu$ m	100 x 2.1mm	9222105088	\$350
5 $\mu$ m	125 x 4.0mm	9222105071	\$375
5 $\mu$ m	150 x 3.0mm	9222105077	\$375
5 $\mu$ m	150 x 4.6mm	9222105062	\$375
5 $\mu$ m	150 x 2.1mm	9222105087	\$375
5 $\mu$ m	250 x 3.0mm	9222105075	\$400
5 $\mu$ m	250 x 4.6mm	9222105060	\$400
5 $\mu$ m	250 x 2.1mm	9222105085	\$400
5 $\mu$ m	250 x 4.0mm	9222105070	\$400
Guard Cartridge			
5 $\mu$ m	10 x 4.6mm	9222105105	\$370/4-pk.

\*This pure silica is essentially metal-free, and the C18 phase is exceptionally stable even at pH extremes. The HyPurity™ manufacturing process ensures the highest levels of reproducibility and quality, and the homogeneous surface ensures uniform bonding coverage. Only the purest grade solvents and raw materials are used, and each component is quality assured.



Restek Analytical Services<sup>SM</sup> (RAS) is a unique chromatography laboratory designed to deliver both quality services and quality products. The ultimate goal of RAS is to provide total security to the customer by being the sole product-service supplier, beginning with stationary phase raw material and ending with a validated chromatographic method. By offering this customized product-service package to our clients, RAS redefines the industry and truly becomes "The Future of Chromatography<sup>SM</sup>."

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## Hypersil® HPLC Columns

Manufactured by Hypersil

**Chromatographic Properties:** Hypersil® stationary phases are widely recognized as an industry standard in HPLC. Hypersil® is manufactured under strict quality guidelines and standards as indicated by BS 5750 part 1 and ISO 9001 accreditation.

**Physical Characteristics:** Particle size: 5µm, spherical; Fully end-capped; Pore size: 120Å (130Å for BDS C18); pH Range: 2.5 to 7.5; Average carbon load: [ODS 10%] [BDS C18 12%] [C8 7%] [BDS C8 7%] [CPS-2 4%] [APS-2 2%] [Phenyl-2 5%] [SAS 3%]

ODS			
Particle size	Dimensions	cat.#	price
3µm	100 x 4.6mm	9230103064	\$305
3µm	150 x 4.6mm	9230103062	\$315
5µm	150 x 4.6mm	9230105062	\$285
5µm	250 x 4.6mm	9230105060	\$310
5µm	100 x 10mm	9230105122	\$800
5µm	100 x 21.2mm	9230105127	\$1800
5µm	150 x 10mm	9230105121	\$900
5µm	150 x 21.2mm	9230105126	\$2000
5µm	250 x 10mm	9230105120	\$1000
5µm	250 x 21.2mm	9230105125	\$2000
Guard Cartridge			
5µm	10 x 4.6mm	9230105105	\$140/4-pk.
BDS C18			
3µm	50 x 4.6mm	9228103123	\$330
3µm	100 x 4.6mm	9228103064	\$330
3µm	150 x 4.6mm	9228103062	\$350
5µm	100 x 10mm	9228105122	\$800
5µm	100 x 20mm	9228105127	\$1000
5µm	150 x 10mm	9228105121	\$1500
5µm	150 x 21.2mm	9228105126	\$3000
5µm	250 x 10mm	9228105120	\$2000
5µm	250 x 21.2mm	9228105125	\$4500
Guard Cartridge			
5µm	10 x 4.6mm	9228105105	\$140/4-pk.
MOS-2 (C8)			
3µm	100 x 4.6mm	9230303064	\$305
3µm	150 x 4.6mm	9230303062	\$315
5µm	150 x 4.6mm	9230305062	\$285
5µm	250 x 4.6mm	9230305060	\$310
BDS (C8)			
3µm	100 x 4.6mm	9228203064	\$330
3µm	150 x 4.6mm	9228203062	\$350
5µm	150 x 4.6mm	9228205062	\$325
5µm	250 x 4.6mm	9228205060	\$350



Please call to request the new HPLC products and services catalog (lit. cat.# 59968).

### Hypersil® HPLC Columns (cont.)

CPS-2 (Cyano)			
5µm	150 x 4.6mm	9231805062	\$285
5µm	250 x 4.6mm	9231805060	\$310
APS-2 (Amino)			
3µm	100 x 4.6mm	9230703064	\$305
3µm	150 x 4.6mm	9230703062	\$315
5µm	150 x 4.6mm	9230705062	\$285
5µm	250 x 4.6mm	9230705060	\$310
Phenyl-2			
5µm	150 x 4.6mm	9231905062	\$285
5µm	250 x 4.6mm	9231905060	\$310
Silica			
3µm	100 x 4.6mm	9230003064	\$295
3µm	150 x 4.6mm	9230003062	\$305
5µm	150 x 4.6mm	9230005062	\$275
5µm	250 x 4.6mm	9230005060	\$300
SAS Methyl			
3µm	100 x 4.6mm	9230503064	\$305
3µm	150 x 4.6mm	9230503062	\$315
5µm	150 x 4.6mm	9230505062	\$285
5µm	250 x 4.6mm	9230505060	\$310
Carbamate			
5µm	250 x 4.6mm	9231305060	\$375
Hypersil® Guard Cartridge Holder			
	10 x 4.6mm	25064	\$75

Note: Other phases and column dimensions are available.

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# Sample Cylinders & Valves

## Silcosteel®-Treated Sample Cylinders

- Sizes range from 75cc to 1000cc.
- Made from 304 stainless steel.
- Department of Transportation rated to 1800psi at room temperature.
- Silcosteel®-treated for use with active samples.

Whether you collect hydrocarbons in refineries or reactive compounds in chemical reaction vessels, use Silcosteel®-treated sample cylinders to ensure sample integrity for sulfur compounds and other trace reactive compounds.

Restek's Silcosteel® process deposits a thin, inert layer on the stainless steel, resulting in a very inert surface. Even trace levels of sulfur compounds can be successfully collected and stored in these cylinders without significant loss (Figure 1). Silcosteel®-treated sample cylinders combine the inertness of glass with the strength of stainless steel and are ideal for the analysis of difficult sulfur compounds.

### Product Listing:

#### Silcosteel®-Treated Sample Canisters

Volume	cat.#	price
75cc	24271	\$125
150cc	24272	\$140
300cc	24273	\$150
500cc	24274	\$180
1000cc	24275	\$320

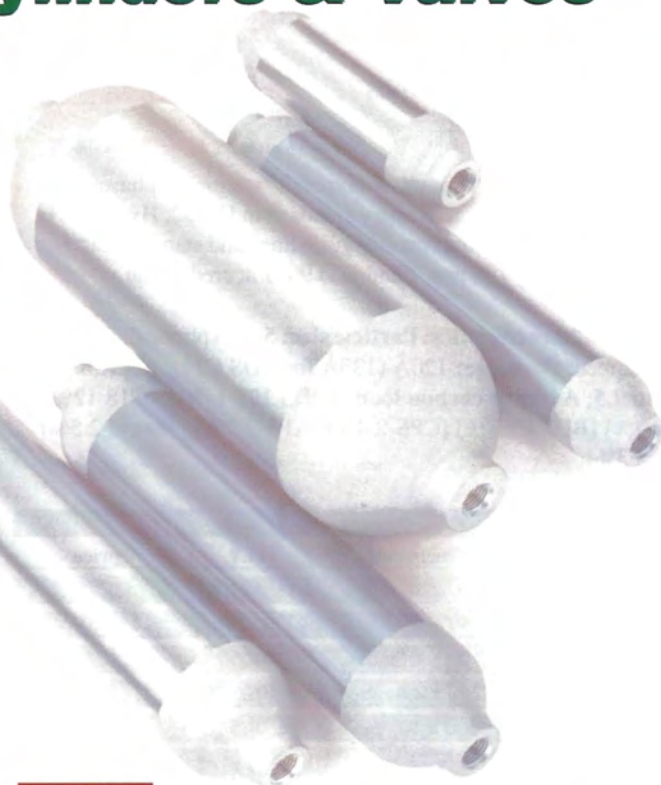
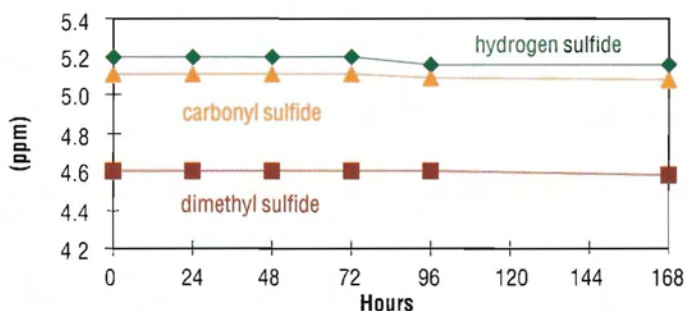


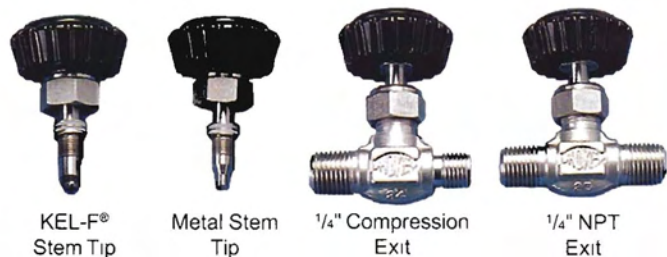
Figure 1:

Storage of sulfur compounds at ~5.0ppm in a Silcosteel®-treated stainless steel cylinder for seven days shows no significant compound loss.



## Silcosteel®-Treated Hoke Sample Cylinder Valves for Maximum Inertness

Hoke valves (to use with the above sample cylinders) are now offered with Silcosteel®-treatment of all parts that come in contact with the sample. The metal stem tip is Hoke model #3712, and the KEL-F® stem tip is Hoke model #3752. Both valves are offered with either a 1/4-inch NPT outlet fitting or a 1/4-inch compression fitting. Maximum operating pressure is 5000 psig. The temperature range for the metal stem tip is -65°F to 450°F, and the KEL-F® stem tip is -20°F to 250°F.



### Product Listing:

#### Silcosteel®-Treated Hoke Sample Cylinder Valves

Description	cat.#	price
KEL-F stem tip: 1/4" male NPT exit	24278	\$130
KEL-F stem tip: 1/4" compression exit	24279	\$130
Metal stem tip: 1/4" male NPT exit	24276	\$120
Metal stem tip: 1/4" compression exit	24277	\$120



# Advances in GC Septa

by Douglas Elliott

## One Chromatographer's Observations—

John Lidgett, QA Specialist, is responsible for using five GCs and one GC-MS (from four different manufacturers) to assure the quality and purity of Restek's reference chemicals. In a typical month he analyzes over 1,000 samples—there can be no margin of error, no inaccurate data or equipment downtime!

When I approached John last Fall and asked him to beta-test the Merlin Microseal™ septa and the new IceBlue™ septa, he was eager to participate. John installed and used a Merlin Microseal™ septum and several IceBlue™ septa in his equipment. His objective was to observe the quality of the chromatography and report on the performance of these septa during routine use.

## Merlin Microseal™ Septa The Long-Life Alternative



After 10 months and thousands of injections on an HP 7673 Autosampler, the Merlin Microseal™ septum still looks and performs like new.

Routine maintenance involved weekly checks with a Restek

Leak Detective™ electronic leak detector (cat. # 21607). An increased carrier gas leak from the septum nut signaled that the septum needed to be examined and cleaned. To do this, John removed, disassembled, and cleaned the septum and nut. Cleaning involved sonicating the two pieces in methanol, air drying, then reassembling them. (The new Merlin 400 series septa have a removable tab that exposes an internal area designed to trap potentially interfering sample media and retain them so that the sealing surface remains clean for a long time.)

John did add one step that Merlin does not recommend; he removed the return support spring in the injection port to allow more solvent to enter the needle path. This is a delicate and potentially risky maneuver but still possible.

John said that he found it hard to "wear-out" the Microseal™ septum if it was maintained and installed properly (remember that it is as important not to over-tighten the septum nut when using a Microseal™ septum as when using another septum).

When it came to chromatographic performance, John noticed no septum bleed and no septum particles shedding into his inlet liner. This second observation led to the realization that he could use a laminar cup inlet liner. Laminar cup liners are recognized for providing excellent vaporization and homogenization of sample vapors. Their disadvantage is that they accumulate septum particles, which is a source of sample adsorption and bleed, and are difficult to clean. With the Microseal™ septum, John was able to use a laminar

cup splitter for better chromatography and not worry about septum particle build-up.

## IceBlue™ Septa They're COOL!



John also installed the new Restek IceBlue™ septa in several injection ports. I figured the Microseal™ septa endorsement would be hard to top, but he found himself even more impressed by the IceBlue™ septa!

The IceBlue™ septa showed no signs of bleed, even when he occasionally set his injection port to 300°C. The manual injections were very smooth and the septum displayed low needle resistance. Resealing after injections was very good up to the time of replacement at 300 injections. John said that the performance was as good as he had seen in any other septa.

The real surprises started when he changed the used IceBlue™ septum for a new one. The septum did not stick to the injection port as he had seen with other septa. Also, there was no oily film under the septum. He had seen this in other septa that had been evaluated. When he removed the inlet liner for cleaning, there were NO septum particles! This meant that he could also use more complex inlet liners, like a laminar cup, for better sample vaporization and mixing. John was surprised that a silicone septum offered the chromatographic performance of a Microseal™ septum.

Restek has long held a good reputation for high-performance, high-temperature Thermolite® and RedLite™ septa. Now Restek is offering you Merlin Microseal™ septa, the finest mechanically engineered septa, and the new IceBlue™ septa, the finest general purpose silicone septa.

For a detailed discussion on septum performance including bleed, bleed impostors, operating variables, and troubleshooting, please request *The Guide to Minimizing Septa Problems* (lit. cat# 59886).

## IceBlue™\* Septa

Septa size	50-Pack cat.#/price	100-Pack cat.#/price
9.5mm	22388 \$35	22389 \$65
10mm	22390 \$35	22391 \$65
11mm	22392 \$35	22393 \$65

## Merlin Microseal® High-Pressure, 400 Series Septa\*

Description	Merlin #	cat.#	price
Nut Kit (1 nut, fits 300 & 400 series septa):	403	22809	\$100
Standard Kit (1 nut, 2 high-pressure septa):	404	22810	\$340
Starter Kit (1 nut, 1 high-pressure septum):	405	22811	\$240
High-Pressure Replacement Septa (1 septum):	410	22812	\$140

\*Only the most popular sizes are listed. For a complete product listing, call to request the Genuine Restek Replacement Parts for HP GCs catalog (lit. cat.# 59627).

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# New Resprep™ SPE Tubes, Disks, and Accessories

## US EPA Drinking Water Methods

We have expanded our solid phase extraction

(SPE) product line to

include many of your

favorite packings,

tubes and disk sizes,

and specialized

products for EPA

Methods!\* Call to ask

for free SPE tube or

disk samples, or to re-

ceive our "Look In-

side" brochure listing

these new SPE prod-

ucts (lit. cat.# 59623).

\* See the applicable method for specific procedural and product requirements.

Method	Analytes	Product description	cat.#	price
525.1	Semi-volatile organic compounds	C18, 6mL, 0.5g	24052	\$60, 30-pk.
		C18, 6mL, 1.0g	24051	\$85, 30-pk.
		C18, 47mm disk	24004	\$95, 20-pk.
508	Organochlorine pesticides	C18, 6mL, 0.5g tube	24052	\$60, 30-pk.
		C18, 6mL, 1.0g tube	24051	\$85, 30-pk.
515	Chlorinated herbicides	C18, 6mL, 0.5g tube	24052	\$60, 30-pk.
515.2		Resin, 47mm disk	26023	\$130, 20-pk.
548.1	Endothal	BioRex® 5, 6mL tube	26063	\$110, 30-pk.
548.2				
549.1	Diquat & paraquat	C8, 6mL, 0.5g tube	26039	\$60, 30-pk.
		C8, 47mm disk	24048	\$95, 24-pk.
550.1	PAHs	C18, 6mL, 1.0g tube	24051	\$85, 30-pk.
		C18, 47mm disk	24004	\$95, 20-pk.
552.1	Haloacetic acids & dalapon	AG 1x8, 1mL tube	26064	\$145, 100-pk.
553	Benzidines & nitrogen pesticides	C18, 6mL, 0.5g tube	24052	\$60, 30-pk.
		C18, 6mL, 1.0g tube	24051	\$85, 30-pk.
554	Carbonyl compounds	C18, 6mL, 0.5g tube	24052	\$60, 30-pk.

## US EPA Solid Waste Methods

Method	Analytes	Product description	cat.#	price
3535	Organochlorine pesticides	C-18, 47mm disk	24004	\$95, 20-pk.
3610B	Nitrosamines	Alumina B, 6mL, 1.0g tubes	55101	\$80, 30-pk.
	Phthalate esters	Alumina N, 6mL, 1.0g tubes	55099	\$80, 30-pk.
3620B	Chlorinated hydrocarbons, Organochlorine pesticides, PCBs, Phthalate esters	Florisil, 6mL, 1.0g tubes	24034	\$70, 30-pk.
3630C	PCBs, Organochlorine pesticides, PFBB derivatized phenols	Silica, 6mL, 1.0g tubes	24038	\$70, 30-pk.

## Special Methods

Method	Analytes	Product description	cat.#	price
CLP	Chlorinated pesticides	Florisil, 6mL, 0.5g	55100	\$80, 30-pk.
Pesticides & Arochlor	Arochlor	Florisil, 6mL, 1.0g	24034	\$70, 30-pk.
1664 update	Oil & grease	Special, 47mm disk	26022	\$75, 20-pk.
		Reprep™ Flowfilter	26024	\$14, 20-pk.
Mass. EPA for EPH/VPH	Hexane extractable hydrocarbons	Silica, 20mL, 5.0g	26065	\$95, 20-pk.

To receive a complete SPE products information

packet, request

lit. cat.# 59664.

## New Resprep™ SPE Manifolds

Restek now offers many versatile SPE manifold models to better suit your processing needs. Our 6D manifold is compact, inexpensive, and enables you to process 6 disk samples simultaneously. Samples are automatically drawn directly from collection

containers or can be transferred into our optional 125mL Diskcover™-47 reservoirs.

For methods that require large collection volumes, our new Maxi-Manifold accepts a wide variety of large volume receiving vessels and can easily be

expanded from one to all six positions as your budget allows or work-load requires.

We also offer both 12- and 24-position manifolds to streamline your SPE tube extraction processes. Ask for help to decide the right model for your needs! Or call for our hardware brochure (lit. cat.# 59621).



Resprep™  
Maxi-Manifold



Resprep™  
12- & 24-Port  
Manifolds

Visit us at EAS to talk about your sample preparation needs!



# Peak Performers

## Genuine Restek Replacement Parts for HP 5890/6890 GCs

- Meets or exceeds original equipment manufacturer specifications.
- Free technical service.
- Fast delivery.

Request our new catalog,  
Genuine Restek Replacement Parts  
for HP GCs (lit. cat.# 59627).



**Replacement FID Jets  
for HP 5890 GCs**

*Standard or Silcosteel®-treated  
high-performance version*

(0.011-inch ID tip)

(Similar to HP part #19244-80560)

**Standard Version:**

cat.# 20670, \$45 ea.

cat.# 20671, \$122/3-pk.

**High Performance:**

cat.# 20672, \$54 ea.

cat.# 20673, \$155/3-pk.



**Replacement FID Jets  
for HP 6890 GCs**

*Standard or Silcosteel®-treated  
high-performance version*

(Similar to HP part #G1531-80560)

**Standard Version:**

cat.# 21621, \$36 ea.

cat.# 21682, \$100/3-pk.

**High Performance:**

cat.# 21620, \$45 ea.

cat.# 21683, \$125/3-pk.



**MSD Source Nut**

cat.# 20643, \$18/2-pk.



**MSD Conversion Fitting**

cat.# 21314, \$40 ea.



**FID/NPD Capillary Adaptor  
for HP 5890/6890 GCs**

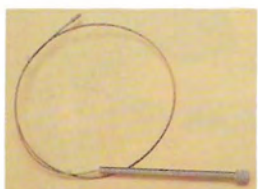
(Similar to HP part #19244-80610)

cat.# 22077, \$52/ea.



**Detector Plug Nut**

cat.# 21883, \$24/2-pk.



**Replacement Chemical Trap  
for HP 5890 GCs**

(Similar to HP part # 05890-61260)

cat. # 21610, \$82 ea.

**New! Replacement Split Trap  
Assembly for HP 6890 GCs**

(Similar to HP part # G1544-80520)

cat. # 22820, \$70 ea.-not shown

**Replacement 24-inch Split  
Vent Line for HP  
5890 GCs**

(Similar to HP part # 5020-8262)

cat. #22800, \$36/2-pk.-not shown



**Silcosteel®-Treated Septum-  
Purged Packed Column  
Port Weldment**

(Similar to HP part # 19243-80570)

cat.# 21691, \$99/ea.



**Low Volume Injector for HP  
Split/Splitless GC Inlets**

cat.# 21692, \$375/kit



**Air Diverter for HP 5890 GCs**

(Similar to HP part # 19247-60510)

cat.# 22076, \$120/ea.



**Stainless Steel Capillary Nuts  
for HP 5890/6890 GCs**

For use with standard ferrules:

cat.# 20883, \$25/2-pk.

For use with "short"

HP-type ferrules:

(Similar to HP part #5181-8830)

cat.# 21884, \$25/2-pk.



**Restek's Leak Detective™**

(110 volts) cat.# 21607, \$595/ea.

(220 volts) cat.# 21609, \$645/ea.



**Reducing Nut for HP  
5890/6890 GCs**

(Similar to HP part # 18740-20800)

cat.# 22078, \$35/ea.

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# A Heart of Gold Lives On...

In September, 1997

Erik D. Steindl, certified personal fitness trainer at Restek, a senior at The Pennsylvania State University (PSU), a Staff Sergeant in the Pennsylvania Air National Guard, and son of Eric Steindl (also an employee of Restek), was diagnosed with a germ-cell cancer. The initial prognosis was excellent.

At the time of diagnosis, the softball-sized tumor had already invaded Erik's left bronchial tube, restricting air and blood flow to his left lung. It also was believed to be dangerously close to his heart. Surgery at this point was not an option. The doctors moved quickly to begin a very aggressive chemotherapy treatment, which they hoped would shrink the size of the tumor and restore blood and air flow to Erik's left lung. Then they would remove the remaining cancerous tissue by surgery. While Erik's family and friends struggled to accept that a strong, otherwise healthy 24-year-old could have developed such a serious medical condition, Erik continued with his studies at PSU and planned for his future.

Initial chemotherapy treatments appeared to have a positive effect, and although chemotherapy would exhaust Erik, he never lost sight of his dreams or forgot his family and friends. He continued to write, phone, and visit people whenever possible. Too sick and tired to

drive himself, his family would take him to visit his friends. Erik would always say "I need to walk in and show people I'm OK so they don't worry so much." At Restek, Erik would walk throughout the entire facility, talking to people about his hopes and plans for the future. To many, he would say "I'm going to beat this cancer and write a book about it."

In October and November, Erik's condition became more complicated. His heart rate would suddenly and dangerously increase, requiring him to be flown by Life Flight helicopter to Hershey Medical Center, PA. He developed severe headaches, and it was discovered that blood clots had formed in several major veins in his brain. While in the hospital, family and friends from across the country came to visit. Each time a person entered Erik's hospital room, Erik would sit up, try to relieve the visitor's worry, and entertain them as if nothing was wrong. Despite his declining health, Erik maintained contact with friends at Restek and PSU.

In December, Erik's younger brother Greg graduated from PSU. Too sick to attend the graduation ceremony, Erik stayed at home with several friends, talking and planning for 1998.

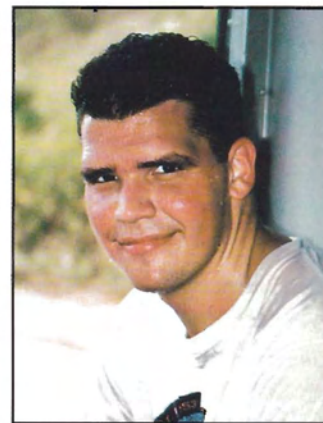
After four chemotherapy treatments and numerous tests, Erik's doctors felt the cancer was finally operable. During

surgery, the doctors found the cancer had spread into the heart tissue itself, part of the left lung, and the chest wall. The night after surgery, Erik suffered a stroke from the blood clots in his brain. Gravely ill and unable to speak because of the ventilator, Erik was still fighting and writing notes to ask if his family and friends were alright. On February 14, Valentine's Day, this young man who touched so many hearts, lost his fight against cancer.

On June 19, what would have been Erik's 25 birthday, a memorial ceremony was held at Restek. Over 100 family members and friends attended from across the country. Erik's parents and brother were presented with several awards for Erik. Restek presented its highest honor posthumously, "The President's Award," to Erik for his courage and strength, which was an example to us all. Also, Restek dedicated an outdoor memorial and renamed the fitness center "Corazon de Oro," meaning "Heart of Gold" in Spanish, a language Erik had studied for many years in the Air Force and at PSU. PSU presented Erik's posthumous diploma, awarded because of Erik's fight to continue with his studies in spite of his illness. The Pennsylvania Air National Guard, 112<sup>th</sup> Air Control Squadron, by order of the President of the United States, awarded Erik the "Air Force Meritorious Service Award." This is the highest award given by the Air Force to

a non-combatant servicemen. The members of the squadron also presented Erik's family with a shadow box containing all the awards, rank insignia, and unit emblems Erik had earned during his military service.

Restek deeply misses Erik and will actively preserve his legacy here and in the community. To ensure Erik's dedication and spirit lives on, a memorial scholarship fund has been established at Penn State University. This scholarship, the "Erik D. Steindl Spanish Scholarship Fund" will be awarded to a student in the Spanish Department who demonstrates outstanding academic excellence and community spirit. If you would like to make a contribution to this fund, please send your tax-deductible donation to: **Erik D. Steindl Spanish Scholarship Fund, The Pennsylvania State University, College of Liberal Arts, 101 Sparks Bldg., University Park, PA 16802.**



**Erik D. Steindl**  
*Deeply missed by all.*



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by David Bliesner

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# Restek Analytical Services<sup>SM</sup>

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"In the modern chromatography laboratory, you want one thing — reliable solutions to your chromatographic problems. Previously, when a method could not be reproduced, questions such as, 'Is it the column or did something change in the sample workup?' could not be answered by one source. By combining product development and method development under one umbrella, you can be assured that your problem will be solved with full accountability.

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David M. Bliesner, Ph.D.  
Director, RAS<sup>SM</sup>







# Chiral Separation of Underivatized Pharmaceuticals Using Capillary GC

by Sherry Sponsler

- **Fast, simple analysis**
- **Thermal stability ensures low column bleed**
- **Reproducible enantioselectivity**

Many pharmaceutical compounds are chiral, where each enantiomer may exhibit different biological activity and toxicological effects. For this reason, manufacture and isolation of a drug as a pure enantiomer may be desired. Although such separations are typically performed on chiral liquid chromatography (LC) columns, many pharmaceuticals can be analyzed by capillary gas chromatography (GC).

GC analysis often requires derivatization of the sample to reduce reactivity, increase volatility, and enhance enantiomeric selectivity. However, ibuprofen and thalidomide can be analyzed by capillary GC, without derivatization. The Rt- $\beta$ DEXsm™ and Rt- $\beta$ DEXcst™ columns provide excellent chiral separation of these underivatized optical isomers within reasonable analysis times.

Ibuprofen is a commonly used pain reliever. Currently it is manufactured in the racemic form. However, researchers determined that R-enantiomer, the enantio-pure form, is much more potent than the racemic mixture. A smaller dosage of the enantio-pure form would be required to provide the same effect or pain relief.<sup>1</sup> The free optical isomers of ibuprofen are quickly resolved on an Rt-

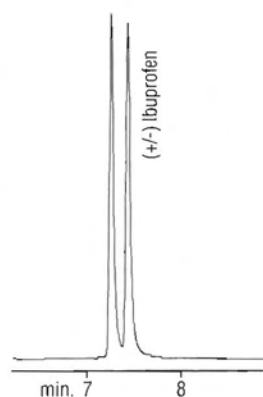
$\beta$ DEXsm™ column in under 10 minutes (Figure 1).

During the 1950's, thalidomide was prescribed to pregnant women for morning sickness, but was banned in 1962 because of its mutagenic effect on fetuses. It was later discovered that the S-enantiomer was the primarily responsible teratogen. However, both enantio-pure forms racemize *in vitro* and *in vivo*.<sup>2</sup> Recently, thalidomide has re-emerged as a therapeutic agent for mouth sores in HIV patients, and for problems associated with skin and autoimmune disorders. Thalidomide is resolved into its enantiomers on the Rt- $\beta$ DEXcst™ column, as shown in Figure 2. The thermal stability of these columns to 230°C allows analysis of larger semi-volatile components, such as thalidomide, without the interference of column bleed.

Chiral separation of many pharmaceutical compounds is possible by capillary GC, and some do not require derivatization. The Rt- $\beta$ DEX™ columns provide unique enantioselectivity and thermal stability for separating other pharmaceutical compounds.

**Figure 1:**

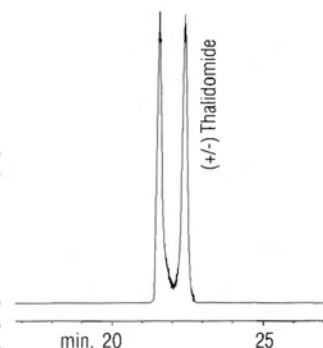
**Ibuprofen Analysis in 10 Minutes on an Rt- $\beta$ DEXsm™ Column.**



30m, 0.32mm ID, 0.25 $\mu$ m Rt- $\beta$ DEXsm (cat.# 13104). On-column injection, concentration approximately 125ng each enantiomer.  
**Oven temp.:** 175°C to 200°C @ 2°C/min.,  
**Inj./det. temp.:** 200°C/230°C;  
**Carrier gas:** helium @ 60cm/sec.,  
**Detector type:** GC-FID;  
**Split ratio:** 13:1 using cup splitter sleeve (cat.# 20709).

**Figure 2:**

**Thermal Stability to 230°C for a Better Thalidomide Separation on an Rt- $\beta$ DEXcst™ Column.**



30m, 0.32mm ID, 0.25 $\mu$ m Rt- $\beta$ DEXcst (cat.# 13102). On-column injection, concentration approximately 15ng each enantiomer.  
**Oven temp.:** 200°C to 230°C @ 1°C/min.,  
**Inj./det. temp.:** 200°C/230°C;  
**Carrier gas:** hydrogen @ 80cm/sec.,  
**Detector type:** GC-FID;  
**Split ratio:** 13:1 using cup splitter sleeve (cat.# 20709).

## References

- <sup>1</sup> Lemko, C.H., C. Gilles, and R. Foster. *Stereospecific High-Performance Liquid Chromatographic Assay of Ibuprofen: Improved Sensitivity and Sample Processing Efficiency*. *J. of Chromat.* 1993; 619 pp 330-335.
- <sup>2</sup> Schmidt, H.J., H. Nav, D. Neubert. *The Anantiomers of the Teratogenic Thalidamide Analogue EM 12:1. Chiral Inversion and Plasma Pharmacokinetics*, *Arch. Toxicol.* 1998; 62 pp. 2-3.

## Product Listing:

**Rt- $\beta$ DEXsm™ 30m, 0.25 $\mu$ m df**

0.25mm ID	0.32mm ID
13105	13104

**Rt- $\beta$ DEXcst™ 30m, 0.25 $\mu$ m df**

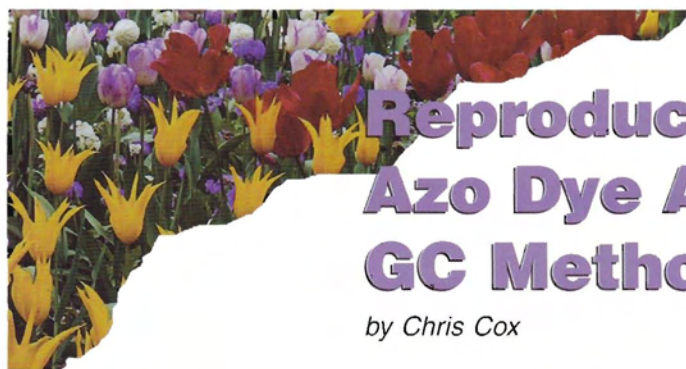
0.25mm ID	0.32mm ID
13103	13102

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INTERNATIONAL





# Reproducible, Accurate Azo Dye Analysis Using New GC Method

by Chris Cox

- **Base-deactivated products improve reproducibility**
- **Pure, stable calibration standards ensure accurate results**

Some consumer goods that come into contact with the human body, such as clothing, jewelry, and toys, are processed using azo dyes, which can metabolize into carcinogenic aryl amines. Because of this, European countries have shown concern over the presence of azo dyes in their manufactured products. Products containing azo dyes that could form into one or more of a list

of amines cannot be distributed in Germany.<sup>1</sup> In 1996, the German Government Institute BgVV<sup>2</sup>, published a method for the analysis of azo dyes.

A few problems have been encountered with this method. Analysts commonly see "priming effects." This is when the response factors rise for the first several injections before they reach an equilibrium state.

- While standard deactivation of columns and sleeves works well in most situations, they typically do not perform well for amine analyses using splitless injections at low concentrations.

Restek has developed an ideal GC method for this azo dye analysis; it has total system inertness, with no priming effects and excellent reproducibility.

An Rtx®-5 Amine column, combined with an amine-deactivated inlet sleeve and wool, provides a significant advantage in the analysis of amines. The priming effect of the standard deactivation versus the amine deactivation was evalu-

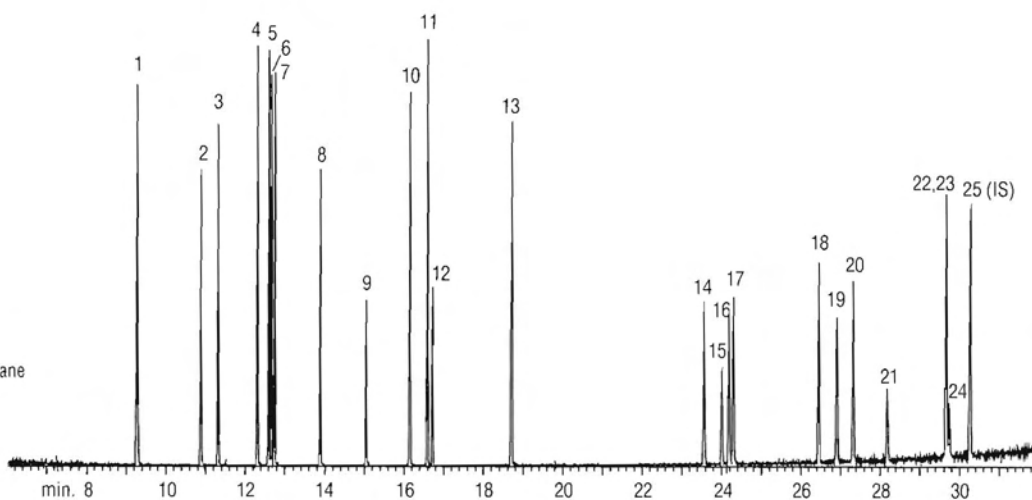
ated by comparing the response factors for each during multiple runs. With the standard deactivation, the response factors for the first injection were as much as 10% lower than the tenth injection. However, with the amine deactivation, there were no priming effects. The elimination of priming effects greatly improves the reproducibility of the analysis.

Another difficulty with the German method is the absence of pure, stable calibration standards. Many of the target compounds often must be tested and re-purified to ensure accurate preparation of the calibration standards. Restek's azo

Figure 1:

Restek's Deactivated System and Azo Dye Standard Ensures Accurate Results.

1. o-toluidine
2. 2-methoxyaniline
3. 4-chloroaniline
4. 2-methoxy-5-methylaniline
5. 2,4,5-trimethylaniline
6. 3-chloro-2-methylaniline
7. 4-chloro-2-methylaniline
8. 2,4-diaminotoluene
9. 2,4-diaminoanisole
10. 2-naphthylamine
11. 2-aminobiphenyl
12. 2-methyl-5-nitroaniline
13. 4-aminobiphenyl
14. 4-phenylazoaniline
15. 4-aminophenylether
16. benzidine
17. bis-(4-aminophenyl)-methane
18. 4'-amino-2,3'-dimethylazobenzene
19. 4,4'-diamino-3,3'-dimethyldiphenyl methane
20. 3,3'-dimethylbenzidine
21. 4-aminophenylthioether
22. 3,3'-dichlorobenzidine
23. 4,4'-methylene-bis(2-chloroaniline)
24. 3,3'-dimethoxybenzidine
25. 3,3',5,5'-tetramethyl benzidine (IS)



30m, 0.25mm ID, 0.25µm Rtx®-5 Amine (cat.# 12323). 1µl splitless injection using a 4mm gooseneck sleeve for HP GCs (cat.# 20798-211.1). 10µg/ml in ethylacetate. Oven temp.: 50°C to 200°C @ 10°C/min. to 270°C @ 5°C/min.; Inj./det. temp.: 250°/300°; Carrier gas: helium @ 1ml/min.; Detector type: MSD; Splitless hold time: 1 min.

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dye standard contains raw materials that are carefully tested and purified to provide the most pure and stable calibration standard available. **Figure 1** shows the analysis of Restek's azo dye standard. We used the procedure as detailed in the German method. By substituting Restek's specially-deacti-

vated column and inlet sleeve, and using Restek's azo dye standard, we achieved reproducible and accurate results. For details on sample preparation, request the *Reproducible, Accurate Azo Dye Analysis Using New GC Method Application Note* (cat.# 59552).

#### References

- <sup>1</sup> German consumers goods act of July 15, 1994, Lebensmittel-und Bedarfsgegenständegesetz, §35 LMBG.
- <sup>2</sup> Bundesinstitut für gesundheitlichen Verbraucherschutz und Veterinärmedizin.

#### Product Listing:

### Azo Dye Precursors and Metabolites Reference Materials

*Carcinogenic Aromatic Amines For European Standard Method*

#### Azo Dye Mix #1

2-aminobiphenyl  
4-aminobiphenyl  
4'-amino-2,3'-dimethylazobenzene (o-aminoazotoluene)  
4-aminophenylether (4,4'-oxydianiline)  
4-aminophenylthioether (4,4'-thiodianiline)  
benzidine  
bis-(4-aminophenyl)-methane (4,4'-diaminodiphenylmethane)  
4-chloroaniline (p-chloroaniline)  
3-chloro-2-methylaniline (3-chloro-o-toluidine)  
4-chloro-2-methylaniline (4-chloro-o-toluidine)  
2,4-diaminotoluene  
3,3'-dichlorobenzidine  
3,3'-dimethoxybenzidine (o-dianisidine)  
3,3'-dimethylbenzidine (o-tolidine)  
2-methoxyaniline (o-anisidine)  
4,4'-diamino-3,3'-dimethyldiphenyl methane  
2-methoxy-5-methylaniline (p-cresidine)  
4-methoxy-1,3-phenylenediamine sulfate hydrate\*\* (2,4-diaminoanisole)  
4,4'-methylene-bis(2-chloroaniline)  
2-methyl-5-nitroaniline (2-amino-4-nitrotoluene)  
2-naphthylamine  
4-phenylazoaniline (p-aminoazobenzene)  
o-toluidine  
2,4,5-trimethylaniline

\*\*concentration reported as free base

10µg/ml each in ethyl acetate, 1ml/ampul

	Each	5-pk.	10-pk.
	31466	31466-510	
w/data pack	31466-500	31466-520	31566

#### Internal Standard for Azo Dye Method

3,3',5,5'-tetramethylbenzidine  
1000µg/ml in ethyl acetate, 1ml/ampul

	Each	5-pk.	10-pk.
	31467	31467-510	
w/data pack	31467-500	31467-520	31567

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#### Rtx®-5 Amine Capillary Column

30m, 0.25mm ID, 0.25µm, cat.# 12323

#### Base-Deactivated Fused Silica Guard Columns

5m, 0.25mm ID 10000 each 10000-600 6-pk.

#### Base-Deactivated Inlet Sleeves Packed with Base-Deactivated Wool

	4mm Gooseneck for HP GCs	4mm Gooseneck for Varian GCs	5mm Gooseneck For CE GCs
each	20798-211.1	20904-211.1	20945-211.1
5-pk.	20799-211.5	20905-211.5	20946-211.5
25-pk.	20800-211.25	20906-211.25	20947-211.25

## Kováts Professor úr 70. születésnapját ünnepeltük a Balaton Szimpóziumon.

*A Hungarian tribute to Dr. Kováts. See page 11 for English version.*

Kováts Professor úr 70. születésnapját ünnepeltük a Balaton Szimpóziumon. Magyarországi disztribútorunk, a Lab-Comp Kft. egyik tulajdonosa, Verebélyi Klára kérésére az 1997. Szeptember 3-5 között Siófokon megtartott Balaton Szimpóziumon előadást tartottam MXT kapilláris kolonnáinkról. Habár a majdnem 20 éves kromatográfiai eladói tevékenységem után e tárgybeli tudásom elég jónak számít, az én ismereteim mégis elhalványulnak a nagy kromatográfus nevek mellett, akik részt vettek a szimpóziumon: Kováts, Nyíredy, Görög, Szepes, Klebovich, Engelhardt, Davankov és Kaiser, illetve akik hozzájuk kapcsolódnak, csak nem tudtak részt venni a Szimpóziumon, mint Horváth és Ettre. Nagy szerencsém volt, hogy együtt dolgozhattam, és sokat tanulhattam Dr. Ettre-től. Kimerítő kanadai szemináriumi körutak során boldogan válaszoltam kérdéseimre, válaszait gyakran cédulákon, éttermi számlákon vagy

repülőtéren szállókártyákon illusztrálva. A szimpózium fénypontja sz. Kováts Ervin Professor úr 70. születésnapjának megünneplése volt. 1927. szeptember 29-én született Budapesten, és itt is nevelkedett. 1945-ben jelentkezett a Műszaki Egyetem Vegyészmérnöki karára, ahol 1949-ben diplomát kapott. A magyar diploma kézhezvétele után Svájcba emigrált ahol 1951-ben vegyészmérnöki diplomát kapott, majd 2 évvel később doktorált a Zürichi Állami Műszaki Egyetemen. Doktori értekezését professor Hans H. Gunthardt vezetése alatt a Szerves Kémiai Laboratóriumban dolgozta ki. Ezt 10 év intenzív munka követte a Nobel díjas Leopold Ruzicka professzorral. A munka nagy részét a vezető Svájci illóolajgyártó cég a Firmenich és Cie finanszírozta.

*Continued on back page...*

*The complete article and translations in German, French, and Italian are located at [www.restekcorp.com](http://www.restekcorp.com).*





# New Rt-U PLOT Columns

## Expanded Selectivity for GC Analysis of Gases and Solvents

by Jingzhen Xu and Andy Schuyler

- A polar, DVB copolymer-based PLOT Column
- A confirmation column for Rt-Q & Rt-S PLOT columns
- Specially designed for gas & solvent analyses

A divinylbenzene (DVB) porous polymer phase has a major advantage over other phases used in PLOT columns—it is less susceptible to the effects of moisture, which can influence column retention characteristics. Restek Rt-S and Rt-Q PLOT columns provide the hydrophobic nature and selectivity of a divinylbenzene porous polymer phase, and the efficiency of a capillary column. Now, Restek has developed a new, more polar column to join the Rt-S and Rt-Q PLOT column family—the Rt-U PLOT column.

The Rt-U PLOT column is manufactured by incorporating ethyleneglycoldimethylacrylate into the polymer chain to provide excellent selectivity for unsaturated compounds. The Rt-Q PLOT column is nonpolar and separates compounds based on volatility. The Rt-S PLOT column provides an intermediate polarity.

As shown in **Figure 1**, these three Restek PLOT columns provide different selectivities for hydrocarbons. On the Rt-U PLOT column, acetylene elutes after ethane, and propylene coelutes with propane. On the Rt-Q PLOT column, acetylene

and ethylene coelute, and propylene elutes before propane. However, on the Rt-S PLOT column, acetylene overlaps with ethane under similar run conditions, and propylene still elutes before propane. For hydrocarbon analyses, the Rt-U PLOT column is the most selective for all unsaturates. The Rt-Q PLOT column is least selective for functional groups. The Rt-S PLOT column is selective for polar compounds having high degrees of unsaturation such as acetylene.

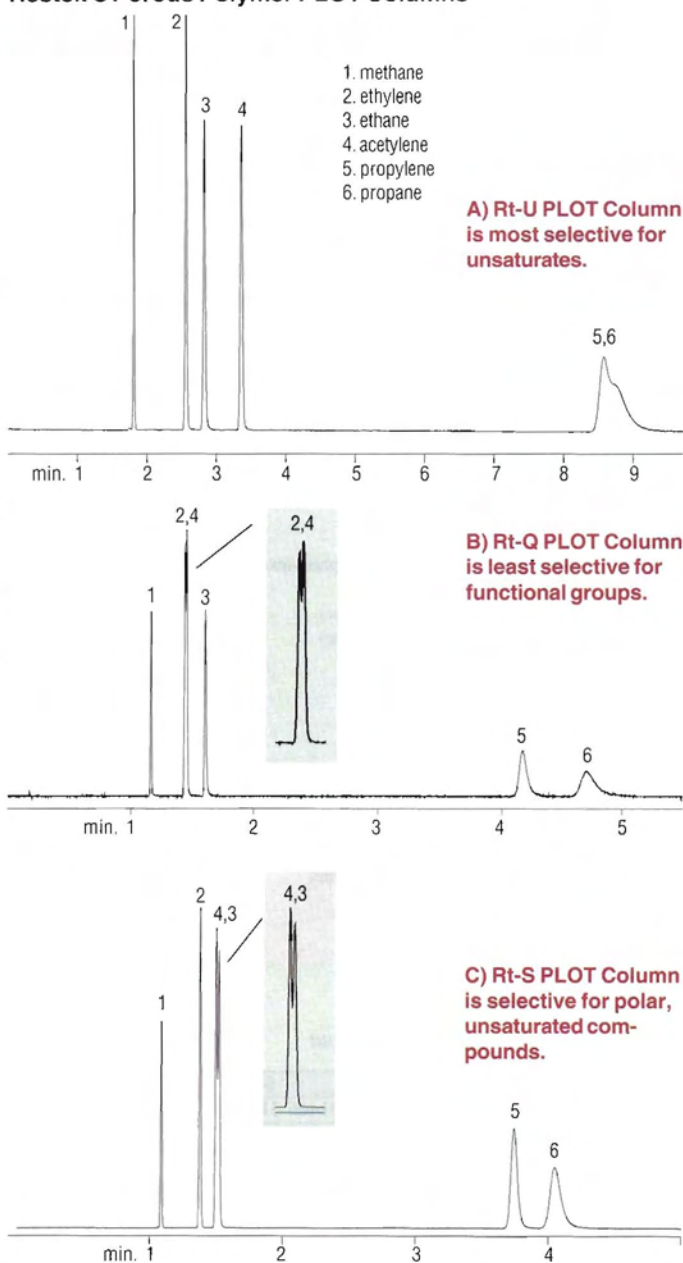
As shown in **Figure 2**, various solvents are separated on the Rt-U and Rt-Q PLOT columns. On the Rt-U PLOT column, polar solvents have much stronger retention. Ethyl acetate elutes after n-hexane, and acetone elutes after n-pentane. On the Rt-Q PLOT column, ethyl acetate elutes before n-hexane, and acetone elutes before n-pentane.

Restek's porous polymer PLOT columns separate a wide variety of chemical functionalities. The line of Rt-Q, Rt-S, and now Rt-U PLOT columns, offers much more versatility for volatile solvent analysis.

Continued top of next page...

**Figure 1:**

Achieve Different Selectivities for C1-C3 Hydrocarbons on Restek's Porous Polymer PLOT Columns



\* 30m, 0.32mm ID, 100µl split injection. 1000ppm (v/v) each in helium with a Cyclosplitter® sleeve (cat.# 20755). Oven temp.: 50°C isothermal; Inj./det. temp.: 200°C; Carrier gas: Hydrogen; Detector: FID; Split ratio: 20/1





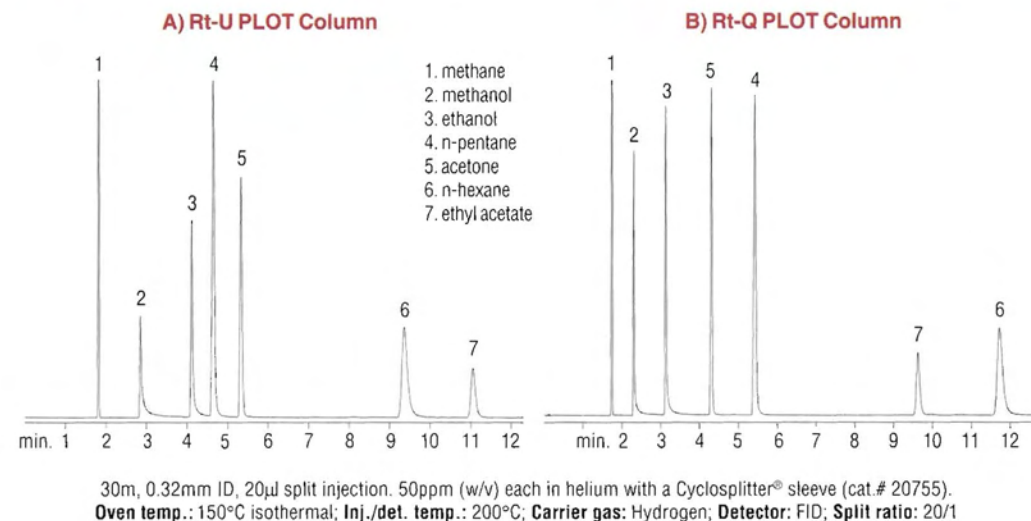
**Figure 2:**

The Rt-U PLOT Column Shows a Stronger Retention for Polar Solvents Compared to the Rt-Q PLOT Column

**Product Listing:**

**Rt-U PLOT Column**

Length	0.32mm ID
15m	19725
30m	19724
Length	0.53mm ID
15m	19727
30m	19726



See Restek's 1998  
Chromatography Products  
Catalog for ordering  
information on other  
PLOT columns.

## New Analytical Reference Materials for GC Analysis of Organo Tins

- Most complete line of organo tin calibration mixes available
- Organo tin analysis technical training offered

### Surrogate Mixtures

2000µg/ml in methylene chloride, 1ml/ampul

#### Tri-n-propyltin Chloride Mixture

	Each	5-pk.	10-pk.
	31476	31476-510	
w/data pack	31476-500	31476-520	31576

#### Triphenyltin Chloride Mixture

	Each	5-pk.	10-pk.
	31477	31477-510	
w/data pack	31477-500	31477-520	31577

### Individual Calibration Compounds for Environmental Analysis and the Electronics Industry

2000µg/ml in methylene chloride, 1ml/ampul

#### Tributyltin Chloride Mixture

	Each	5-pk.	10-pk.
	31478	31478-510	
w/data pack	31478-500	31478-520	31578

### Calibration Mixes for Environmental Analyses

2000µg/ml ea. in methylene chloride, 1ml/ampul

#### Butyltin Chlorides Calibration Mixture

tetrabutyltin, tributyltin chloride, dibutyltin dichloride, butyltin trichloride.

	Each	5-pk.	10-pk.
	31472	31472-510	
w/data pack	31472-500	31472-520	31572

#### Phenyltin Chlorides Calibration Mixture

tetraphenyltin, triphenyltin chloride, diphenyltin dichloride, phenyltin trichloride.

	Each	5-pk.	10-pk.
	31473	31473-510	
w/data pack	31473-500	31473-520	31573

### Internal Standard Mixtures

2000µg/ml in methylene chloride, 1ml/ampul

#### Tetra-n-propyltin Mixture

	Each	5-pk.	10-pk.
	31474	31474-510	
w/data pack	31474-500	31474-520	31574

#### Tetraphenyltin Mixture

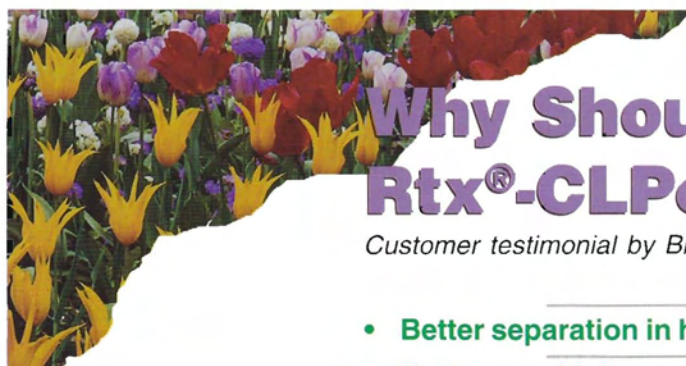
	Each	5-pk.	10-pk.
	31475	31475-510	
w/data pack	31475-500	31475-520	31575

Restek Corporation

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INTERNATIONAL





# Why Should You Use Rtx®-CLPesticides Columns?

Customer testimonial by Bill DesJardins, ITS-Environmental

If you are involved with the analysis of chlorinated pesticides and want to improve your resolution and increase your throughput, just read what Bill DesJardins has to say about Restek's Rtx®-CLPesticides capillary GC columns:

**"The Rtx®-CLPesticides screening column gives me better separation in half the time than a comparable 5% phenyl column. The narrower, taller peaks increased my sensitivity two-fold, and I did not experience any increase in interferences.**

The total run time for a pesticide screen used to be 14 minutes with a 15m Rtx®-5 column. Even with the "long" run time, I had several co-elutions, including gamma BHC and beta BHC. With the Rtx®-CLPesticides 15m column, all 22 pesticides are resolved with a run time of only 5.5 minutes (see **Figure 1**). The better separation is not just for pesticides. The column also produces about 50% more peaks for an Aroclor mixture, allowing better identification of compounds in complex matrices.

**The Rtx®-CLPesticides screening column gives better sensitivity than columns previously used for pesticide and PCB extract screening.** Because of the faster retention times, the peaks on the Rtx®-CLPesticides are sharper and taller. This increases the signal-to-noise ratio, thus

- **Better separation in half the time**
- **Better sensitivity and highly stable**
- **Increased throughput**

increasing sensitivity. For screening, that means I can achieve the required detection limit (i.e. the upper half of the normal analytical calibration range) with a higher extract dilution. This, in turn, produces a cleaner screening system that is able to screen more samples and better withstand big hits from complex samples.

**Retention time stability is rock solid — as it has to be.** With only a few hundredths of a minute between peaks, I cannot have very wide peak ID windows. So far (several

thousand runs), I have not had peaks drift outside the ID window using the Rtx®-CLPesticides screening column, unless I have done maintenance.

**The high thermal stability helps to maintain analytical integrity.**

The accumulation of high-boiling compounds can be a bother during normal analysis but, since the thermal stability (TMax) of the Rtx®-CLPesticides column is 330°C, they can be quickly baked-out with a slight increase in run time.

An interesting dilemma: Because I can make many more injections per day on an Rtx®-CLPesticides screening column, I have found that a 100-place autosampler doesn't have enough capacity to hold 14 hours worth of runs. Now my real time-limitation is the size of my autosampler!"

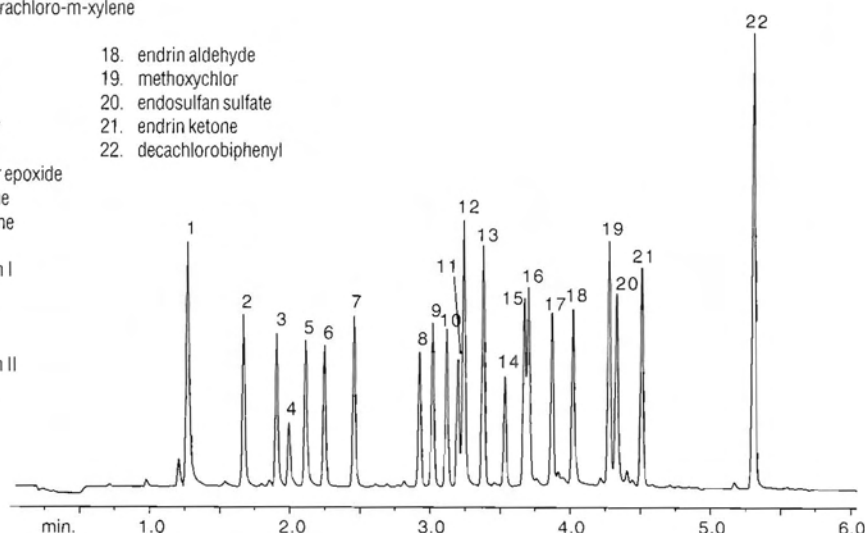
Bill DesJardins, Chemist  
ITS - Environmental  
55 South Park Drive  
Colchester, VT 05446

**For more information on these columns designed specially for chlorinated pesticide analyses, call our customer service group and ask for the Rtx®-CLPesticides brochure (cat.# 59699).**

**Figure 1:**

**Twenty-two Chlorinated Pesticides Calibration Standard Acquired After the Analysis of 3000 Sample Extracts.**

- |                                 |                        |
|---------------------------------|------------------------|
| 1. 2,4,5,6-tetrachloro-m-xylene | 18. endrin aldehyde    |
| 2. α-BHC                        | 19. methoxychlor       |
| 3. γ-BHC                        | 20. endosulfan sulfate |
| 4. β-BHC                        | 21. endrin ketone      |
| 5. δ-BHC                        | 22. decachlorobiphenyl |
| 6. heptachlor                   |                        |
| 7. aldrin                       |                        |
| 8. heptachlor epoxide           |                        |
| 9. γ-chlordane                  |                        |
| 10. α-chlordane                 |                        |
| 11. 4,4'-DDE                    |                        |
| 12. endosulfan I                |                        |
| 13. dieldrin                    |                        |
| 14. endrin                      |                        |
| 15. 4,4'-DDD                    |                        |
| 16. endosulfan II               |                        |
| 17. 4,4'-DDT                    |                        |



15m, 0.25mm ID, 0.25µm Rtx®-CLPesticides column (cat.# 11120). 2µl splitless injection. **Concentration:** 40 to 80ppb. **Oven temp.:** 125°C to 290°C @ 29°C/min. (hold 3 min.); **Inj./det. temp.:** 175°C/300°C; **Inj. port:** Restek split/splitless; **Carrier gas:** hydrogen @ 20psi constant pressure.

Restek Corporation

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INTERNATIONAL



# Silcosteel®-Treated GC Injection Ports Prevent Adsorption

by Gary Barone

- Reduces sample breakdown
- Proven inert coating technique

A common problem with splitless, direct, and flash injections in gas chromatography (GC) is containment of a portion of the sample within the volume of the injection port liner. During these injection techniques, large volumes of vapor are formed that exceed the volume of the inlet liner — this is called backflash. Once outside the inlet liner, the solvent and sample vapor may contact the metal surface of the injection port body, causing adsorption or breakdown of the

sample. Restek can coat your injection port body with an inert Silcosteel® treatment that will reduce adsorption or breakdown when backflash occurs.

Restek has been applying Silcosteel® since 1987 to passivate transfer lines and components used in analytical systems. The Silcosteel® process permanently bonds a thin, flexible, and inert fused silica coating to the surface of steel and stainless steel components.

This coating acts as a rugged barrier between active components and the adsorptive surface.

Application of the Silcosteel® coating to injection port systems has been used extensively in the process industry and is now available for benchtop instruments. If you have experienced decomposition or adsorption that is not attributable to your column, liner, or detector, try Silcosteel® treatment for your injection port — it may be the answer.

To have your injection port Silcosteel®-coated, simply con-

tact one of our customer service representatives. A catalog number and a Return Authorization (RA) number will be assigned, allowing you to return the injection port to Restek. Send us only the injection port body, without liners or heating block attached. The typical turnaround time is 10 working days from receipt of the port.

**Call today for your free brochure on the Silcosteel process (cat.# 59654).**

## Ultra-Clean XAD®-2 Resin

Amberlite XAD®-2 resin is an excellent adsorbent for trapping PAHs, although it requires extensive clean-up prior to use due to impurities introduced during manufacturing. The TO-13 method specifies a clean-up procedure to prepare the resin for use. However, to meet the minimum contamination level, the XAD®-2 resin may require several cleanings. Many laboratories use their own clean-up procedure, taking a week or more to effectively meet TO-13 method requirements.

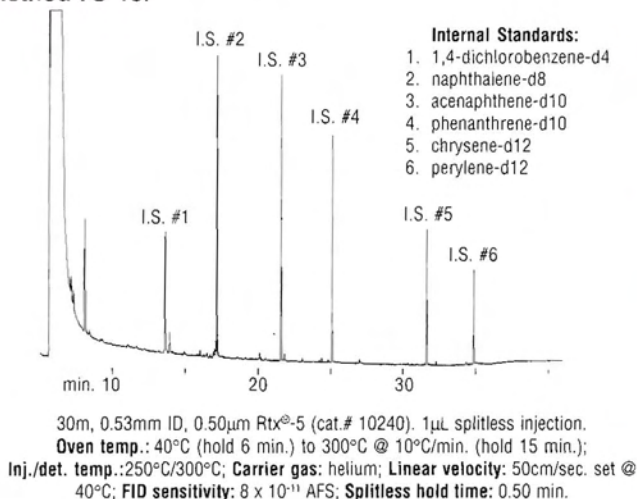
To help laboratories eliminate time-consuming clean-up and still meet TO-13 method requirements, Restek developed Ultra-Clean XAD®-2 resin for semi-volatile air sampling. The clean-up procedure meets the

specified minimum contamination levels and each batch is tested by capillary GC using a flame ionization detector (FID)

to ensure cleanliness. **Figure 1** shows an analysis of a cleaned batch of Amberlite XAD®-2 resin.

**Figure 1:**

**Ultra-Clean XAD®-2 resin meets the requirements of EPA method TO-13.**



### Product Listing:

**Ultra-Clean XAD®-2 Resin**

100 grams: cat.# 24230

500 grams: cat.# 24231

Restek Corporation

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INTERNATIONAL





# Rtx®-624 for Analysis of Rum and Scotch

by Sherry Sponsler

The maturity and quality of rum and scotch can be determined by the inspection of primary alcohols, acids, ketones, trace esters and fusel oils. The Rtx®-624 is a capillary column that provides excellent resolution and quantitation for these important volatile compounds that characterize aroma and flavor and determine proof value.

Some analysts have traditionally used packed columns. CarboBlack™ packed columns, for example, offer better sample capacity and accommodate larger volume injections. They also provide consistent and adequate separation of isoamyl alcohol and active amyl alcohol (also

known as 3-methyl-1-butanol and 2-methyl-1-butanol), two compounds that influence aroma strength of rum and scotch.

Capillary columns with Carbowax® phases have been used to provide better resolution of the volatile alcohols, acids and esters in rum and scotch, but there are three problems. One problem is consistent baseline resolution between isoamyl alcohol and active amyl alcohol. These compounds are barely resolved on PEG columns, and analysts have complained that this separation is not always consistent nor always achieved from column-to-column. The second problem is that isopropanol and ethanol coelute on Stabilwax® and Rtx®-Wax columns. This affects the

quantitation of ethanol and inhibits the detection of denaturants such as isopropanol and methanol. Third, a thin film is required to achieve the separation of active amyl and isoamyl alcohols, but methanol exhibits a strange asymmetrical peak shape that is only corrected by a thicker-filmed 1.0µm Carbowax® column. So sample capacity is very limited.

The 60m x 0.25mm ID x 1.4mm Rtx®-624 column does not have these problems. Isopropanol does not coelute with ethanol, methanol exhibits symmetrical peak shape, and the separation is much more than just baseline resolution for the active amyl and isoamyl alcohols. Note that these compounds exhibit a reversed elution order than on a CarboBlack™ packed column

or on a Stabilwax® capillary column. This is an excellent column for the analysis of rum (Figure 1). This column also provides a good response to acetic acid, which is quantitated to determine the stage of fermentation or maturity of scotch. (Figure 2).

The Rtx®-624 contains a stationary phase that is 6% cyanopropylphenyl methyl-94% dimethylpolysiloxane. The selectivity of the phase provides excellent resolution and quantitation of the flavor volatiles in rum and scotch, and should be the capillary column of choice for GC.

## Product Listing:

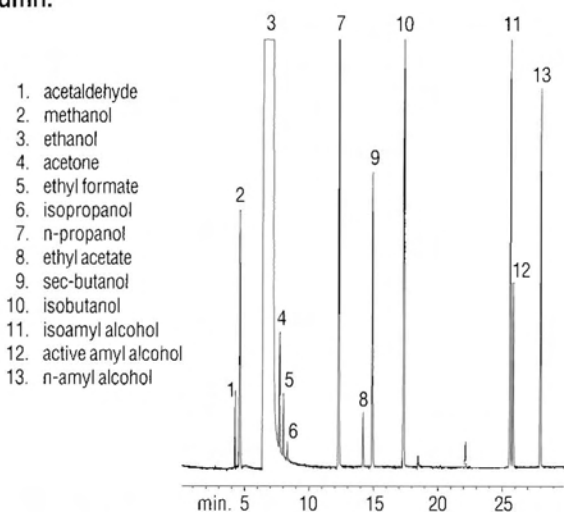
### Rtx®-624 Column

60m, 0.25mm ID, 1.4µm

cat.# 10969

Figure 1:

Baseline resolution of rum components on an Rtx®-624 column.

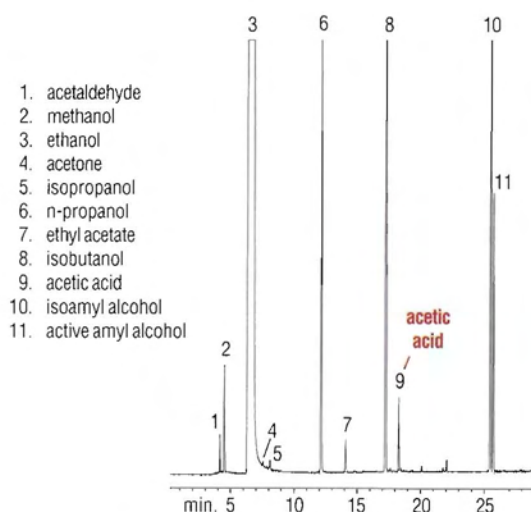


60m, 0.25mm ID, 1.4µm Rtx-624 (cat.# 10969).

1.0µl split injection using a Cyclosplitter® sleeve (cat.# 20706). Concentration: neat; Oven temp.: 35°C (hold 5 min.) to 100°C @ 1°C/min.; Inj./det. temp.: 150°C/200°C; Carrier gas: hydrogen @ 40cm/sec.; Split ratio: 100:1

Figure 2:

Improved analysis of acetic acid on an Rtx®-624 column.



60m, 0.25mm ID, 1.4µm Rtx-624 (cat.# 10969).

1.0µl split injection using a Cyclosplitter® sleeve (cat.# 20706). Concentration: neat; Oven temp.: 35°C (hold 5 min.) to 100°C @ 1°C/min.; Inj./det. temp.: 150°C/200°C; Carrier gas: hydrogen @ 40cm/sec.; Split ratio: 100:1



# Professor Kováts Celebrates 70th Birthday at Balaton Symposium

by Dr. David Mannus

I had the pleasure of presenting a paper on our MXT® capillary columns at the Balaton Symposium in Siófok, Hungary during September 3-5, 1997 at the invitation of Klára Verébelyi, owner of LAB-COMP, our distributor in Hungary. After almost twenty years in chromatography sales, my knowledge of the subject would be described as fairly good. However, my knowledge pales into insignificance alongside the great names in chromatography who attended the symposium: Kováts, Nyiredy, Görög, Szepesy, Klebovich, Engelhardt, Davankov and Kaiser as well as those who are associated, but were unable to attend: Horváth and Ettre. It has been my good fortune to work with, and learn a great deal from, Dr. Ettre. During hectic seminar tours of Canada, he was happy to answer all of my questions often illustrating his replies on scraps of paper such as restaurant receipts or airline boarding cards.

The highlight of the symposium was the celebration of the 70th birthday of Professor Ervin sz. Kováts. He was born on September 29, 1927, in Budapest, Hungary, where he grew up. In 1945 he entered the Technical University to study Chemical Engineering. After receiving his Diploma in 1949 he emigrated to Switzerland and, in 1951, received a Chemical Engineering Diploma and two years later his Doctorate from the Federal Technical

University in Zurich. He worked on his Doctoral thesis in the Laboratory of Organic Chemistry under the guidance of Professor Hans H. Gunthardt. This was followed by ten years of intensive work with the Nobel Laureate Professor Leopold Ruzicka on a major project financed by Firmenich & Cie., the leading Swiss manufacturer of essential oils. In 1968 Dr. Kováts was appointed a full professor at the Ecole Polytechnique Fédérale (EPFL) in Lausanne where one year later he became head of the Laboratoire de Chemie Technique. He retired from the EPFL in 1994, after twenty-six years, when he became a research professor at the University of Veszprém, in Hungary, where he continues his work today.

Professor Kováts is best known for the Kováts Retention Index System for substance characterisation in gas chromatography, which he developed during his research into the systematic study of essential oils. Dr. Kováts says "I realized that the logarithmic distance of the peak maxima of the homologues gives marks on the chromatogram similar to those on tape measures used by the tailors. In re-evaluating all my data, the simplest homologous series, that of n-paraffins, was chosen as a base. The fixed points, the peak maxima, were defined as 100 times the carbon number: 500 at the peak of n-pentane, 600 at that of n-hexane, etc. The retention indices of other compounds were found by

linear interpolation." It must be remembered that this work was carried out in 1958. Dr. Kováts and his team made their own instrument; the world's first automated preparative gas chromatograph. In the next decade Rohrschneider and also McReynolds used the retention index system for their characterisation of the polarity of stationary phases used in gas chromatography. This characterisation system is still universally used today by Restek and other phase and column manufacturers.

At the gala evening as I enjoyed my coffee and my slice of Dr. Kováts' birthday cake, it occurred to me that, if it

had not been for Dr. Kováts, our president, Paul Silvis, may not have founded Restek Corporation, MXT® columns may never have been invented, and I would not be here enjoying the beauty and ambience of Balaton nor traveling to the many other symposia and trade expositions in Europe. Thank you, Dr. Kováts, from everyone at Restek Corporation.

Dr. Dave Mannus  
European Sales Manager

Visit [www.restekcorp.com](http://www.restekcorp.com)  
for translated versions of  
this tribute to Dr. Kováts.



Dr. David Mannus (right), and Klára Verébelyi (left), are proud to honor Dr. Kováts (center) at the Balaton Symposium this past fall.

Restek Corporation

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INTERNATIONAL





## Koni's Korner

### Working Safely with Hydrogen as a Carrier Gas

by Dr. Konrad Grob

**Broad agreement is  
that hydrogen is  
the best carrier gas  
for capillary GC  
applications.**

Nitrogen cannot seriously be considered as a carrier gas option, because diffusion speeds of the solute molecules are roughly four times lower than in hydrogen or helium, rendering the separation process exceedingly slow. Helium is the best alternative if hydrogen cannot be used, but hydrogen enables faster chromatography whenever inlet pressure exceeds roughly 0.7 bar, with a rapidly expanding difference when the required inlet pressure increases. Hydrogen is almost a must for high-temperature work such as triglyceride analysis, and analysis with long columns such as fatty acid methyl ester analysis on 100m columns. Also, hydrogen is available in unlimited amounts (using helium depletes limited natural resources). Hydrogen cannot be used with thermoionic detectors and some mass spectrometers, but the main argument against hydrogen concerns safety because it forms an explosive mixture with air. Can a lab manager take the responsibility for using hydrogen as carrier gas? Yes, if some simple safety measures are taken.

#### Exploding GC ovens

In the past, there have been many hydrogen explosions in GC ovens. I know of four in Switzerland, the latest one probably being 15 years back. I caused one myself in the seventies during production of glass capillary columns. I prepared about five columns a day using a procedure requiring five heat treatments in a GC oven. I could install a column in hardly more than a second, virtually without turning a screw. I set a high inlet pressure to remove the air and heated the GC at full power to 280°C. One day, a column had enough tension to pull the inlet out of the ferrule. Hydrogen ran into the oven at about 1 bar, without any restriction. The heating filaments were red-hot (2.5 kW, in an old instrument) and ignited the mixture. The explosion was heard through several labs. The instrument seemed to jump 1-2cm from the bench and lost the dust from its top. Opening the door, I was amazed to see the glass capillary column hanging a bit lower than before, but not being broken. The door did not open as smoothly as before, this being the only reminder of the event.

The other explosion in our lab occurred shortly after an on-column injection for triglyceride analysis. We changed the columns almost daily, and the inlet was installed rapidly, with a soft fitting used many times, tightening by fingers. As the syringe needle did not enter the column inlet easily, my coworker pushed a bit harder than usual. The needle went down and he did the injection.

He started the program (with ballistic heating from the injection temperature of 70°C to 250°C) and switched on the recorder. He had just turned his back to the instrument when there was the bang. In fact, he injected into the oven, because the syringe had pushed the column out of the fitting. Again, there was no damage, neither to the instrument, nor to the glass capillary column.

The two other explosions happened in other laboratories, in both instances because it remained unnoticed that the column had been removed from the instrument. Somebody wanted to bake out the column, set high inlet pressure and heated the oven at full power. There was no need for coffee to wake up that morning. The instruments were slightly deformed, but were used for another ten years at least.

#### 4% Hydrogen, 630°C

We had many large leaks with broken columns, poor connections, and experiments more outrageous than can be described here - and there were no explosions. Beside some good luck, this is due to the fact that explosions of hydrogen are much less likely to occur than explosions of solvent vapors. A minimum of 4% hydrogen in air is required for an explosion to occur, and the mixture must be heated to about 630°C to be ignited (in contrast to around 0.1% and hardly over 200°C for vapors of many solvents). In fact, all the explosions I know of occurred with old instruments, characterized by high heating power and

relatively small filaments, which turned red-hot when heating ballistically. Newer instruments with less heating power and larger filaments hardly reach the temperature for ignition. Also, the 4% concentration is not that easily reached: concluding from the experience of many critical situations, it seems that the two most common risks in practice, breakage of the column or a leaking connection to an injector, hardly ever result in an explosion. All explosions I heard of were the result of unhindered flow of hydrogen from the injector into the oven.

#### MEASURES ENSURING SAFETY

##### Hydrogen sensors

Risks must be taken seriously, even when it seems unlikely that severe damage may occur. Since the early eighties we gradually equipped all of our instruments with hydrogen sensors, a small device available from various sources. Some air is picked from the zone of the oven ventilator and brought to a sensor detecting hydrogen in the concentration range of 0.1 to 1%. When 1% is reached, the gas chromatograph is switched to cooling, which stops the heating and purges the oven with ambient air. Usually a lamp blinks and an alarm signal calls for attention. A luxury version even replaces the hydrogen in the carrier gas line with nitrogen. The sensor also goes off if concentrations of solvent vapors in the laboratory are high, which eliminates a further risk (independently of whether hydrogen or helium is used as carrier gas).





### Flow-regulated carrier gas supply

Classical gas chromatographs regulate the carrier gas by pressure. When there is no column, such systems may deliver many liters of gas per minute into the oven. Hewlett Packard introduced a flow-regulated system, in the worst case limiting the flow into the oven to the rate adjusted for the application, i.e. column plus split plus septum purge flow rate. The maximum flow rate is 500ml/min. Flow regulation strongly reduces risks, but is not considered to be safe. A rough estimation provides some clues. A GC oven has an internal volume of around 40 liters. The explosion limit of 4% is reached when the oven contains 1.6-liter of hydrogen. If the system leaks by 500ml/min, it takes little more than 3 minutes to render the oven atmosphere explosive. If the imposed flow rate is 100ml/min only, however, the critical limit is calculated to be reached in 16 minutes. This assumes a tight oven, which is not realistic; hydrogen diffuses extremely rapidly and the oven atmosphere is exchanged in far less time than 16 minutes. Hence, with a leak of 100ml/min, the hydrogen concentration in the oven cannot reach the explosion limit. Potentially dangerous flow rates exceeding 100ml/min are of interest for split injection with high split ratios. If the high flow rate is turned down 1 minute after the injection, this eliminates this risk and also conserves carrier gas. In splitless injection, the adjusted flow rate will be substantially below 100ml/min anyway. In on-column injection, the flow rate during analysis is far below the critical minimum. It depends on the construction of the injector seal, however,

whether the flow rate can be limited to a few milliliters per minute: during injection, a rotating valve leaks at a far higher flow rate, and pressure would collapse when limiting the flow rate to less than about 100ml/min. Hence, flow regulation or a restrictor limiting flow in a pressure-regulated system can exclude a dangerous hydrogen concentration in the oven.

### Intermediate storage in small cylinders

Rather frequently a column is dismantled without switching off the carrier gas supply. Other times a septum leaks to such an extent that the hot carrier gas can burn fingers held many centimeters above the septum. In both these situations, hydrogen may leak into the laboratory at high flow rates. The risk of a laboratory explosion is minimal, however. A small laboratory contains maybe 50,000 liters of air. 4% of hydrogen corresponds to 2000 liters, i.e., to the content of a full 10-liter cylinder at 200 bar pressure. Admittedly, mostly 25- or 50-liter cylinders are used,

but since the laboratory atmosphere is exchanged many times per hour, these cylinders would have to be emptied in minutes to reach the explosion limit. Again, hydrogen is far less dangerous than solvents, for many of which 50-liters of vapor is the limit, corresponding to a spill of around 250ml of liquid solvent. And, since the solvent vapors tend to form a "lake" above the floor, the explosion limit is reached locally even more rapidly. A simple safety measure rules out large scale loss of hydrogen and is also a warning of massive leaks: The main reservoir, maybe a 50-liter cylinder, is not directly connected to the laboratory gas supply, but via a small daily reservoir. The large tank is closed (except when almost empty). The gas is consumed from a 3- to 10-liter cylinder, adjusted to the daily consumption. A 3-liter cylinder filled up to 50 bar contains 150 liters of hydrogen. If, for instance, three gas chromatographs consume 100ml/min each (25ml/min for the FID), the cylinder must be refilled every 25 hours, which

suits when these instruments work day and night. If filled to 20 bar only, it would contain an amount well fitting the consumption of a working day. A manometer or an electronic readout is positioned such that everybody will see it. If the daily reservoir must be refilled on the same day, this is a warning and a leak will be detected long before the large reservoir is emptied. The manometer can be equipped with an alarm indicating low pressure. In Switzerland, this is a standard installation found in many laboratories for more than 20 years.

### Hydrogen generators

Hydrogen generators are an alternative to the small daily reservoir. They deliver the gas at limited flow rates and totally avoid the necessity of storing gas. If split injection must be possible at high split flow rate, however, rather large generators and/or one for every few instruments are needed.

### Conclusion

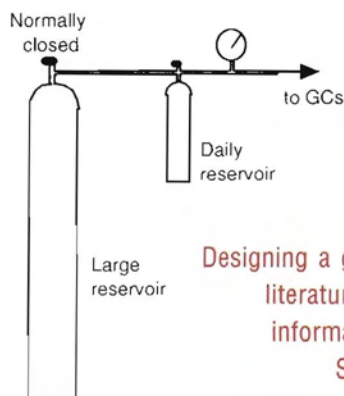
I understand the dilemma of the laboratory manager: he or she may recognize that hydrogen is preferable and cheaper than helium, but does not want to take risks. Can these risks be managed? Hydrogen sensors rule out explosions in the oven, and daily reservoirs or hydrogen generators eliminate risks in the laboratory (as well as costs caused by large losses). Checking tightness of the gas plumbing every 6-12 months is also advisable whether hydrogen or helium is used.

**Dr. Konrad Grob can be reached by E-Mail at [Koni@grob.org](mailto:Koni@grob.org)**

Figure 1:

### Gas supply system ruling out large losses:

Gas is consumed from a daily reservoir and the well-observable manometer provides control over the gas consumption.



Designing a gas system? Request literature packet #59625 for information on Purus™ Gas Systems from Restek.





# Genuine Restek Replacement Parts

*Making GCs Work Better*

**“Restek was founded on supplying superior quality, low-cost manufactured parts and accessories for GCs. Restek offers a complete line of GC operating supplies and maintenance parts — from our design innovations to our original manufacturer replacement parts. At Restek, we do whatever it takes to provide you with the best products.”**

*The GC Accessories Team*



## Innovation

This is more than making new columns or superior chemical standards. This also means helping you optimize your entire GC system, from inlet to detector. Restek has designed and manufactured GC accessories for 13 years, and we improve upon, or innovate, the existing products. For example, we offer deactivated injection ports to minimize sample breakdown. Whether it's to make your job easier, to improve your chromatography, or to save you time and money, we strive to be innovative.

## Quality

Every product we manufacture is carefully evaluated and tested to ensure high quality. We actually use the products we make in our applications and QA labs — before you use it, we use it. However, we think quality goes beyond in-house testing. A lot tracking system lets us trace products to the end user so we know from where your individual product originated. And, we have a customer response team dedicated to ensuring your satisfaction after the sale.

## Technical Service

We have chemists on call to answer your chromatography questions. Before joining Restek, our experienced

scientists have used the products and worked in outside labs. Real life answers for your real-world problems.

## Price

We offer competitive pricing on all GC accessories. Compare our prices to what you currently pay and you'll see we offer the best value.

## Delivery

The final element in meeting your needs is fast delivery. More than 97% of our products are shipped the same day the order is placed. We pride ourselves on getting the job done right and on time. Need a part in an emergency? We can ship via overnight delivery for emergency orders placed as late as 5:30pm EST.

**If you have a specific accessory design requirement, please contact your local distributor.**

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INTERNATIONAL

		<b>Australian Distributors</b> Importers & Manufacturers <a href="http://www.chromtech.net.au">www.chromtech.net.au</a>
<b>ECHnology</b> Pty Ltd Website NEW : <a href="http://www.chromalytic.net.au">www.chromalytic.net.au</a> E-mail : <a href="mailto:info@chromtech.net.au">info@chromtech.net.au</a> Tel: 03 9762 2034 ... in AUSTRALIA		+61(0)3 9762 2034



# Peak Performers

## Replacement Reducing Nut for the HP 5890/6890 GC



Restek now offers the replacement reducing nut for the HP 5890/6890 GCs. Made from high quality stainless steel and meets original equipment specifications.

Genuine Restek Replacement for HP Part No. 18740-20800. Cat.# 22078, ea.

## Replacement FID/NPD Capillary Adaptor for the HP 5890/6890 GC



Restek now offers the FID/NPD replacement capillary adaptor for the HP 5890/6890 GCs. Made from high quality stainless steel and meets original equipment specifications.

Genuine Restek Replacement for HP Part No. 19244-80610. Cat.# 22077, ea.

**Restek Trademarks:** Rtx, MXT, Stabilwax, Rt- $\beta$ DEXsm, Rt- $\beta$ DEXcst, Rt- $\gamma$ DEXsa, Silcosteel, SilcoCan, CarboBlack, Pinnacle, RAS, Cyclosplitter, and the Restek logo.

**Other trademarks:** Hypersil (Hypersil), Inertsil (GL Sciences), Kromasil (Eka Nobel), Nucleosil (Macherey-Nagel), and Carbowax (Union Carbide Corp.), XAD (Rohm & Haas, Co.).

## Capillary FID Jets for the HP 6890 GC



Restek now offers two jets specifically designed for the HP 6890 GC. The Standard version made from high quality stainless steel and the High Performance version which is Silcosteel®-treated for optimum inertness. Both are designed with a fluted tip to guide the capillary column into the jet.

Genuine Restek Replacement for HP Part No. G1531-80560.

### Standard:

Cat.# 21621, ea.  
Cat.# 21682, 3-pack

### High Performance:

Cat.# 21620, ea.  
Cat.# 21683, 3-pack

## Air Diverters for the HP 5890 GC



*Direct the GC exhaust heat away from the lab!*

These Air Diverters are made from high-quality aluminum and meet OEM specifications. Installation is easy.

Genuine Restek Replacement for HP Part No. 19247-60510. Cat. #22076, ea.

## Updated Toxic Organic (TO) Methods Available on CD-ROM

Restek now offers an easy-to-use, updated version of the *Compendium of Methods for the Determination of Toxic Organic Compounds in Air*\* on CD-ROM. This version includes the new second edition methods: TO-4a, TO-9a, TO-10a, TO-11a, TO-13a, TO-14a, TO-15, TO-16 and TO-17, and the non-edited first edition methods: TO-1, TO-2, TO-3, TO-5, TO-6, TO-7, TO-8 and TO-12.

A built-in search feature easily tracks down specific information. The key words entered will be searched across the entire compendium and all

- Contains methods TO-1 through TO-17
- Includes the new second edition methods and the non-edited first edition methods
- Includes Adobe® Acrobat Reader® software

individual documents containing the term will appear in a list.

Two tables are also included to help find the appropriate method for your application: Brief Method Description and Applicability, and Method Applicability to Compounds of Primary Interest.

The compendium CD-ROM is easy to use. The entire collection is built around a

familiar and intuitive "homepage" model, and navigating the exhaustive list of TO-Methods is as easy as pointing and clicking. Visit our web site at <http://www.restekcorp.com> for a demonstration, or call us and try it yourself!

\* These methods were written by the U.S. Environmental Protection Agency's (EPA) National Risk Management Research Laboratory (NRMRL). The 2nd edition was released in the fall of 1997.



### Product Listing:

**Compendium of Methods for the Determination of Toxic Organic Compounds in Air, on CD-ROM**

cat.# 24280

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# RESTEK

## The Advantage

Innovators of  
High Resolution  
Chromatography  
Products

### Rtx<sup>®</sup>-CLPesticides & Rtx<sup>®</sup>-CLPesticides2

*A complete solution for chlorinated  
pesticide, herbicide, and PCB analysis.*

by Frank Dorman

For years, environmental laboratories have struggled with various chlorinated pesticide analytical methods. Keeping track of resolution requirements and breakdown performance criteria while analyzing extracts containing high-boiling contaminants is not easy. With the introduction of the Rtx-CLPesticides and Rtx-CLPesticides2 columns from Restek, it is now possible to analyze the 22 common chlorinated pesticides with baseline resolution on both columns (see **Figures 1 and 2**). In addition, the analysis time is less than 24 minutes on both columns, resulting in higher throughput of samples. Since the columns exhibit baseline resolution, it is possible to combine the calibration standards (Mix A and B) for most methods, again resulting in increased throughput by decreasing the number of injections for calibration runs.

Stationary phase thermal stability and low bleed are also important column features so that sample contamination can

- **Baseline resolution of the 22 chlorinated pesticides in Methods 8081, CLP, and 608.**
- **Less than 25 minute analysis time.**
- **High thermal stability with low bleed.**
- **Excellent inertness.**
- **Unsurpassed performance for the 508 pesticides and the 8151 herbicides.**

be minimized by programming to a high temperature at the end of the analysis. The Rtx-CLPesticides and Rtx-CLPesticides2 columns have 330°C temperature limits, allowing for high temperature "bake out" to minimize the effects of high-boiling contaminants. While these contaminants don't always appear in the GC-ECD chromatogram, they can cause shifts in retention time, elevated baselines, and target compound breakdown.

The inertness of the stationary phase is important to minimize the possibility of on-column breakdown. Restek has published the details of this in previous presentations<sup>1</sup> and

issues of *The Restek Advantage*<sup>2</sup>. This is most often observed in cyanopropyl capillary column stationary phases ("1701") which very often provides the best resolution between target compounds, but suffers from poor inertness and reduced thermal stability. Both the Rtx-CLPesticides and the Rtx-CLPesticides2 columns have excellent inertness and will not be prone to the on-column breakdown problems observed with the "1701" phases.

While the performance of these columns is unmatched for the 22 chlorinated pesticides as listed in USEPA 8081, many laboratories also use the same GCs for other analyses. It is

*Continued on page 2.*

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# Rtx®-CLPesticides & Rtx®-CLPesticides2

Continued from Page 1

common for the same instruments to be used for herbicide analysis (EPA Method 8151) and the extended pesticide compound lists (Method 508). The Rtx-CLPesticides and Rtx-CLPesticides2 columns also exhibit excellent performance for these analyses as well, making them the clear choice for any pesticide/PCB/herbicide analysis.

In summary, the combination of the Rtx-CLPesticides and Rtx-CLPesticides2 columns provide unsurpassed performance for the analysis of chlorinated pesticides. They can be conditioned at the end of each analysis to remove high-boiling contaminants without degrading the stationary phase. They also give improved response and reduced breakdown of methoxychlor and DDT. By designing the

stationary phase with the requirements of the method in mind, Restek is leading the way into the next era of chromatography. If you are involved with the analysis of chlorinated pesticides and want to improve your resolution capacity to analyze dirty extracts, and increase your throughput, try the Rtx-CLPesticides and Rtx-CLPesticides2 columns.

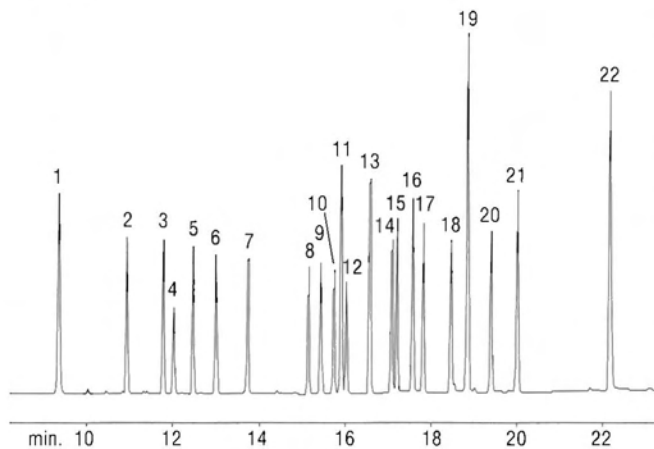
1. 1997 Pittsburgh Conference, Paper 1187.
2. The Restek Advantage, Summer 1996.

Convenient, cost-saving kits are available that include both Rtx-CLPesticides columns, a guard column, and a 5-pack of Press-Tight® connectors.

Call your local distributor for more information!

Figure 1:

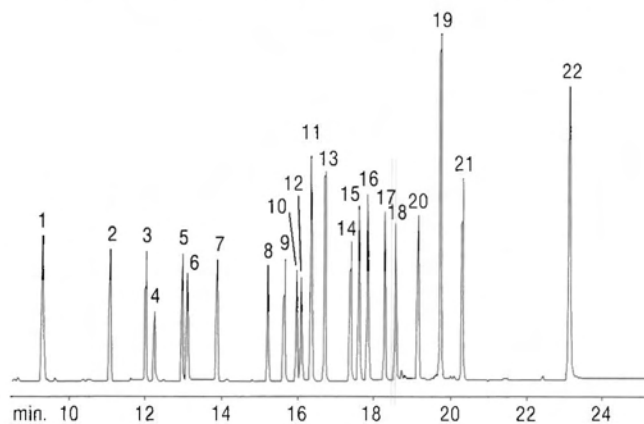
The Rtx-CLPesticides column provides baseline resolution of 22 chlorinated pesticides in EPA 8081 in less than 23 minutes.



30m, 0.32mm ID, 0.50µm Rtx-CLPesticides column (cat # 11139) run in parallel with the Rtx-CLPesticides2 column. Oven temp.: 120°C (hold 1min.) to 300°C @ 9°C/min. (hold 10min.); Dead time: 1.4 min. Carrier gas: He.

Figure 2:

The Rtx-CLPesticides2 column is the ideal confirmation column to the Rtx-CLPesticides column, with 4 elution order changes, baseline resolution of 22 components, and a run time of less than 24 minutes for EPA 8081 pesticides.



30m, 0.32mm ID, 0.25µm Rtx-CLPesticides2 column (cat # 11324) run in parallel with the Rtx-CLPesticides column. Oven temp.: 120°C (hold 1min.) to 300°C @ 9°C/min. (hold 10min.); Dead time: 1.4 min. Carrier gas: He.

## Peak List for Figures 1 and 2

- |                                 |                        |
|---------------------------------|------------------------|
| 1. 2,4,5,6-tetrachloro-m-xylene | 12. endosulfan I       |
| 2. α-BHC                        | 13. dieldrin           |
| 3. γ-BHC                        | 14. endrin             |
| 4. β-BHC                        | 15. 4,4'-DDD           |
| 5. δ-BHC                        | 16. endosulfan II      |
| 6. heptachlor                   | 17. 4,4'-DDT           |
| 7. aldrin                       | 18. endrin aldehyde    |
| 8. heptachlor epoxide           | 19. methoxychlor       |
| 9. γ-chlordane                  | 20. endosulfan sulfate |
| 10. α-chlordane                 | 21. endrin ketone      |
| 11. 4,4'-DDE                    | 22. decachlorobiphenyl |

## Product Listing:

*New*

## Rtx-CLPesticides2 Column

Description	Cat.#
30m, 0.25mm ID, 0.20µm	11323
30m, 0.32mm ID, 0.25µm	11324
30m, 0.53mm ID, 0.42µm	11340
Original Rtx-CLPesticides Columns	
Description	Cat.#
30m, 0.25mm ID, 0.25µm	11123
30m, 0.32mm ID, 0.50µm	11139
30m, 0.53mm ID, 0.50µm	11140

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# Restek Breathes New Life into Gas Analysis with the new Rt-Msieve 5A PLOT Column

by Andy Schuyler

Restek's novel Rt-Msieve 5A porous layer open tube (PLOT) column is now available for the separation of the permanent gases (e.g. hydrogen, argon, oxygen, nitrogen, methane and carbon monoxide) commonly analyzed in natural gas and petrochemical samples. Unlike packed molecular sieve columns, PLOT columns have fast analysis times and can baseline resolve difficult to separate components like argon and oxygen without cryogenic cooling of the column oven. This critical resolution, demonstrated in **Figure 1**, is easily obtained at 30°C in less than 7 minutes.

PLOT columns are an excellent choice for fast separations of permanent gases and light hydrocarbons because they provide the high efficiency and retention required without the high pressures, wide peaks and long analysis times encountered with packed columns. Gas analysis systems often use multiple columns and valve switching, so it is critical that the micron-sized particles coating the inside of the PLOT column are completely immobilized. Restek has developed a series of revolutionary column coating and particle adhesion technologies that produces PLOT columns with no particle generation (which can cause detector noise and permanent damage to delicate column switching valves) and consistently high

efficiency and retention time reproducibility.

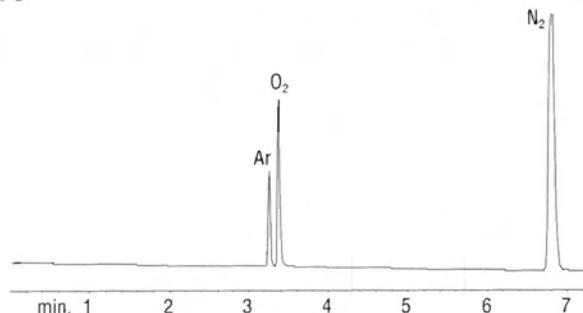
Selection of PLOT columns for the analysis of gases couldn't be easier now that Restek offers a fully re-engineered product line of PLOT columns including the Rt-Alumina, Rt-Msieve 5A and 13X and several porous polymer columns such as Rt-Q PLOT and Rt-S PLOT. The Rt-Msieve 5A is ideal for the separation of Hydrogen/Helium, Ar/O<sub>2</sub>, and noble gases. It is also an excellent choice for rapid separation of permanent gases in refinery gas or natural gas as shown in **Figure 2**. Note the excellent resolution, peak symmetry, and the rapid analysis time of less than 4 minutes.

Restek's re-engineered PLOT columns incorporate the latest technological advances in column coating and particle binding to provide affordable, high quality columns without particle generation. These columns are rugged, easy to use, and provide excellent separations in less time than conventional columns.

For a complete listing of PLOT columns, see Restek's 1998 *Chromatography Products Catalog* or call your local distributor for technical support.

**Figure 1:**

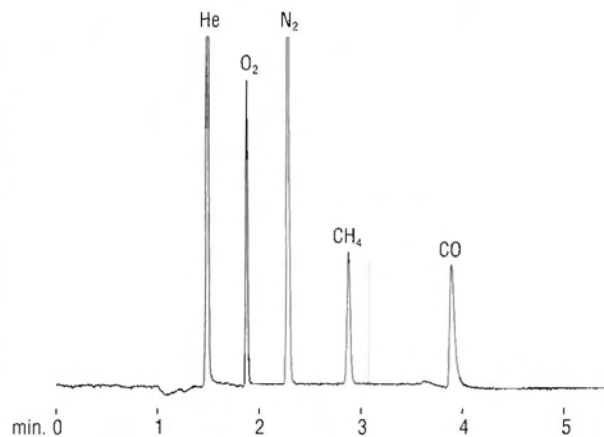
The Rt-Msieve 5A provides baseline resolution of Argon/Oxygen in Air in under 7 minutes without cryo-cooling.



30m, 0.32mm ID Rt-Msieve 5A PLOT column (cat # 19722). Column temp.: 30°C; Carrier gas: hydrogen; Linear velocity: 30 cm/sec.; Detector: MicroTCD.

**Figure 2:**

The Rt-Msieve 5A resolves permanent gases in refinery gas, natural gas, and transformer oil gas.



30m, 0.53mm ID Rt-Msieve 5A PLOT column (cat # 19723). Column temp.: 100°C; Carrier gas: hydrogen; Linear velocity: 30 cm/sec.; Injector temp.: 30°C; Detector: TCD. 150°C; Attenuation: 8; Range: 0.05 AFS.

## Product Listing:

### Rt-Msieve 5A Columns

*great value!*

Description	Cat.#
15m, 0.32mm ID	19720
15m, 0.53mm ID	19721
30m, 0.32mm ID	19722
30m, 0.53mm ID	19723

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INTERNATIONAL



# Get More Dirt on the Trident™ HPLC Guard Column System

by Randy Romesberg

- **New 20 x 4mm cartridge offers twice the protection for extremely dirty samples.**
- **10 x 2mm design reduces band broadening in LC-MS.**
- **Trident's integral design eliminates troublesome tubing connections.**

Now you can catch even more dirt on the Trident HPLC guard column system with Restek's new 20 x 4mm guard cartridge. The longer cartridge offers twice the protection as the original 10 x 4mm for extremely dirty samples. For small internal diameter columns a 10 x 2mm cartridge is now available which reduces band broadening in the small internal diameter columns used in LC-MS.

The new sizes offer the same versatile configurations and convenient leak-free operation without the need for troublesome connecting tubing just like the original Trident system.

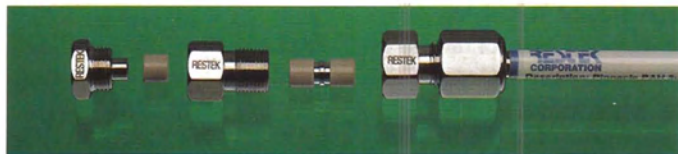
Restek's innovative Guard Column system consists of the analytical column configured with our exclusive Trident™ end fitting and XF filter fitting. This configuration contains the standard internal frit as well as a replaceable external frit, which can be easily changed without disturbing the packed bed. Changing the external frit can reverse the effects of accumulated particles, such as high back pressure or peak distortion. Add a "-700" to any Restek HPLC column catalog number to order this basic configuration.



The system can also be configured to accept an integral guard cartridge for greater protection against sample contaminants. The integral design eliminates the need for a separate holder and connecting tubing, which can cause additional band broadening. To obtain this configuration, order any Restek HPLC column (include the -700 suffix), the XG male fitting, and the appropriate pack of guard cartridges.



For maximum protection against contaminants and particulate matter, the system can be configured with both an integral guard cartridge and a replaceable external frit. To obtain this configuration, order any Restek HPLC column (include the -700 suffix), the XG-XF male fitting, and the appropriate pack of guard cartridges.







## Product Listing:

### Trident<sup>SM</sup> HPLC Guard Column Cartridges

3-pack Guard Cartridge (10x4mm)	3 $\mu$ m cat.#	5 $\mu$ m cat.#
Amino	911730210	911750210
Butyl	N/A*	911250210
Cyano	N/A*	911650210
Cyano Amine	918630210	918650210
EcoSep	N/A*	917150210
Methyl	911130210	911150210
Octyl	911330210	911350210
Octyl Amine	918330210	918350210
ODS	911430210	911450210
ODS Amine	918430210	918450210
PAH	917030210	917050210
Phenyl	N/A*	911550210
Phenyl Amine	918530210	918550210
SAX	N/A*	911850210
Silica	911030210	911050210
TO-11	917230210	917250210
Ultra C18	N/A*	917450210
Kromasil C4	N/A*	920250210
Kromasil C8	N/A*	920350210
Kromasil C18	N/A*	920450210
Nucleosil C8	N/A*	930350210
Nucleosil C18	N/A*	930450210
Nucleosil CN	N/A*	930650210
Nucleosil SCX	N/A*	930950210

\* A 5 $\mu$ m guard column can be substituted.

All cartridges listed can be used with the appropriate in-line holder!

**Holder for 1cm Guard Cartridge:** cat.# 25021, ea.

**Holder for 2cm Guard Cartridge:** cat.# 25061, ea.

**XG Fitting for 1cm Guard Cartridge:** cat.# 25025, ea.

**XG Fitting for 2cm Guard Cartridge:** cat.# 25063, ea.

**XG-XF Fitting for 1cm Guard Cartridge:** cat.# 25026, ea.

**XG-XF Fitting for 2cm Guard Cartridge:** cat.# 25062, ea.

**Replacement XF Filter Fitting:** cat.# 25024, ea.

**Replacement Cap Frits:** cat.# 25022, 5-pk.



### Informative Technical Presentations at PittCon.

- Development of a Unique, Stable Hydrophilic Bonded Phase for HPLC. (Paper # 742, Tues., March 3, 4:05p.m.)
- Method Optimization for the HPLC Analysis of Aldehydes and Ketones (Poster # 1869P, Tues., March 3)
- Evaluation of a Novel HPLC Cartridge Design and Column Protection System (Poster #1928P, Tues., March 3)
- Enhanced Resolution of Parabens and Amines in Oral Solutions (Paper # 1219, Thurs., March 5, 10:45a.m.)

2-pack Guard Cartridge (20x4mm)	5 $\mu$ m cat.#
Amino	911750220
Butyl	911250220
Cyano Amine	918650220
Cyano	911650220
EcoSep	917150220
Methyl	911150220
Octyl Amine	918350220
Octyl	911350220
ODS Amine	918450220
ODS	911450220
PAH	917050220
Phenyl Amine	918550220
Phenyl	911550220
SAX	911850220
Silica	911050220
TO-11	917250220
Ultra C18	917450220
WP Butyl	913250220

3-pack Guard Cartridge (10x2mm)	5 $\mu$ m cat.#
Amino	911750212
Butyl	911250212
Cyano Amine	918650212
Cyano	911650212
EcoSep	917150212
Methyl	911150212
Octyl Amine	918350212
Octyl	911350212
ODS Amine	918450212
ODS	911450212
PAH	917050212
Phenyl Amine	918550212
Phenyl	911550212
SAX	911850212
Silica	911050212
TO-11	917250212
Ultra C18	917450212
WP Butyl	913250212
WP Butyl	913250210

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INTERNATIONAL



# Grape Flavor Analysis using the New Rt- $\gamma$ DEXsa Column

by Sherry Sponsler

- **Gamma-cyclodextrin provides unique chiral separations.**
- **0.25mm and 0.32mm IDs are ideal for use in GC/MS flavor analysis.**
- **Thermally stable to 230°C with low bleed.**

Recent articles have demonstrated the utility of *beta*-cyclodextrin columns for flavor analysis, but sometimes the larger *gamma*-cyclodextrins provide better enantioselectivity of chiral indicating compounds. Restek's new Rt- $\gamma$ DEXsa column provides better separation of specific chiral compounds in grape flavor than most *beta*-cyclodextrin phases.

The enantiomeric ratios of certain chiral compounds found in flavorings can sometimes reveal adulteration. Two chiral indicators in grape flavor are methyl 3-hydroxybutyrate and ethyl 3-hydroxybutyrate. Methyl 3-hydroxybutyrate is racemic and (R)-ethyl 3-hydroxybutyrate is at least 77% predominant in natural grape flavor. Extreme alterations of these ratios can indicate that the flavor is not completely authentic.

Beta-cyclodextrin stationary phases provide enantioselectivity for a variety of chiral compounds, but not for methyl 3-hydroxybutyrate and ethyl 3-hydroxybutyrate.

**Figure 1** illustrates analysis of these compounds on an Rt- $\beta$ DEXsm, a 2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl- $\beta$ -cyclodextrin column. The compounds tail and exhibit poor enantiomer separation.

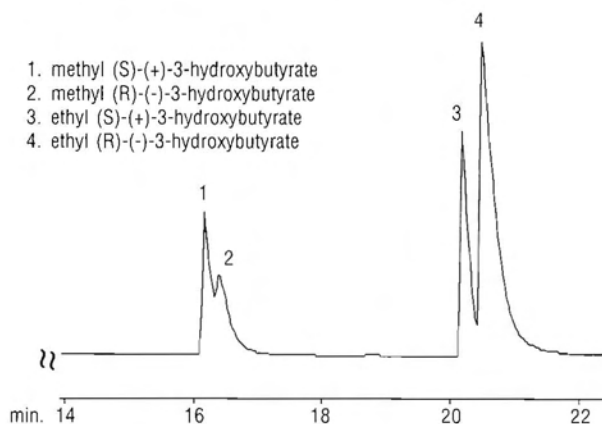
*Beta*-cyclodextrins with different derivatives can help increase selectivity. **Figure 2** reveals that the Rt- $\beta$ DEXsa, a 2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl- $\beta$ -cyclodextrin column, improves enantioselectivity for methyl 3-hydroxybutyrate but not for ethyl 3-hydroxybutyrate.

Although the exact mechanisms of compound-cyclodextrin interaction are not known, it is evident that the size of the cyclodextrin cavity is important. Perhaps some compounds may be too large to effectively interact with the cavity size of *beta*-cyclodextrin, which is composed of 7 glucopyranose units. *Gamma*-cyclodextrins are composed of 8 glucopyranose units and possess a larger cavity, which may be more interactive with larger chiral molecules.

Analysis of methyl 3-hydroxybutyrate and ethyl 3-hydroxybutyrate on the new Rt- $\gamma$ DEXsa column reveals excellent chiral selectivity for both, as shown in **Figure 3**.

**Figure 1:**

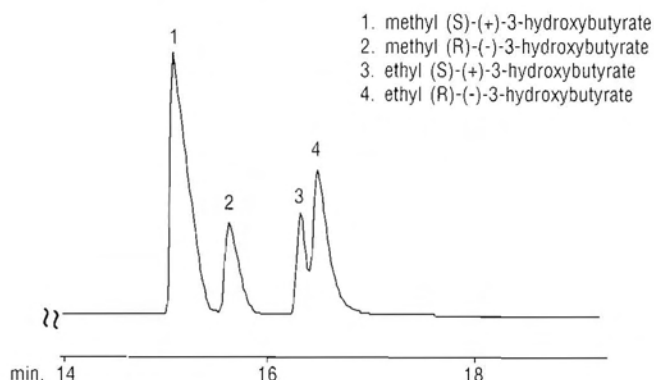
The Rt- $\beta$ DEXsm column exhibits poor enantiomeric separation of methyl 3-hydroxybutyrate and ethyl 3-hydroxybutyrate in grape flavor extract.



30m, 0.32mm ID, 0.25 $\mu$ m Rt- $\beta$ DEXsm (cat # 13104). 1 $\mu$ l split injection of methyl and ethyl 3-hydroxybutyrate. On-column concentration: ~150ng/enantiomer. **Oven temp.:** 40°C (hold 1min.) to 200°C @ 2°C/min., **Inj. & Det. temp.:** 200°C & 230°C; **Carrier gas:** hydrogen; **Linear velocity:** 80cm/sec. set @ 40°C; **Split ratio:** 25:1.

**Figure 2:**

The Rt- $\beta$ DEXsa column exhibits improved enantiomeric separation of methyl 3-hydroxybutyrate but not for ethyl 3-hydroxybutyrate in grape flavor extract.



30m, 0.32mm ID, 0.25 $\mu$ m Rt- $\beta$ DEXsa (cat # 13108). 1 $\mu$ l split injection of methyl and ethyl 3-hydroxybutyrate. On-column concentration: ~150ng/enantiomer. **Oven temp.:** 40°C (hold 1min.) to 200°C @ 2°C/min., **Inj. & Det. temp.:** 200°C & 230°C; **Carrier gas:** hydrogen; **Linear velocity:** 80cm/sec. set @ 40°C; **Split ratio:** 25:1.



Peak shape and enantiomer separation are improved using the larger cyclodextrin molecules.

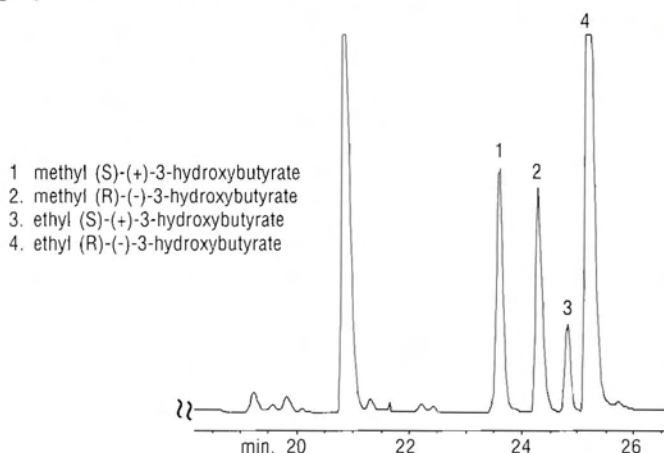
The Rt- $\gamma$ DEXsa contains 2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl- $\gamma$ -cyclodextrins that are dissolved into cyanopropyl phenyl stationary phase (Rtx<sup>®</sup>-1701). This composition promotes thermal stability to a maximum temperature of 230°C and longevity that is comparable to other capillary columns. This column is available in 0.32 mm ID and also 0.25 mm ID for direct interfacing into a mass spectrometer. Column dimension and stationary phase stability promote utility for GC/MS analysis as well.

Beta-cyclodextrin phases can separate a variety of chiral

indicating compounds in flavors, but are not effective in all applications. Using different cyclodextrin derivatives can help chiral selectivity, but going to a larger cyclodextrin sometimes is necessary. Switching from a 2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl- $\beta$ -cyclodextrin to a 2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl- $\beta$ -cyclodextrin column partially improved enantiomer separation for chiral indicating compounds in grape flavor. However, the 2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl- $\gamma$ -cyclodextrin column provided the best results. The new Rt- $\gamma$ DEXsa column can offer chiral selectivity for certain separations that are not achievable with beta-cyclodextrin columns.

**Figure 3:**

The new Rt- $\gamma$ DEXsa column reveals excellent chiral selectivity for both methyl and ethyl 3-hydroxybutyrate in grape flavor extract.

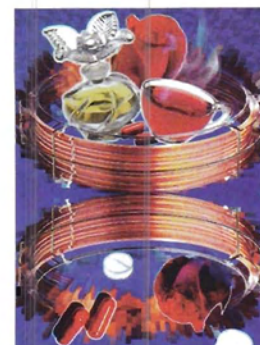


1. methyl (S)-(+)-3-hydroxybutyrate
2. methyl (R)-(-)-3-hydroxybutyrate
3. ethyl (S)-(+)-3-hydroxybutyrate
4. ethyl (R)-(-)-3-hydroxybutyrate

30m, 0.32mm ID, 0.25 $\mu$ m Rt- $\gamma$ DEXsa (cat # 13113). 1 $\mu$ l split injection of methyl and ethyl 3-hydroxybutyrate. On-column concentration: ~150ng/enantiomer. **Oven temp.:** 40°C (hold 1min.) to 200°C @ 2°C/min., **Inj. & Det. temp.:** 200°C & 230°C; **Carrier gas:** hydrogen; **Linear velocity:** 80cm/sec. set @ 40°C; **Split ratio:** 25:1

Call today for your free  
copy of Restek's  
"A Guide to the  
Analysis of Chiral  
Compounds by GC"

cat.# 59889



**Product  
Listing:**

*New*

**Rt- $\gamma$ DEXsa Columns**

Description	Cat.#
30m, 0.32mm ID, 0.25 $\mu$ m	13112
30m, 0.25mm ID, 0.25 $\mu$ m	13113

Restek offers a wide range of cyclodextrin columns for the analysis of many chiral compounds.

**Rt- $\beta$ DEXsm Columns**

30m, 0.25mm ID, 0.25 $\mu$ m	13105
30m, 0.32mm ID, 0.25 $\mu$ m	13104

**Rt- $\beta$ DEXse Columns**

30m, 0.25mm ID, 0.25 $\mu$ m	13107
30m, 0.32mm ID, 0.25 $\mu$ m	13106

**Rt- $\beta$ DEXsp Columns**

30m, 0.25mm ID, 0.25 $\mu$ m	13111
30m, 0.32mm ID, 0.25 $\mu$ m	13110

**Rt- $\beta$ DEXsa Columns**

30m, 0.25mm ID, 0.25 $\mu$ m	13109
30m, 0.32mm ID, 0.25 $\mu$ m	13108

**Rt- $\beta$ DEXcst Columns**

30m, 0.25mm ID, 0.25 $\mu$ m	13103
30m, 0.32mm ID, 0.25 $\mu$ m	13102

**Rt- $\beta$ DEXm Columns**

30m, 0.25mm ID, 0.25 $\mu$ m	13100
30m, 0.32mm ID, 0.25 $\mu$ m	13101

**For more information on this analysis,  
call your local distributor and request the  
"Grape Flavor Analysis using the New  
Rt- $\gamma$ DEXsa Column" Application Note.**

(cat.# 59553)

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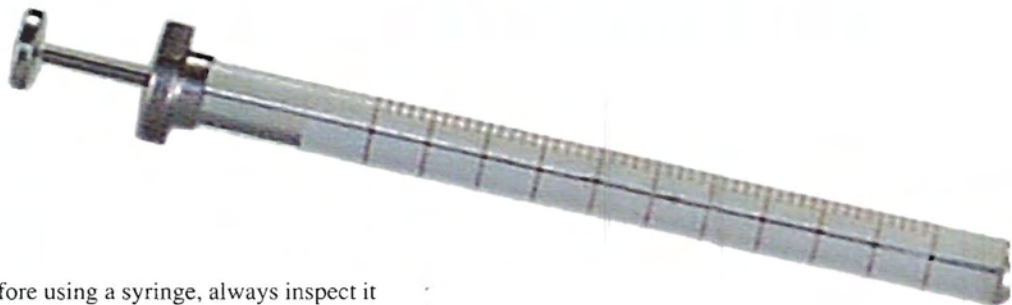
INTERNATIONAL



# On The Care and Handling

Compiled by Doug Elliott from the Technical Literature

High quality laboratory syringes are precision fluid measuring and delivery devices. They are not designed to be disposable. Maintenance is the key factor in determining the life-time of a syringe. With proper care and handling, quality laboratory syringes will provide superior performance for many years.



## Needle Gauge Size

Gauge	Nominal OD	Nominal ID
26s	.47mm	.13mm
26	.46mm	.26mm
25	.51mm	.26mm
23s	.64mm	.15mm
23	.64mm	.34mm
22s	.72mm	.15mm
22	.72mm	.41mm

Gauge	Nominal OD	Nominal ID
26s	.019 in.	.0050 in.
26	.018 in.	.0102 in.
25	.021 in.	.0102 in.
23s	.025 in.	.0060 in.
23	.025 in.	.0132 in.
22s	.028 in.	.0060 in.
22	.028 in.	.0162 in.

Before using a syringe, always inspect it for cracks in the barrel or for needle tip burrs. A cracked barrel syringe may leak or break during use and should be disposed of properly. Needle tips can be dressed by using fine emery paper, carborundum, or a soap stone to sharpen and remove burrs. Prevent sample carryover by flushing the syringe with solvent 5–20 times. *Caution! The first 2–3 washes should be discarded into a waste container and not returned to the clean solvent vessel.*

### Routine Maintenance

To clean the syringe and plunger for storage it is best to use a solvent known to be effective in solvating the sample and preferably be non-alkaline, non-phosphate, and non-detergent based. Remove the plunger from the barrel, rinse with solvent and gently wipe it with a lint free tissue. Always avoid touching the plunger since any abrasions, scratches, or skin oils will often interfere with proper plunger operation. Reinsert the plunger into the barrel and pump deionized water, acetone, or suitable solvent through the needle and syringe. (Although the adhesives used to cement the needles to the syringes are chemically resistant, some solvents and

cleaning conditions may deteriorate the bond, resulting in frozen plungers and plugged needles. For this reason, you should avoid immersing the syringe in the cleaning solution for prolonged times.) Allow the syringe to air dry before being placed into storage.

### Tech Tip:

When reinserting a Teflon®-tipped plunger into a syringe barrel, lubricate the tip with water or a solvent compatible with the sample. Do not pump the plunger when it is dry to prevent premature wear and possible leakage. To restore the Teflon®-tipped plunger seal or to reduce excess plunger force, take advantage of Teflon's flow properties by heating the assembled syringe in an oven at 60°C for 10-20 minutes.





# ing of Analytical Syringes

ure of Hamilton Company and SGE, Incorporated

## Needle Considerations

When selecting a needle, choose the largest outside diameter effectively possible to minimize the chance of bending. The inside diameter should be kept to a minimum ('s' designation). This minimizes the needle dead volume while increasing the wall thickness to strengthen the needle without compromising the ability of the syringe to draw normal viscosity samples. Medium to high viscosity samples should be diluted prior to use or a larger ID needle should be selected.

Should a needle become clogged, do not attempt to clean it by forcing liquid or compressed air through the syringe.

Excessive pressure will split the barrel. Remove the plunger from the syringe and use needle cleaning wires to dislodge any foreign material or residue build-up. Rinse with the appropriate solvent and air dry to complete the process.

Heated syringe cleaners were designed primarily for cleaning the plunger-in-needle microvolume syringes. They use a combination of heat ( $>350^{\circ}\text{C}$ ) and vacuum to vaporize and extract contaminants from syringe needles. Because they require an open flow to work effectively, they cannot work in cleaning plugged needles and freeing seized plungers. By using a voltage rheostat to lower the chamber temperature to  $\sim 50^{\circ}\text{C}$ , the syringe cleaners can be used successfully to clean gas-tight and microliter volume syringes.

**WARNING!** *Never force a plunger. Do not pump the plunger when the needle is blocked as the high pressure generated could crack the barrel. Always avoid any unnecessary movement of plungers when the syringe is dry. Damage to the inner barrel or plunger could result.*

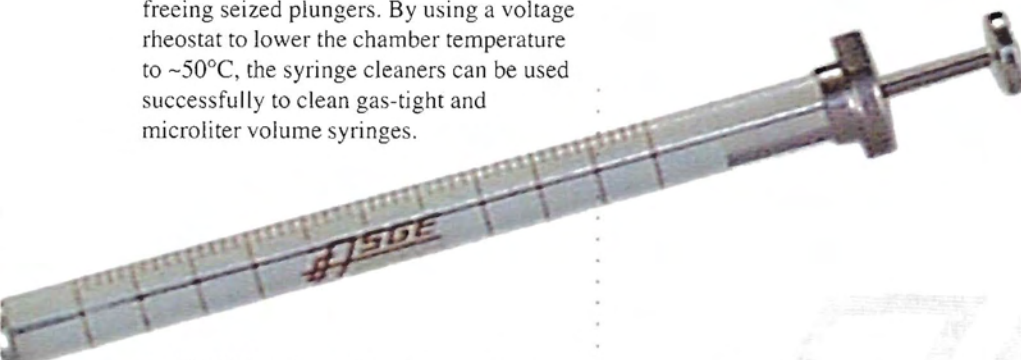
## Conclusion

Today's high quality laboratory syringes are engineered devices designed for accuracy and precision. With the proper use and care they will provide years of superior performance. This performance is directly related to the cleanliness of the syringes.

You will find the most popular styles of laboratory syringes from Hamilton and SGE in Restek's 1998 Chromatography Products Guide. Special orders can be placed if there is a syringe type or service component you need that is not listed in the catalog. Please call us at 1-800-356-1688, ext. 4.

We would like to hear from you on how you keep your syringes working well for a long time. We will pass this information along to the other readers when we write about recommended sampling and injection techniques in a future article. Contact Doug by e-mail at: [doug@restekcorp.com](mailto:doug@restekcorp.com).

Restek is pleased to announce that we now distribute a full line of SGE Syringes. For over 25 years SGE has been providing a comprehensive range of analytical syringes unsurpassed in design, quality, and performance. Please review Restek's 1998 Chromatography Products Guide for the SGE syringes we have in stock for immediate delivery to you.



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INTERNATIONAL



# Practical Time Savings in Gas Chromatography Method Development

by Chris Linton, Sherry Sponsler, & Jingzhen Xu

## Theory

Selectivity, efficiency and time are interdependent in chromatography. Theoretically we can predict the minimum time required to maximize selectivity and efficiency.

### Considering:

- $T_p$  = the time to get a solute past 1 plate
- $N_{req}$  = the number of plates required for resolution
- $T_r$  = retention time required for desired resolution
- $H$  = height equivalent to a theoretical plate
- $\mu$  = linear velocity
- $k'$  = partition coefficient
- $\alpha$  = selectivity ratio
- $1+k'$  = the amount of time a solute spends in the stationary phase
- $R$  = resolution

$$T_r = N_{req} \cdot T_p$$

$$T_p = (1+k')/\mu$$

so:  $T_r = N_{req}(1+k')(H/\mu)$

substituting:

$$N_{req} = 16R^2(\alpha-1)^2/(1+k')^2 \quad \text{Purnell equation}$$

so:  $T_r = 16R^2(\alpha-1)^2[(1+k')^3/(k')^2](H/\mu)$   
solve for the first derivative holding all values constant except  $k'$

$$T_r = C(k'^3 + 3k'^2 + 3k' + 1)/k'^2$$

$$dT_r/dk' = C - 3Ck'^{-2} - 2Ck'^{-3}$$

in order to find  $T_r$  minimum,  $dT_r/dk'$  must be set to 0

$$k'^3 - 3k' - 2 = 0 \quad k' = 2 \text{ or } -1$$

Theoretically, the optimum time for a solute to exit a column is at  $k'=2$ . At this time, selectivity and efficiency are optimized. Unfortunately, this model breaks down if a multicomponent sample is considered (all analytes cannot have  $k'=2$ ). In reality, column length, initial starting temperature and temperature programming can be utilized to adjust retention times close to the theoretical optimum  $k'=2$ . **The stationary phase must provide adequate retention (film thickness) and selectivity for the resolution requirements of the separation at hand.**

The speed of analysis in capillary gas chromatography can be significantly improved by reducing the bore size of the column ( $<100\mu\text{m}$ ). Researchers have successfully produced chromatograms on the order of milliseconds using this approach. Unfortunately, heavy demands are placed on sample introduction and peak detection which are not within the realm of capabilities of common instrumentation. The following discussion concentrates on practical time reduction in gas chromatographic separations using standard diameter columns and sample introduction with the Hewlett-Packard 5890 GC.

### Temperature Programming

Sample mixtures may contain analytes that vary in volatility to the extent where temperature programming becomes essential in the analysis. A temperature program can be selected such that high volatility compounds elute at low column temperature and low volatility compounds elute at higher column temperature ensuring the minimization of  $k'$  values. Program rates may be as high as 25 degrees per minute or greater in order to achieve near optimum  $k'$  values.

Another important aspect of temperature programming and column selection is the requirement that all analytes elute during the temperature program. This general rule helps to define column length and limits analysis time and band broadening by preventing isothermal elution of low volatility analytes. Since the temperature program was not optimized for the 45-meter column in **Figure 1**, the last five components were forced to elute under isothermal conditions.

### The Limitation of Length

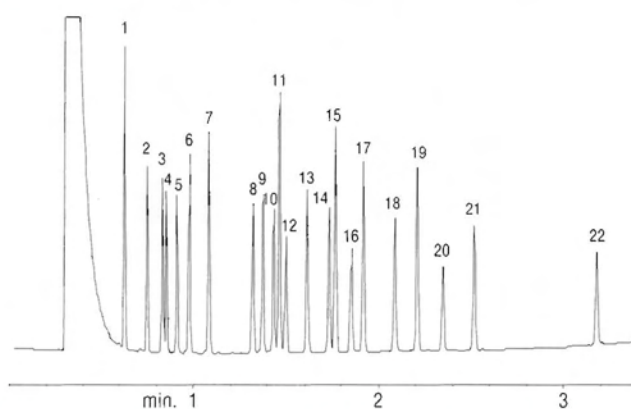
Longer columns generally provide greater challenge for the generation of time savings in gas chromatography. Stationary phases have inherent maximum operating temperatures which in turn limits the extent of a temperature program. **Therefore, a minimum length of column should be chosen to provide not only the minimum required plates but the shortest retention times around the optimum  $k'$  value of 2.** The stationary phase must provide adequate retention (film thickness) and selectivity for the resolution requirements of the separation at hand. The following set of chlorinated pesticide chromatograms illustrates the differences between a near optimized separation on a 15-meter column and the same chromatographic conditions using a 45-meter column. Notice that the retention times are nearly half on the 15-meter column and all components are fully resolved.



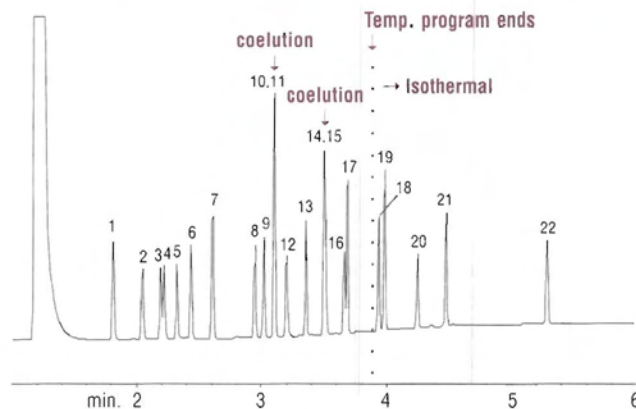


**Figure 1:**

Under optimized conditions, a shorter column can provide improved resolution and decreased analysis time compared to a longer column.



15m, 0.25mm ID, 0.25 $\mu$ m Rtx-CLPesticides column.  
Oven temp.: 200°C to 300°C @ 25°C/min. Carrier gas: hydrogen.



45m, 0.25mm ID, 0.25 $\mu$ m Rtx-CLPesticides column.  
Oven temp.: 200°C to 300°C @ 25°C/min. Carrier gas: hydrogen.

### Initial Starting Temperature

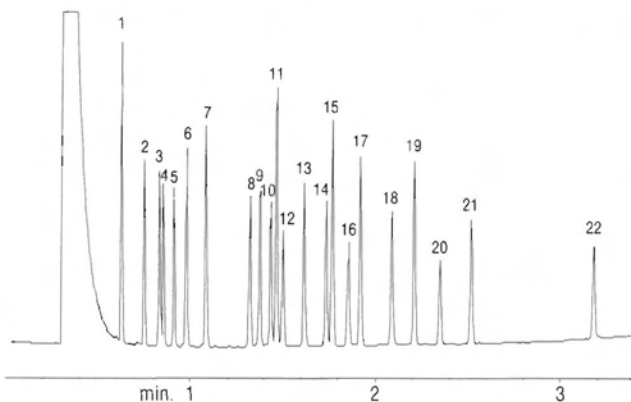
In temperature programming and isothermal gas chromatography, a time savings may be generated by choosing an initial starting temperature which allows for the elution of the first analyte near the solvent front. Starting at initial temperatures which are too low creates an empty gap in time which does not make sense theoretically due to  $k'$  elevation. The chromatograms in **Figure 2** illustrate the retention time differences generated by starting temperatures of 40°C and 200°C in the chlorinated pesticide analysis.

### Peak List for Figures 1 & 2

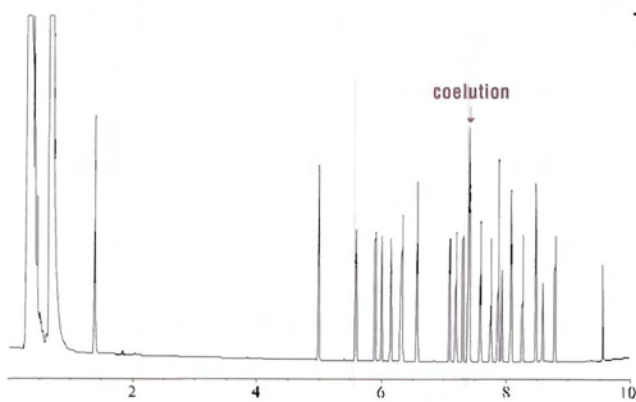
- |                                 |                        |
|---------------------------------|------------------------|
| 1. 2,4,5,6-tetrachloro-m-xylene | 12. endosulfan I       |
| 2. $\alpha$ -BHC                | 13. dieldrin           |
| 3. $\gamma$ -BHC                | 14. endrin             |
| 4. $\beta$ -BHC                 | 15. 4,4'-DDD           |
| 5. $\delta$ -BHC                | 16. endosulfan II      |
| 6. heptachlor                   | 17. 4,4'-DDT           |
| 7. aldrin                       | 18. endrin aldehyde    |
| 8. heptachlor epoxide           | 19. methoxychlor       |
| 9. $\gamma$ -chlordane          | 20. endosulfan sulfate |
| 10. $\alpha$ -chlordane         | 21. endrin ketone      |
| 11. 4,4'-DDE                    | 22. decachlorobiphenyl |

**Figure 2:**

At higher initial temperatures, analysis times can be decreased without sacrificing resolution.



15m, 0.25mm ID, 0.25 $\mu$ m Rtx-CLPesticides column.  
Oven temp.: 200°C to 300°C @ 25°C/min. Carrier gas: hydrogen.



15m, 0.25mm ID, 0.25 $\mu$ m Rtx-CLPesticides column.  
Oven temp.: 40°C to 300°C @ 25°C/min. Carrier gas: hydrogen.



# Practical Time Savings in Gas Chromatography Method Development

*Continued from page 11.*

## Choosing a Carrier Gas

**Figure 3** illustrates the importance of using hydrogen carrier gas to create time savings in gas chromatography. Notice the slopes of the van Deemter plot curves for each of the represented gases. Hydrogen can be used at much higher speeds without significant increases in HETP. Therefore, hydrogen carrier can generate more plates per time than nitrogen or helium making it the time saving carrier gas of choice. As an example, the chromatogram to the left in Figure 1 was generated at approximately 80 cm/sec., which is twice the theoretical optimum for hydrogen carrier.

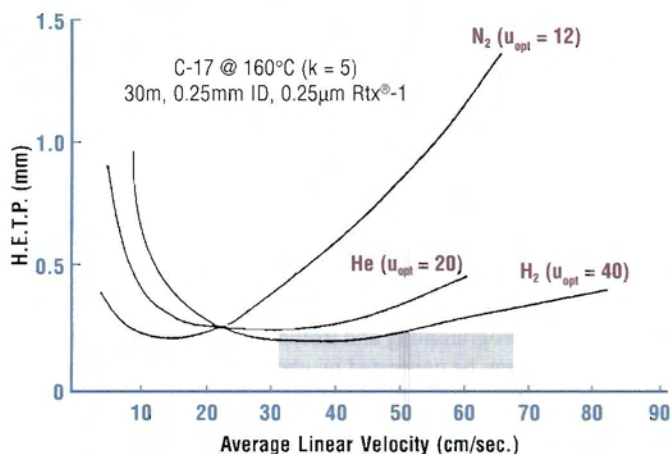
## Conclusion

The preceding discussion was presented in order to provide some practical guidelines in gas chromatography method development in a fashion that may help the analyst save time—our most precious resource. The relationship of selectivity, efficiency and time must be realized by the analyst allowing one to choose the appropriate column dimensions, stationary phase, and carrier gas.

In theory, it is fairly easy to derive optimum retention in gas chromatography. Unfortunately, real world samples provide many challenges resulting in deviation from theoretical calculations. The separation of multicomponent mixtures will always require a selective stationary phase and thoughtful selection of chromatographic parameters such as initial starting temperature and

**Figure 3:**

The van Deemter plots demonstrate the benefits of using hydrogen carrier gas at increased speeds and low HETP values.



temperature programming. The culmination of these ideas should be useful to the analyst concerning practical time reduction in capillary gas chromatography.

## Chromatography Reference Books

### *Modern Practice of Gas Chromatography, 3rd Edition*

(Edited by Robert L. Grob, Villanova University)

A book for both beginners and specialists, this work covers principles, instrumentation techniques, and applications of GC.

John Wiley & Sons, Inc., 1995 • 800pp.

cat.# 20464, ea.

### *Sampling and Analysis of Airborne Pollutants*

(Eric D. Winegar and Lawrence H. Keith)

This book provides you with the tools, techniques, and procedures you need to understand and conduct successful sampling and analysis projects. From electro-optical remote sensing to new directions in sampling techniques, this is your guide!

1993 • 384 pp.

cat.# 20468, ea.

### *Split and Splitless Injection in Capillary GC, 3rd Edition*

(Konrad Grob)

Represents one of the most comprehensive, single-volume treatment of all aspects of split and splitless injection. The book is divided into four sections: split injection, splitless injection, problems arising from the heated syringe needle in vaporizing injection, and Programmed Temperature Vaporizing (PTV) injection.

Huethig Publishing, Ltd., 1993 • 547pp.

cat.# 20451, ea.



# Silcosteel® Sample Cylinders

*For the accurate collection & analysis of sulfur compounds in refinery & natural gas.*

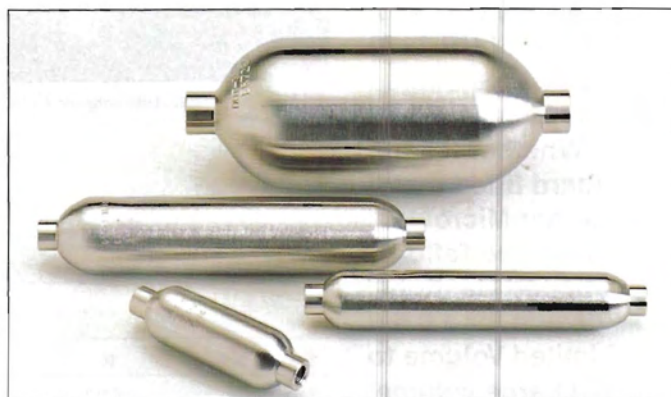


Stainless steel sample cylinders are commonly used in the collection and analysis of refinery and natural gas samples. These samples often contain trace amounts of sulfur containing compounds such as hydrogen sulfide, mercaptans and disulfides. Sulfur compounds in refinery gas can interfere with reactions or damage catalysts in many petrochemical processes. Since sulfur compounds quickly react with stainless steel surfaces, accurate determination of these active compounds is impossible when using normal sample cylinders.

The solution to this problem is Silcosteel®-treated sampling cylinders. Restek's Silcosteel® process produces a thin layer of silica on the stainless steel, resulting in a very inert surface. Even trace levels of sulfur compounds can be collected and stored in a Silcosteel®-treated cylinder without significant loss. **Figure 1** shows the stability of several common sulfur compounds at ~5.0 ppm in a Silcosteel®-treated stainless steel cylinder. The results indicate the stability of sulfur compounds over a 168 hour (7-day) period.

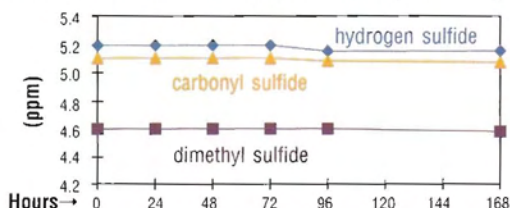
Silcosteel®-treated sample cylinders combine the inertness of glass with the strength of stainless steel and are ideal for

the analysis of sulfur compounds in refinery and natural gas samples.



**Figure 1:**

Results indicate no significant loss of sulfur compounds when stored in a Silcosteel® cylinder for up to 7 days.



**New**

## Sample Cylinders

- 1/4" NPT double-ended cylinders.
- Rated to 1800 psig D.O.T.

Cylinder Size	Cat.#
75cc	24271
150cc	24272
300cc	24273
500cc	24274
1000cc	24275

## Silcosteel® Sample Loops

- Available in sizes from 5µl to 5mls.
- For GC and HPLC use.
- Fittings supplied for easy connection to valve.

Prevent your analytes from being adsorbed in the sample loop of your chromatographic system by using Silcosteel®-treated sample loops from Restek.

**New**

## Silcosteel® Sample Loops for:

GC		HPLC	
Size	Cat.#	Size	Cat.#
250µl	22803	5µl	22805
500µl	22804	10µl	22806
1ml	22801	20µl	22807
5ml	22802		



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# Peak Performers

## Syringes for the Hewlett-Packard 7673 Autosampler

Restek now carries a full line of HP 7673 Autosampler syringes made by Hamilton and SGE. Whether it's the standard fitted metal plunger Microliter® style or the Teflon®-tipped Gastight® design, or a 0.5µl Limited Volume to 250µl Large Volume, we have the HP Autosampler syringe to meet your analytical requirements.

- Hamilton and SGE syringes are designed to meet critical autosampler specifications.
- The cone-shaped needle point style is developed to withstand multiple and fast septum injections.
- The 23s single gauge needle is the most popular size for the HP 7673 and the one that Restek stocks for same day shipment. For 26 and 23-26 gauge needle syringes please call for availability.
- 23 gauge needle fits the Merlin Microseal® Septum as well as standard septa on split/splitless and packed column injection ports.



HP 7673 10µl, Autosampler Cemented Needle (ASN)



HP 7673 Fixed Needle (F)

Single Syringes		
Volume (µl)	Needle Termination	
0.5	R	
5	ASN/F	
5	ASRN/R	
10	ASN/F	
10	ASRN/R	

HAMILTON		
Hamilton Model	Hamilton cat.#	Restek cat.#
Standard Microliter® Syringes		
7000-5	86276	24895
75	87987	20168
75	87957	20172
701	80387	20167
701	80357	20171

SGE		
SGE Model	SGE cat.#	Restek cat.#
0.5BR-HP-.63	000410	24791
5F-HP-.63	001810	24781
5R-HP-.63	001815	24793
10F-HP-.63	002810	24785
10R-HP-.63	002815	24795

Gastight® Syringes		
5	ASN	
10	ASN/F	
10	ASRN/R	
25	R	
50	R	
100	R	
250	R	

175	80074	24893
1701	80080	24894
1701	80087	24896
—	—	—
—	—	—
—	—	—
—	—	—

—	—	—
10F-HP-GT-.63	002812	24789
10R-HP-GT-.63	002818	24797
25R-HP-.63	003665	24798
50R-HP-.63	004665	24799
100R-HP-.63	005665	24800
250R-HP-.63	006665	24801

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5	ASN/F	
10	ASN/F	
10*	ASN/F	
10**	ASN	

75	87990	20170
701	80390	20169
701*	80391	24600
701**	80389	24599

SK-5F-HP-.63	001814	24783
SK-10F-HP-.63	002814	24787
—	—	—
—	—	—

\* 23-26 gauge needle; \*\* 26 gauge needle.

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SYRINGE SPECIFICATIONS:	
Needle Length:	1.71"/44mm
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Point Style:	HP/Cone
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ASRN/R:	Removable needle





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Crimp Top  
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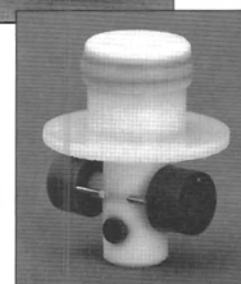
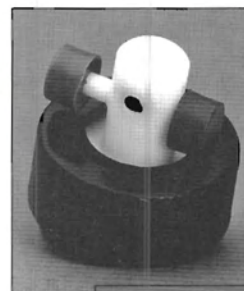
Description	100-pk.	1000-pk.
2.0mL Clear Vial w/ White Graduated Marking Spot*	24383	24384
2.0mL Amber Vial w/ White Graduated Marking Spot*	24385	24386

\* Blue, green, rust, or yellow colored marking spots are available on request.

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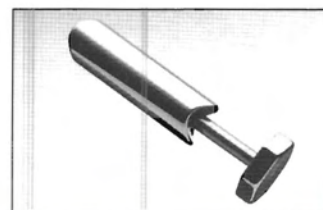
Models are available for screw-cap and crimp-top vials. The screw-cap Mininert is available in 13, 15, 18, 20, and 24 mm cap sizes. The crimp-top valve for 20 mm ID glassware slides into the neck of the vial and features a threaded flange which is turned to secure a tight fit.

### VICI Precision Sampling Mininert Valves for Vials

Cap Size	Thread Size	12-pk.
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20mm Screw Cap	20 mm-400	24903
24mm Screw Cap	24 mm-400	24904
20mm Crimp Top	—	24905

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### *New, Highly Retentive HPLC Columns Designed for LC/MS*

by David Bell and Keith Duff, Senior Research Chemists

Liquid chromatography/mass spectrometry (LC/MS) has developed into one of the most powerful analytical techniques available. Although the MS can, in itself, provide enhanced selectivity, quality chromatographic separations are essential in order to take full advantage of the technique. Many column manufacturers are simply repackaging and marketing their existing, standard HPLC phases as LC/MS columns. Restek, on the other hand, is actively involved in real-world research and development of new LC/MS stationary phases and columns.



Among the advantages of LC/MS technology is an increased level of sensitivity compared to traditional ultraviolet (UV) detection. For electrospray-interfaced LC/MS systems, the sensitivity is related to the efficiency of the ionization/evaporation process. A greater percentage of organic modifier in the mobile phase improves the ionization/evaporation efficiency and thus provides increased sensitivity.

The Allure™ family of HPLC columns has been designed to provide the high retention and resolution required for successful LC/MS method development. Research efforts have concentrated on obtaining maximum retention for analytes based on their functional group, (cont. on pg. 2)

**Figure 1** LC/MS Column Selection Based on Analyte Functional Group.

High Bonding Density of Conventional HPLC Phase Chemistries

Novel Bonding Chemistries Engineered for Maximum LC/MS Sensitivity

#### Analyte Functional Group



Low Molecular Weight (MW) = <200 Daltons

High Molecular Weight (MW) = >200 Daltons

NP = Normal Phase

#### LC/MS Columns

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without compromising quality and reproducibility. The research produced three superior products—Allure™ C18, Allure™ Basix, and Allure™ Acidix columns—that target four classes of analytes as diagrammed in Figure 1 (front). Because these columns retain analytes longer than conventional columns, a higher percentage of organic modifier in the mobile phase may be employed. The result is a significant increase in MS sensitivity (Figure 2).

We also offer Ultra HPLC columns. The high-percentage carbon and phase coverage provides heightened solute retention compared to other manufacturers' columns. Additionally, the bonding chemistry of each phase is reproducible and reliable.

Try unique stationary phase chemistries for enhanced, state-of-the-art selectivity. We offer the ultimate in HPLC column quality and reproducibility, guaranteed lot-to-lot confidence, and minimal phase bleed.

For Allure™ columns with Trident™ inlet fittings and Allure™ guard cartridges, as well as a complete listing for Ultra columns, please request the **LC/MS Columns Catalog** (lit. cat. #59607\*), or visit our web site at [www.restekcorp.com](http://www.restekcorp.com).

\*International customers please request lit. cat. # 59606.

## Product Listing

### Allure™ Acidix Columns

Particle Size: 5µm	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	9162531	9162532	9162533	9162535
50mm length	9162551	9162552	9162553	9162555
100mm length	9162511	9162512	9162513	9162515
150mm length	9162561	9162562	9162563	9162565
200mm length	9162521	9162522	9162523	9162525
250mm length	9162571	9162572	9162573	9162575

### Allure™ C18 Columns

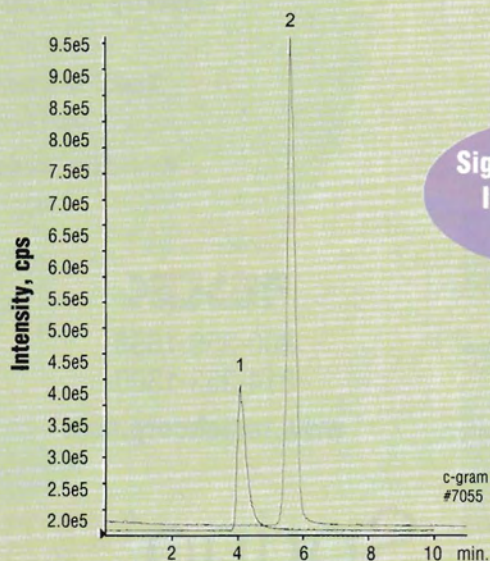
Particle Size: 5µm	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	9164531	9164532	9164533	9164535
50mm length	9164551	9164552	9164553	9164555
100mm length	9164511	9164512	9164513	9164515
150mm length	9164561	9164562	9164563	9164565
200mm length	9164521	9164522	9164523	9164525
250mm length	9164571	9164572	9164573	9164575

### Allure™ Basix Columns

Particle Size: 5µm	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	9161531	9161532	9161533	9161535
50mm length	9161551	9161552	9161553	9161555
100mm length	9161511	9161512	9161513	9161515
150mm length	9161561	9161562	9161563	9161565
200mm length	9161521	9161522	9161523	9161525
250mm length	9161571	9161572	9161573	9161575

Figure 2

Achieve 240% Signal-to-Noise Increase Using an Allure™ Basix LC/MS Column.



Signal-to-Noise  
Increase of  
240%!

#### Amitriptyline LC/MS

Mass Spectrometer: PE SCIEX API 150 EX  
LC/MS Interface: PE SCIEX TURBO IONSpray®  
Flow: 0.2mL/min.  
Injection: 25ng Amitriptyline HCl

#### 1) Column: Conventional C18, 50 x 2.1mm, 5µm

Mobile phase: 5mM NH<sub>4</sub>CH<sub>3</sub>COO, pH 4.5: ACN (65:35, v/v)  
Intensity: 288373.72 cps  
Area: 2.794 x 10<sup>5</sup> cps<sup>2</sup>  
Noise: 580.620 cps

#### 2) Column: Allure™ Basix, 50 x 2.1mm, 5µm

Mobile phase: 5mM NH<sub>4</sub>CH<sub>3</sub>COO, pH 4.5: ACN (20:80, v/v)  
Intensity: 962220.81 cps  
Area: 1.544 x 10<sup>7</sup> cps<sup>2</sup>  
Noise: 798.673 cps



# VOLATILE METHODS

## 0.45mm ID Rtx®-502.2 Column Provides Faster GC Volatile Analysis

by Christopher English, Environmental Applications Chemist

The Rtx®-502.2 column is one of the most popular choices for a long list of volatile Environmental Protection Agency (EPA) methods. An optimized 0.45mm ID Rtx®-502.2 column now provides even better resolution and faster analysis times. The Rtx®-502.2 column originally was designed to address the growing number of compounds that the EPA added to its GC volatile methods; namely the transition from Methods 502.1/503.1 (40 compounds) to Method 502.2 (60 compounds).

Although the 502.2 method has remained the same, many of the SW-846 8000-series methods have

changed the target analytes. Additionally, states have required monitoring of other compounds, such as methyl-*tert*-butyl ether (MTBE), a gas additive, and Freon®113. The most recent change is the revision of Method 8021A to Method 8021B. This newly promulgated method removes 13 compounds from the previous list—mostly the branched aromatics—and adds 10 more target analytes. Many environmental labs are interested in analyzing the 502.2 compound list, the added compounds introduced in Method 8021B, and state-regulated compounds. This results in a target list that may exceed 70 compounds.

The new 0.45mm ID Rtx®-502.2 column has excellent resolution for the volatile compounds and a 15-minute faster analysis time compared to the 0.53mm ID equivalent. Figures 1 and 2 illustrate optimized run conditions using a 75m, 0.45mm ID, 2.55µm df, Rtx®-502.2 column. By using a smaller internal diameter, a faster run is possible with enough column flow to effectively sweep the volatiles off of the trap, resulting in excellent chromatography.

### Advantages of the Rtx®-502.2 Column

The Rtx®-502.2 column is unique in that it resolves

#### Analysis Conditions for Figures 1 & 2

**Column:** 75m, 0.45mm ID, 2.55µm Rtx®-502.2 (cat.# 10986).

**Concentration:** 20ppb in 5mL of RO water (unless otherwise noted, peak 14).

**Injection:** a combination of the following reference materials was used:

502.2 Cal2000 MegaMix™ (cat.# 30431); 502.2 Calibration Mix #1A (cat.# 30439); 502.2 Internal Standard Mix #2 (cat.# 30041); 1-chloro-2-fluorobenzene (cat.# 30040); 4-bromochlorobenzene (cat.# 30230); 2-chlorethyl vinyl ether (cat.# 30265); 1,4-dichlorobutane (cat.# 30227); MTBE (cat.# 30402); and custom mixtures of Freon® 113, allyl chloride, chloroprene, and benzyl chloride.

**Purge & Trap concentrator:** Tekmar LSC-3000

purge and trap; **Trap:** VOCARB™ 3000; **Purge:**

11 min. @ 40mL/min.; **Dry purge:** 1 min. @

40mL/min. (MCS off); **Desorb preheat:** 245°C;

**Desorb:** 250°C for 2 min.; **Bake:** 260°C for

8 min.

**GC:** Finnigan 9001

**Oven temp:** 35°C (hold 6 min.) to 115°C @ 11°C/min. (hold 7 min.) to 130°C @ 7°C/min.

(no hold), to 220°C @ 9.2°C/min. (hold 4 min.);

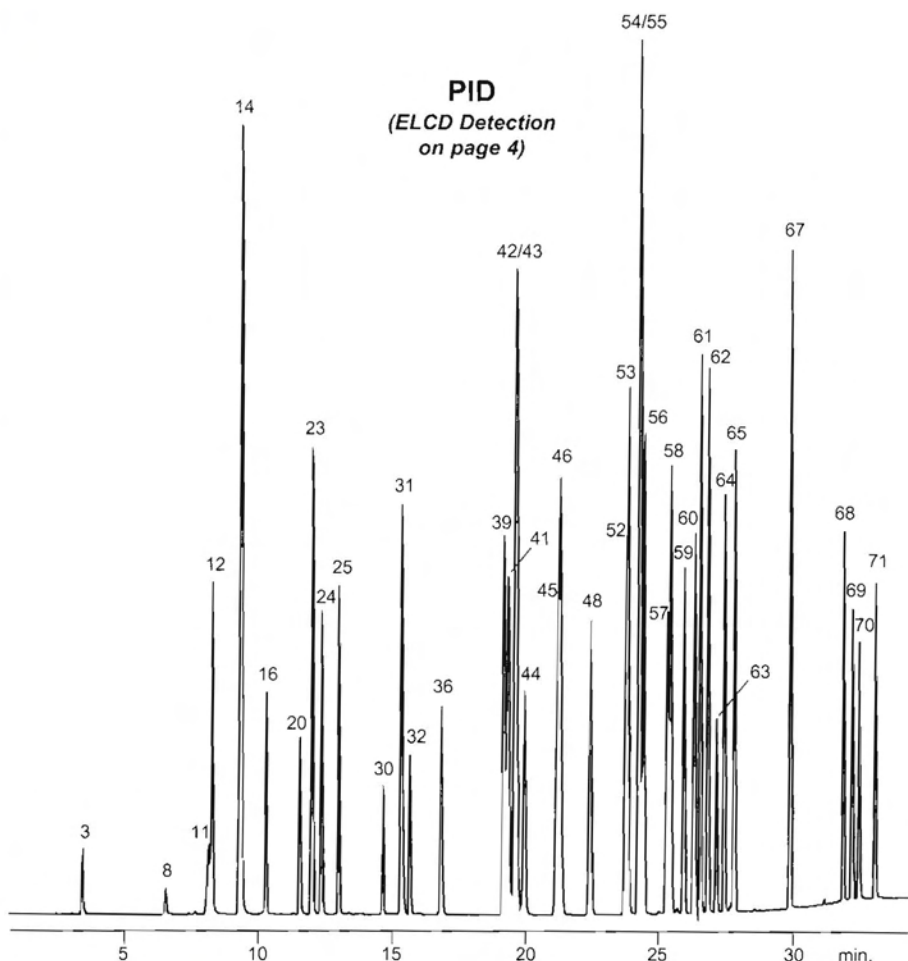
**Carrier gas:** helium (9mL/min.); **Detectors:**

µGold Tandem PID/HALL; **PID:** makeup 7mL/min., purge 7mL/min. set @ 0.35mV, base temp. 200°C; **ELCD:** (Hall 2000) Rxn gas 25mL/min., Rxn temp. 940°C, propanol flow 470µL/min.

**Acknowledgement:** Finnigan 9001 GC, µGold Tandem Photoionization Detector & Hall 2000 Detector provided courtesy of ThermoQuest/CE Instruments.

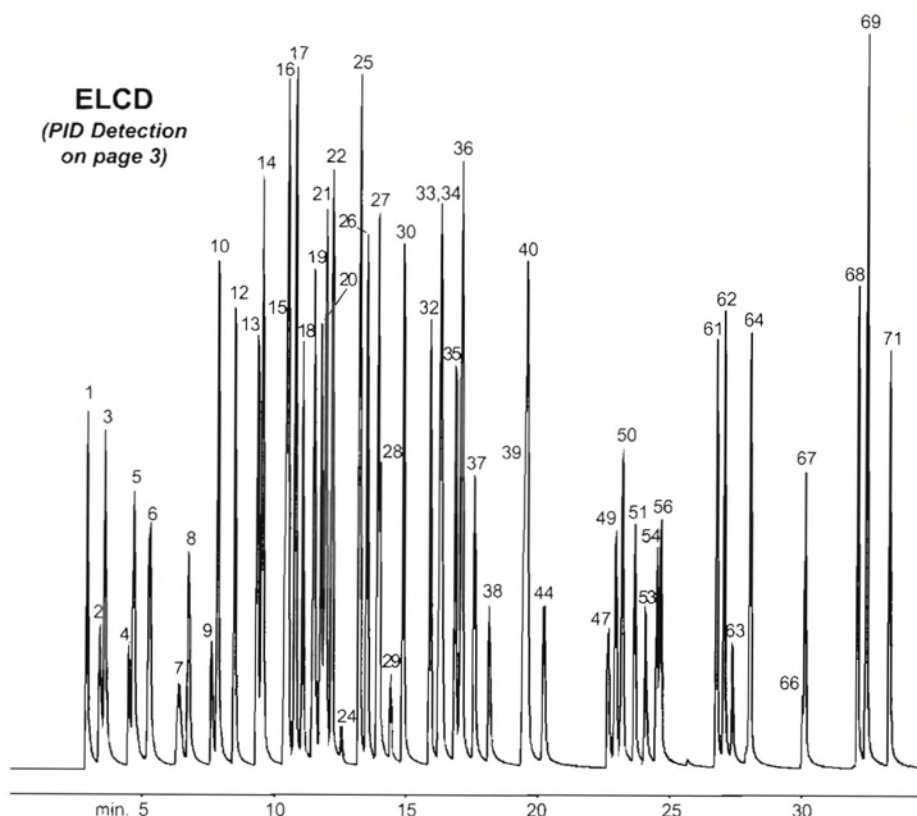
**Figure 1**

The New 0.45mm ID Rtx®-502.2 Column Provides a 15-Minute Faster Analysis Time Than the 0.53mm ID Column.





**Figure 2** The New 0.45mm ID Rtx®-502.2 Column Provides a 15-Minute Faster Confirmational Analysis Than the 0.53mm ID Column.



MTBE, Freon®113, and the trihalomethanes (THMs) from Method 502.2 analytes. These compounds are commonly found in "real world" samples. MTBE in groundwater is considered widespread and is currently regulated in 15 states across the country, with others expected to follow. Several states have added MTBE to their Total Petroleum Hydrocarbon (TPH) methods.

Freon®113 is used in industrial air conditioners and for cleaning metal surfaces. In the environment, it is a contributor of chlorine atoms to the atmosphere, which ultimately damages the earth's ozone layer. Freon®113 is still produced in 25 states nationwide. Both of these early eluting compounds have characteristically broad peaks when analyzed by purge and trap.

The THMs are disinfectant byproducts and are found in drinking water, which accounts for half of the workload in the gas chromatography (GC) lab. Column selection becomes very important when analyzing these added analytes along with the regular EPA volatile compounds. Figures 1 and 2 were achieved using a photoionization detector (PID) and the electrolytic conductivity detector (ELCD) in series. Additional compounds from Method 8021B,

such as allyl chloride, chloroprene, 2-chloroethylvinylether, and benzyl chloride also are well resolved. Because these were analyzed under ambient purge conditions, the alcohols were not added as part of our target list. Suggested surrogates for the Rtx®-502.2 and Rtx®-1 column pair include fluorobenzene, 1-chloro-2-fluorobenzene, and 1,4-dichlorobutane. These performance-monitoring compounds are almost baseline resolved and will produce excellent recoveries. The outstanding thermal stability of the Rtx®-502.2 column minimizes bleed, which improves PID and ELCD performance and reduces instrument downtime. These bonded phase columns can be solvent rinsed if contaminated and have long lifetimes.

For more information on the new 0.45mm ID Rtx®-502.2, including chromatograms of confirmational analysis on the Rtx®-1, request lit. cat.# 59808. Call Technical Service at 800-356-1688, ext. 4, or contact your local Restek representative.

#### Peak List for Figures 1 & 2

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. Freon® 113
8. 1,1-dichloroethene
9. allyl chloride
10. methylene chloride
11. methyl-*tert*-butyl-ether
12. *trans*-1,2-dichloroethene
13. 1,1-dichloroethane
14. chloropropene (40ppb)
15. 2,2-dichloropropane
16. *cis*-1,2-dichloroethene
17. chloroform
18. bromochloromethane
19. 1,1,1-trichloroethane
20. 1,1-dichloropropene
21. carbon tetrachloride
22. 1,2-dichloroethane
23. benzene
24. fluorobenzene (surrogate)
25. trichloroethene
26. 1,2-dichloropropane
27. bromodichloromethane
28. dibromomethane
29. 2-chloroethylvinylether
30. *cis*-1,3-dichloropropene
31. toluene
32. *trans*-1,3-dichloropropene
33. 1,1,2-trichloroethane
34. 2-bromo-1-chloropropane (surrogate)
35. 1,3-dichloropropane
36. tetrachloroethene
37. dibromochloromethane
38. 1,2-dibromoethane
39. chlorobenzene
40. 1,1,1,2-tetrachloroethane
41. ethyl benzene
42. *m*-xylene
43. *p*-xylene
44. 1-chloro-2-fluorobenzene (surrogate)
45. *o*-xylene
46. styrene
47. bromoform
48. isopropyl benzene
49. 1,4-dichlorobutane (surrogate)
50. 1,1,2,2-tetrachloroethane
51. 1,2,3-trichloropropane
52. *n*-propyl benzene
53. bromobenzene
54. 2-chlorotoluene
55. 1,3,5-trimethylbenzene
56. 4-chlorotoluene
57. *tert*-butylbenzene
58. 1,2,4-trimethylbenzene
59. *sec*-butylbenzene
60. *p*-isopropyl toluene
61. 1,3-dichlorobenzene
62. 1,4-dichlorobenzene
63. benzyl chloride
64. *n*-butylbenzene
65. 1,2-dichlorobenzene
66. 1,2-bromo-3-chloropropane
67. 4-bromo-1-chlorobenzene (surrogate)
68. 1,2,4-trichlorobenzene
69. hexachlorobutadiene
70. naphthalene
71. 1,2,3-trichlorobenzene



# 5 Faster GC Volatile Analysis

## Product Listing

### Rtx®-502.2 Fused Silica Columns

ID	df (µm)	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	10915	10916	-----	-----
0.32mm	1.80	10919	10920	-----	10921
0.45mm—NEW!	2.55	-----	-----	10986	-----
0.53mm	3.00	10908	10909	-----	10910

ID	df (µm)	20-Meter	40-Meter
0.18mm	1.00	40914	40915

Most column configurations are available as metal MXT® columns. See pg. 46 of the 1999 Annual Product Guide.

### 502.2 CAL2000 MegaMix™ Mixture

benzene	2,2-dichloropropane
bromobenzene	1,1-dichloropropene
bromochloromethane	cis-1,3-dichloropropene
bromodichloromethane	trans-1,3-dichloropropene
bromoform	ethylbenzene
n-butylbenzene	hexachlorobutadiene
sec-butylbenzene	isopropylbenzene
tert-butylbenzene	p-isopropyltoluene
carbon tetrachloride	methylene chloride
chlorobenzene	naphthalene
chloroform	n-propylbenzene
2-chlorotoluene	styrene
4-chlorotoluene	1,1,1,2-tetrachloroethane
dibromochloromethane	1,1,2,2-tetrachloroethane
1,2-dibromo-3-chloropropane	tetrachloroethene
1,2-dibromoethane	toluene
dibromomethane	1,2,3-trichlorobenzene
1,2-dichlorobenzene	1,2,4-trichlorobenzene
1,3-dichlorobenzene	1,1,1-trichloroethane
1,4-dichlorobenzene	1,1,2-trichloroethane
1,1-dichloroethane	trichloroethene
1,2-dichloroethane	1,2,3-trichloropropane
1,1-dichloroethene	1,2,4-trimethylbenzene
cis-1,2-dichloroethene	1,3,5-trimethylbenzene
trans-1,2-dichloroethene	m-xylene
1,2-dichloropropane	o-xylene
1,3-dichloropropane	p-xylene

In P&T methanol, 1mL/ampul

200µg/mL Ea.	Each	5-pk.	10-pk.
	30432	30432-510	-----
w/ data pack	30432-500	30432-520	30532
2000µg/mL Ea.	Each	5-pk.	10-pk.
	30431	30431-510	-----
w/ data pack	30431-500	30431-520	30531

Custom single or multiple component mixes are available.  
Please call for a quote.

### 502.2 Calibration Mix #1A

bromomethane	dichlorodifluoromethane
chloroethane	trichlorofluoromethane
chloromethane	vinyl chloride

In P&T methanol, 1mL/ampul

200µg/mL Ea.	Each	5-pk.	10-pk.
	30439	30439-510	-----
w/ data pack	30439-500	30439-520	30539
2000µg/mL Ea.	Each	5-pk.	10-pk.
	30042	30042-510	-----
w/ data pack	30042-500	30042-520	30142

### 502.2 Internal Standard Mix #1

1-chloro-2-fluorobenzene  
2000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30040	30040-510	-----
w/ data pack	30040-500	30040-520
		30140

### 502.2 Internal Standard Mix #2

2-bromo-1-chloropropane  
fluorobenzene  
2000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30041	30041-510	-----
w/ data pack	30041-500	30041-520
		30141

### VOA Individual Standards for EPA Methods, (2000µg/mL ea. in P & T Methanol)

1mL/ampul	Ind.	Ind. w/Data Pack	5-pk.	5-pk. w/data pack	w/data pack
4-bromochlorobenzene	30230	30230-500	30230-510	30230-520	30330
2-chloroethyl vinyl ether	30265	30265-500	30265-510	30265-520	30365
1,4-dichlorobutane	30227	30227-500	30227-510	30227-520	30327
MTBE	30402	30402-500	30402-510	30402-520	30502



# USP <467> GC Analysis of Organic Volatile Impurities According to USP <467>

by Christopher Cox, Senior R&D Chemist

The analysis of residual solvents using US Pharmacopoeia (USP) Method <467> presents many technical challenges to the pharmaceutical analyst with limited gas chromatography (GC) experience. Some of these challenges include poor injection reproducibility at high temperatures, poor reproducibility of peak area response, and difficult standard preparations. Issues related to USP <467> have been addressed in the Pharmacopoeial Forum, including sample introduction, standards preparation, detection limits, sample amounts, and column selection. We would like to further clarify these issues in the following article.

Methods I and V of USP <467> are the most commonly used methods for OVI analysis. One of the drawbacks associated with these methods is the use of aqueous injections for sample introduction. High injection port temperatures can produce large expansion volumes for injections of water as low as 1 µL. When the expansion volume of the sample exceeds the buffer volume of the injection port liner,

backlash can occur and some sample can be lost through the septum purge line. Because of backlash, injection reproducibility is poor at high temperatures. Originally, injection port temperatures were specified to be 180°C for Method I, and 140°C

For more information and additional chromatograms, call 800-356-1688 or 814-353-1300, ext. 4 for "GC Analysis of Organic Volatile Impurities According to USP <467>" (Lit. Cat.# 59577).

for Method V. Data supplied by Bergren and Foust demonstrated that a decrease in injection port temperature from 180°C to 70°C yielded lower relative standard deviations for peak area response on replicate injections.<sup>1</sup> Revisions have been made

to Method I to lower the injection port temperature to 70°C, but the injection port temperature for Method V has not been revised as of the publication of this article.

Poor reproducibility of peak area response is also related to the amount of analyte reaching the column. The response of chloroform on an FID is very poor. At the concentrations originally specified for the standard solutions, the chloroform response cannot be reproducibly detected above the baseline noise. Krasowski et al.<sup>2</sup> proposed two solutions that previously have been mentioned in the *In Process Revision*<sup>3</sup> comments in the Pharmacopoeial Forum. The answer was to double the concentration of OVIs in the standard solution, and double the amount of bulk pharmaceutical used to produce the test solution. This modification increased the on-column concentration and produced a more consistent peak area response for each analyte. USP also increased the allowable relative standard deviation to 15% after finding that 10% was too demanding.

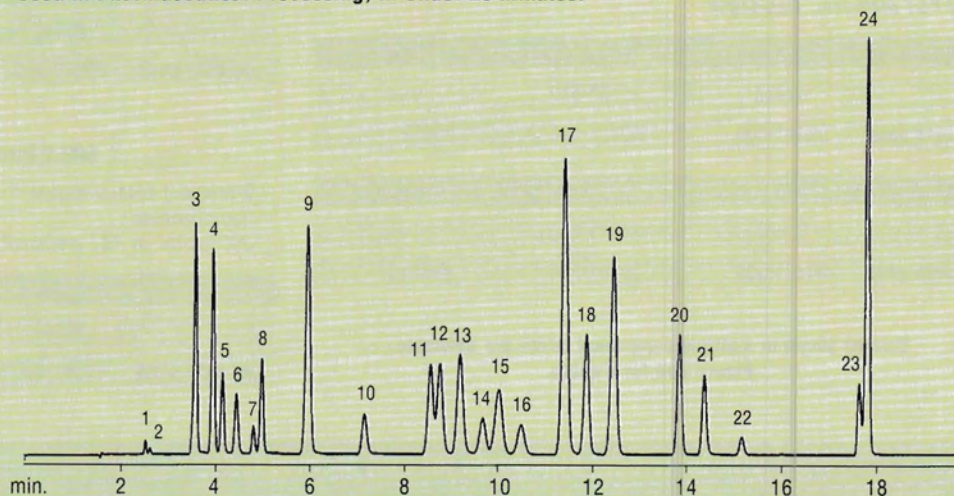
**Figure 1**

The Rtx®-G43 Column Provides Good Resolution of Solvents Commonly Used in Pharmaceutical Processing, in Under 20 Minutes.

#### Run Conditions:

Rtx®-G43 (30m, 0.53mm ID, 3.0µm) with 5m phenylmethyl Integra-Guard™ (cat.# 16085-126). Headspace injection of 24 common residual solvents for pharmaceutical processing. Prepared to equal approximately 500ppm in the bulk pharmaceutical. Samples shaken and heated at 90°C for 15 minutes, 1mL headspace injection. **Oven temp.:** 35°C (hold 10 min.) to 100°C @ 5°C/min., to 240°C @ 25°C/min. (hold 5 min.); **Inj./det. temp.:** 220°C/240°C; **FID sensitivity:** 1.25 x 10<sup>-11</sup> AFS; **Carrier gas:** helium, 35cm/sec. set @ 35°C; **Split ratio:** 2:1.

**Acknowledgement:** Thermoquest Trace GC and HS2000 Autosampler provided courtesy of ThermoQuest/CE Instruments.



#### Peak List for Figure 1

- |                       |                       |                         |                           |                        |                 |
|-----------------------|-----------------------|-------------------------|---------------------------|------------------------|-----------------|
| 1. ethylene oxide     | 5. acetone            | 9. n-hexane             | 13. tetrahydrofuran       | 17. benzene            | 21. n-butanol   |
| 2. methanol           | 6. isopropanol        | 10. n-propanol          | 14. chloroform            | 18. 1,2-dichloroethane | 22. 1,4-dioxane |
| 3. diethyl ether      | 7. acetonitrile       | 11. methyl ethyl ketone | 15. 1,1,1-trichloroethane | 19. heptane            | 23. pyridine    |
| 4. 1,1-dichloroethene | 8. methylene chloride | 12. ethyl acetate       | 16. carbon tetrachloride  | 20. trichloroethylene  | 24. toluene     |



In the Fifth Supplement, USP-NF<sup>6</sup>, the requirement to analyze for trace levels of ethylene oxide was removed from Method <467>, and a test for ethylene oxide now only is performed when specified in the individual monograph.

In the May/June 1993 edition of Pharmacopoeial Forum<sup>5</sup>, additional method modifications were made. The limit test concentration for methylene chloride was increased from 100ppm to 500ppm. The solvent used to prepare stock calibration standards also was changed from dimethyl sulfoxide to methanol.

Furthermore, USP has simplified the standard preparation procedure. The solubility for OVIs in water is very poor and direct dissolution of these compounds in water is difficult. Standard stability and lifetime can be improved by using stock solutions of the OVIs prepared in dimethyl sulfoxide, and then making dilutions of the stock standard into water to produce working standards. Comments in the September/October 1992 Pharmacopoeial Forum<sup>6</sup> propose the use of dimethyl sulfoxide as the solvent for stock standard, but this has not been approved as of the date of this publication.

Choosing a capillary column to perform OVI analysis also has been a subject for debate. Method I uses a capillary column interfaced with an FID, and sample introduction via a direct injection of aqueous sample. The column specified is a 30m x 0.53mm ID x 3µm, 5% phenyl/95% methyl polysiloxane (G27) column (Restek's Rtx®-G27 column). When Method I originally was published, a 1.5 resolution factor

was included in the system suitability parameters. Many analysts have difficulty achieving baseline resolution between trichloroethylene and 1,4-dioxane. The 1.5 resolution factor was reduced to 1.0 in the Fifth Supplement of Method I, in order to make the system suitability requirements easier to achieve.

Method V also was introduced in the Fifth Supplement and incorporated the use of a 30m x 0.53mm ID x 3µm, 6% cyanopropylphenyl/94% dimethylpolysiloxane (G43) column (Restek's Rtx®-G43 column) as an alternative to the 5% phenyl/95% methyl polysiloxane column. Figure 1 shows the use of an Rtx®-G43 column for the analysis of OVIs. Baseline resolution of all the compounds is obtained and the resolution criteria of 3.0 is easily met on the Rtx®-G43 column.

In the Sixth Supplement, USP-NF<sup>7</sup>, USP <467> changes were made to overcome the difficulties resulting from unregulated solvents coeluting with regulated solvents, thereby over-representing their concentration using GC/FID methods. GC/MS, or a second validated column with a different stationary phase, may be used to confirm the presence of the

coeluting unregulated solvent and report the correct concentration of regulated solvent.

In addition to giving superior resolution for the OVIs listed in USP <467>, the Rtx®-G43 column also shows improved performance for analyzing other commonly used solvents, including many listed by the International Conference on Harmonization of Technical Requirements for Registration of Pharmaceutical for Human Use, Impurities: Guidelines for Residual Solvents.<sup>8</sup>

In a review of the current status of USP <467><sup>9</sup>, the USP is advocating the conversion of monograph requirements from Method I to Method V, to allow the use of the cyanopropyl stationary phase for better resolution. They are also anticipating that more laboratories will begin to use the static headspace technique as a means of replacing the direct injection of aqueous samples and its associated problems. The European Pharmacopoeia lists only static headspace methods for the testing of OVIs.<sup>10</sup> These changes, along with the current revisions to USP <467>, should result in an easier to use and more reproducible method for the future.

## Product Listing

### Rtx®-G27 Integra-Guard™ Column (with built-in 5m phenylmethyl guard column)\* (5% phenyl/95% methyl polysiloxane)

Dimensions	Temp. Limits	cat.#
30m x 0.53mm ID x 5µm	-60 to 270/290°C	10279-126

### Rtx®-G43 Integra-Guard™ Column (with built-in 5m phenylmethyl guard column)\* (6% cyanopropylphenyl/94% dimethyl polysiloxane)

Dimensions	Temp. Limits	cat.#
30m x 0.53mm ID x 3µm	-20 to 240°C	16085-126

\*Restek is the only capillary column manufacturer that offers Integra-Guard™ columns. These innovative columns come with a built-in guard column that eliminates the need for a connector and assures a leak-free connection between the guard column and analytical column. In addition to capillary columns, Restek offers a variety of calibration standards, headspace vials, and inlet liners for this application. Request Applications Note "GC Analysis of Organic Volatile Impurities According to USP <467>" (lit. cat.# 59577) for a complete product listing.

### USP 467 Calibration Mixture #3

benzene	100µg/mL
chloroform	50
1,4-dioxane	100
methylene chloride	500
trichloroethene	100
Prepared in DMSO, 1mL/ampul	

Each: cat.# 36004  
10-Pack: cat.# 36104

Restek offers a complete line of  
**reference materials for USP <467> analysis,**  
and other OVI Methods. Custom standards are also available.

Call 800-356-1688, ext. 4, for more information or contact your  
local Restek representative.

## References

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8. "ICH Harmonized Tripartite Guideline, Impurities: Guideline for Residual Solvents," *The Fourth International Conference on Harmonization*, July 17, 1997.
9. V. Gray, "Organic Volatile Impurities Testing Initiative: An Update," *Pharmacopoeial Forum*, March/April 1992, Vol. 18, No. 2, p. 3205.
10. *European Pharmacopoeia*, Supplement 1999, pp. 14-15, 208.

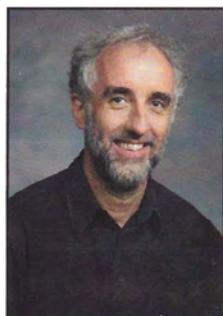
References not available from Restek.



# KONI'S KORNER

## *The Value of Education*

by Dr. Konrad Grob, Contributing Author



In my last Korner, I concluded that only an education and qualification system could prevent chromatography from further decline. Instead of devoting time and effort to force better chromatography

through quality management schemes, validation, accreditation, and bureaucratic piles of paper, I suggested that institutions invest in improving the quality of their employees. In this way, both the

employees and the employers share the responsibility for improvement. Employers should realize that knowing how to manage a crimper for closing autosampler vials (despite what some instrument vendors may claim) is not "all" it takes to make a gas chromatography (GC) laboratory successful. At the same time, if analysts want to be considered valuable assets, then they should be ready to take an examination that affirms their education and training. How much education is necessary?

I received an overwhelming response to my article. Almost all of which confirmed my conclusion. For the sake of argument, though, I would have liked to hand over the microphone to somebody who

disagrees. However, without refuting my point, I will take a moment to gain a perspective on the questions of 1) How simple is GC really? and 2) How much education is necessary? When observed from a safe distance, the work of a GC analyst appears simple. A gas chromatographer performing routine analyses should be able to help troubleshoot when results are not appropriate. The analyst should, in addition, be able to "rapidly" analyze x in sample y, and know how to select the right column, the injection technique, and all the many parameters finally determining whether the analysis will be successful. If capable of developing methods, the analyst needs to overview the possibilities and the problems to be expected; the clever choosing of strategies, tools, and conditions that may save trouble over years and reduce the time needed per sample by a factor of more than two.

No doubt analysts who are able to answer common GC questions achieve more reliable results because they can find the pitfalls. While other people waste several days because an analysis does not turn out adequately, these analysts find tests that can rapidly localize the problem. They know beforehand that aqueous samples are more difficult to analyze and should be injected in small volumes.

In industrial countries, every working day costs around \$1,000. Eliminating three days of expense per month by improved troubleshooting saves \$3,000 for that month. You easily can save several days per month by using better methods or improving performance of given methods. A knowledgeable gas chromatographer can prevent visits by the service engineer and avoid other delays disturbing production or delivery. If half of the profit generated by more competent work goes to shareholders, the analyst's salary should increase by \$3,000 at least.

I would like to substantiate my call for education by asking some questions about a specific technique in capillary GC: split injection. Test your knowledge; see if you think more education is required for efficient and appropriate GC analysis. A lab supervisor who deals with GC should be able to answer ALL of the questions on page 9. Please review the answers; if you have difficulty with any of them, perhaps a convenient refresher course would be helpful.

## *Coming to a Location Near You...*

### **Comprehensive GC Seminar**

by Andy Schuyler, Seminar Coordinator

This is a great opportunity to learn tips for saving time and money, such as when columns can be operated above their maximum temperatures and how to convert a packed column GC to a capillary GC in under 5 minutes! The seminar will also cover the sample/column/GC system—from sample introduction to detection—and will extensively survey proper injection techniques, column selection, column installation, system maintenance, and detector operation.

If you have trouble with the questions on page 9, help is available! As a direct response to your interest in improved GC education, "Restek On-the-Road" presents its new "Comprehensive GC for the Practicing Chromatographer Seminar Series." The new seminars are a full-day course, presented in an engaging multimedia format that teaches key chromatographic concepts, tricks of the trade, and little-known secrets that are of benefit to the novice or the seasoned veteran. There is no sales pitch presented during the seminar, just the facts on how to make your chromatography results better.

Restek guarantees that after you apply what you have learned in our seminar, the savings you

create in the first month alone will exceed the registration fee!

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# Do you need a brush-up on your chromatography basics?

*Take this simple test to see if you would benefit from Restek training.*

## Concept Questions

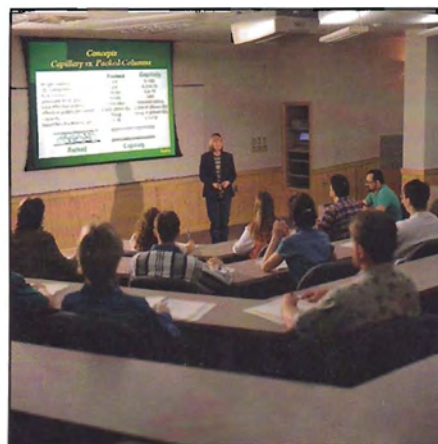
1. Describe split injection and what determines the split ratio?
2. Name the principal parts of a split injector.
3. Describe two concepts of pneumatic systems controlling the gas supply and split flow rate.
4. Does an increase of the split ratio increase or decrease peak areas?
5. What are the two steps required to adjust the split ratio?
6. Does the split ratio depend on a) the injector temperature or b) the column temperature?
7. Do autosamplers produce the same results as manual injections (within the normal standard deviation)?
8. What are the problems resulting from partial sample evaporation in the syringe needle?
9. Why is an empty, straight liner usually used for manual injections?
10. If performed manually: does a 1 $\mu$ L injection produce twice as large peaks as a 0.5 $\mu$ L injection, to within 10%?

## Questions on Selecting Conditions

11. When does the liner need to be packed?
12. What are the factors determining the best sample volume for split injection?
13. What is the best injector temperature?
14. What is the effect of solvent recondensation in the column inlet?
15. What should you be careful about when using split injection with the external standard methods?
16. What length of inserted syringe needle is suitable for split injection?

## Troubleshooting Questions

17. What can cause absolute peak areas to have relative standard deviations exceeding 10%?
18. What is "non-linear splitting" and how can this disturb quantitative results?
19. How do you test for a deviation of the effective split ratio?
20. How do you test for discrimination by losses inside the syringe needle?
21. Does a minor leak in the septum have an effect on quantitative analysis?
22. What is the effect of a leaking fitting between the liner and the injector body?
23. If the plunger of the syringe moves with friction and the manual injection is slower, will it have an effect on quantitative results?



## We'll Bring the Seminar to You!

"Restek On-the-Road" presents its new "Comprehensive GC for the Practicing Chromatographer Seminar Series" at 41 locations nationwide in 1999. However if these aren't convenient for you, we'll bring the seminar to you! Seminars at your facility are presented by experienced chromatographers and provide customized instruction. But don't take our word for it...

“As the Training Coordinator for the University of Iowa Hygienic Laboratory, I worked with Restek to coordinate a Gas Chromatography Workshop at our facility. The workshop was very informative and was well received by all. I would recommend this training to anyone using a GC—regardless of education or experience level.”

- Beth Hochstedler  
University of Iowa Training Coordinator

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Call us at 800-356-1688, ext. 4.  
We'll fax or email you the answers.



# REFERENCE MATERIALS

## CLP Volatiles OLM 04.1

by Eric Steindl, Analytical Reference Materials Product Line Manager

### Standards for the EPA Superfund Contract Lab Program (CLP):

- ✓ Meets latest Statement of Work.
- ✓ Fewest number of calibration solutions possible.
- ✓ Immediately available.
- ✓ Maximum shelf life.

### CLP 04.1 VOA CAL2000 MegaMix™

2000µg/mL each in P & T methanol  
benzene  
bromodichloromethane  
bromoform  
carbon disulfide  
carbon tetrachloride  
chlorobenzene  
chloroform  
1,2-dibromo-3-chloropropane  
cyclohexane  
dibromochloromethane  
1,2-dibromoethane  
1,2-dichlorobenzene  
1,3-dichlorobenzene  
1,4-dichlorobenzene  
1,1-dichloroethane  
1,2-dichloroethane  
1,1-dichloroethene  
*cis*-1,2-dichloroethene  
*trans*-1,2-dichloroethene  
1,2-dichloropropane  
*cis*-1,3-dichloropropene  
*trans*-1,3-dichloropropene  
ethylbenzene  
isopropylbenzene  
methyl acetate  
methylcyclohexane  
methylene chloride  
styrene  
methyl-*tert*-butyl-ether (MTBE)  
1,1,2,2-tetrachloroethane  
tetrachloroethene  
toluene  
1,2,4-trichlorobenzene  
1,1,1-trichloroethane  
1,1,2-trichloroethane  
trichloroethylene  
1,1,2-trichloro-1,2,2-trifluoroethane  
*m*-xylene  
*o*-xylene  
*p*-xylene

Each	5-pk.	10-pk.
30456	30456-510	-----
w/ data pack		
30456-500	30456-520	30556

### VOA Calibration Mix #1

5000µg/mL each in P & T methanol  
acetone  
2-butanone  
2-hexanone  
4-methyl-2-pentanone

Each	5-pk.	10-pk.
30006	30006-510	-----
w/ data pack		
30006-500	30006-520	30106

### 502.2 Calibration Mix #1

2000µg/mL each in P & T methanol  
bromomethane  
chloroethane  
chloromethane  
dichlorodifluoromethane  
trichlorofluoromethane  
vinyl chloride

Each	5-pk.	10-pk.
30042	30042-510	-----
w/ data pack		
30042-500	30042-520	30142

### CLP 04.1 VOA Internal Standard/SMC Spike Mix

2500µg/mL each in P & T methanol  
bromochloromethane  
1,4-difluorobenzene  
chlorobenzene-d5  
4-bromofluorobenzene  
1,2-dichloroethane-d4  
toluene-d8

Each	5-pk.	10-pk.
30457	30457-510	-----
w/ data pack		
30457-500	30457-520	30557

### VOA Surrogate Spike Mix

(System Monitoring Compounds Spike Mix)

2500µg/mL each in P & T methanol  
4-bromofluorobenzene  
1,2-dichloroethane-d4  
toluene-d8

Each	5-pk.	10-pk.
30004	30004-510	-----
w/ data pack		
30004-500	30004-520	30104

### VOA Matrix Spike Mix

2500µg/mL each in P & T methanol  
benzene  
chlorobenzene  
1,1-dichloroethene  
toluene  
trichloroethene

Each	5-pk.	10-pk.
30005	30005-510	-----
w/ data pack		
30005-500	30005-520	30105

### VOA Screening Mix #1

1000µg/mL each in P & T methanol  
benzene  
ethylbenzene  
toluene  
*o*-xylene  
*p*-xylene

Each	5-pk.	10-pk.
30001	30001-510	-----
w/ data pack		
30001-500	30001-520	30101

### VOA Screening Mix #2

1000µg/mL each in P & T methanol  
n-dodecane  
n-nonane

Each	5-pk.	10-pk.
30002	30002-510	-----
w/ data pack		
30002-500	30002-520	30102

### VOA Tuning Compound

5000µg/mL in P & T methanol  
4-bromofluorobenzene

Each	5-pk.	10-pk.
30003	30003-510	-----
w/ data pack		
30003-500	30003-520	30103

### VOA Internal Standard Mix

2500µg/mL each in P & T methanol  
bromochloromethane  
chlorobenzene-d5  
1,4-difluorobenzene

Each	5-pk.	10-pk.
30011	30011-510	-----
w/ data pack		
30011-500	30011-520	30111

### CLP 04.1 VOA Kit #1

Contains 1mL each of:

VOA Screening Mix #1  
VOA Screening Mix #2  
VOA Tuning Compound  
VOA Surrogate Spike (System Monitoring Compounds (SMC))  
VOA Matrix Spike Mix  
VOA Calibration Mix #1 (ketones)  
VOA Internal Standard Mix  
502.2 Calibration Mix #1 (gases)  
CLP 04.1 VOA CAL2000 MegaMix™

Restek Cat. #

30001  
30002  
30003  
30004  
30005  
30006  
30011  
30042  
30456

Each	Ea. w/Data Pack
30458	30458-500

### CLP 04.1 VOA Kit #2

Contains 1mL each of:

VOA Screening Mix #1  
VOA Screening Mix #2  
VOA Tuning Compound  
VOA Matrix Spike Mix  
VOA Calibration Mix #1 (ketones)  
502.2 Calibration Mix #1 (gases)  
CLP 04.1 VOA CAL2000 MegaMix™  
VOA Internal Standard/SMC Mix

Restek Cat. #

30001  
30002  
30003  
30005  
30006  
30042  
30456  
30457

Each	Ea. w/Data Pack
30459	30459-500

### CLP 04.1 VOA Kit #3

Contains 1mL each of:

VOA Calibration Mix #1 (ketones)  
502.2 Calibration Mix #1 (gases)  
CLP 04.1 VOA CAL2000 MegaMix™

Restek Cat. #

30006  
30042  
30456

Each	Ea. w/Data Pack
30460	30460-500



# SILCOSTEEL® TREATMENT

## Silcosteel®-Treated Tubing & Fittings

by Gary Barone, Metals Passivation Group Product Line Manager

The inertness and flexibility of Silcosteel®-treated tubing makes it ideal for sample transfer lines. In fact, Restek first developed this inert, deactivated stainless steel tubing specifically for transfer lines used in purge-and-trap systems. Our application chemists frequently found that standard fused silica lines became brittle and chemically active after repeated cycling. This often led to extended down-time of the instruments. Because we are in the business of developing new phases and applications, we needed better durability and performance from our transfer lines; the kind that Silcosteel®-coated tubing provides.

Today Restek has expanded the Silcosteel®-treated product line to include a wide variety of tubing and fittings for many process and lab instruments: purge-and-trap transfer lines, heated headspace transfer lines, sample loops, sample storage vessels, valving,

and more. Restek has been working extensively with instrument manufacturers to incorporate Silcosteel®-treated components to improve system performance.

When peak performance and inertness are required for your analysis, use Silcosteel®-coated tubing and fittings.

*Silcosteel®-Treated  
Tubing & Fittings are  
Ideal for Transfer Lines  
from Purge-and-Trap  
and Headspace*

### Silcosteel®-Treated Welded/Drawn 304\* Grade Stainless Steel Tubing

ID, OD	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
0.011" ID (0.28mm ID), 0.022" OD (0.56mm OD) <b>cat.# 20590</b>	ft.	ft.	ft.	ft.
0.021" ID (0.53mm ID), 0.029" OD (0.74mm OD) <b>cat.# 20591</b>	ft.	ft.	ft.	ft.
0.010" ID (0.25mm ID), 1/16" OD (1.59mm OD) <b>cat.# 20592</b>	ft.	ft.	ft.	ft.
0.020" ID (0.51mm ID), 1/16" OD (1.59mm OD) <b>cat.# 20593</b>	ft.	ft.	ft.	ft.
0.030" ID (0.76mm ID), 1/16" OD (1.59mm OD) <b>cat.# 20594</b>	ft.	ft.	ft.	ft.
0.040" ID (1.02mm ID), 1/16" OD (1.59mm OD) <b>cat.# 20595</b>	ft.	ft.	ft.	ft.
0.085" ID (2.16mm ID), 1/8" OD (3.18mm OD) <b>cat.# 20596</b>	ft.	ft.	ft.	ft.
0.210" ID (5.33mm ID), 1/4" OD (6.35mm OD) <b>cat.# 20597</b>	ft.	ft.	ft.	ft.

### Silcosteel®-Treated Seamless 316\* Grade Stainless Steel Tubing

0.055" ID (1.40mm ID), 1/8" OD (3.18mm OD) <b>cat.# 20598</b>	ft.	ft.	ft.	ft.
0.180" ID (4.57mm ID), 1/4" OD (6.35mm OD) <b>cat.# 20599</b>	ft.	ft.	ft.	ft.

\*The surface properties of 304 and 316 stainless steel are virtually indistinguishable when Silcosteel®-treated.

### Tees



sizes	cat.#	
1/16"	20513	ea.
1/8"	20514	ea.
1/4"	20515	ea.

### Unions



sizes	cat.#	
1/16"	20510	ea.
1/8"	20511	ea.
1/4"	20512	ea.

### Reducing Fittings



sizes	cat.#	
1/16" to 1/8"	20519	ea.
1/16" to 1/4"	20520	ea.
1/8" to 1/4"	20521	ea.

### Elbows



sizes	cat.#	
1/16"	20516	ea.
1/8"	20517	ea.
1/4"	20518	ea.

### Zero Dead Volume Unions



sizes	cat.#	
1/16"	20580	ea.
1/8"	20582	ea.

### Zero Dead Volume Tees



sizes	cat.#	
1/16"	20581	ea.
1/8"	20583	ea.



# INLET LINERS

## What Type of Inlet Liner is Best for My Analysis?

by Brad Righnour, GC Accessories Product Line Manager

With so many different inlet liner designs and deactivation chemistries available, how do you determine which one is best suited for your analysis? Each liner geometry offers the analyst a unique sample flow, from the liner to the analytical column, through cups, cyclos, and packings designed in the inner bore of the liner. How does each design affect sample flow? Which deactivation chemistry is best for your particular analysis?

Let's look at these questions to determine the answers to liner geometry selection and deactivation.

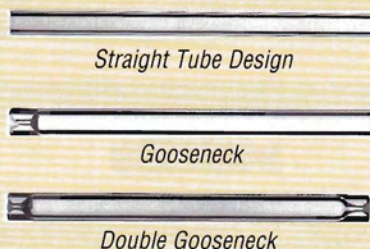
### Splitless Inlet Liners (Figure 1)

Splitless liners are designed to hold the sample in the liner from between 0.5 and 2.0 minutes. A large surface area is not critical for splitless injections.

It is common to use packing materials whenever dirty samples are analyzed.

Figure 1

**Splitless liners** are designed to hold the sample in the liner for 0.5 to 2 minutes. A large surface area for sample vaporization is not a factor in splitless injections.



The *straight tube design* is the most common splitless sleeve design. This liner is ideal for low molecular weight samples that are not prone to thermal decomposition. If used for high molecular weight sample analysis, packing material is recommended to aid in sample vaporization. The drawback with using packing material is that the

resulting increased residence time of the sample can cause adsorption of the high molecular weight compounds.

The *gooseneck liner* isolates the sample from the metal injection port parts situated at the base of the injector. This design funnels the sample onto the analytical column for increased splitless efficiency and decreases the breakdown of highly active compounds, such as endrin and DDT. A *double gooseneck design* helps to contain the sample cloud in the liner, for increased performance with larger sample introductions, but cannot be packed with wool.

### Split Inlet Liners (Figure 2)

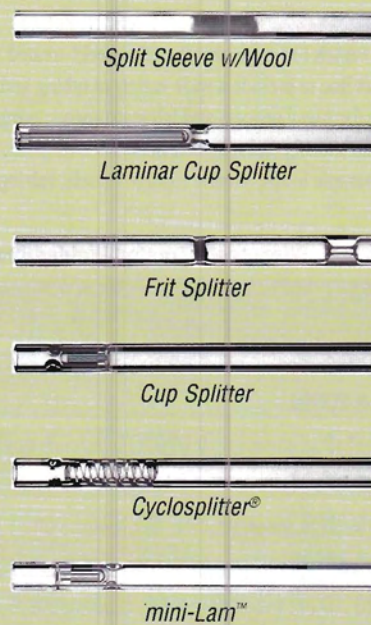
Split liners are designed to help vaporize the sample before it enters the column using mixing chambers and tortuous flow paths. Materials such as deactivated fused silica wool or beads, CarboFrit™ packing, and other packings are used to increase sample vaporization.

The most common liner for split analysis is the *4mm straight liner with deactivated wool*. This offers the analyst a wide variety of options. The wool has a high surface area for more sample evaporation to occur, and promotes a uniform vapor cloud to enter the split point. This liner is the most economical of the split liners; the drawback is that the wool increases breakdown of highly active compounds. Extensive upkeep is required to maintain analysis reproducibility when using this liner, as the wool needs to be changed frequently and its position and quantity inside the liner is critical.

*Cup splitter liners* offer a more homogenous vaporization through increased sample residence time in the liner. The sample passes through a series of tortuous flow paths, which aids in sample vaporization. First, the sample travels around an elongated cup and is trapped at the base of the liner, where vaporization occurs. Then it travels back up the liner and onto the column. These liners are best suited for high molecular weight compounds. The *Cycloplitter® liners* incorporate a cylindrical glass screw in the sample pathway. The screw helps to mix and vaporize the sample. The increased

Figure 2

**Split liners** are designed to use mixing chambers and tortuous flow paths to help vaporize the sample before it enters the column.



surface area in the cylindrical glass screw also helps to trap non-vaporized sample, therefore making it ideal for dirty samples.

### Does Deactivation Make a Difference?

Deactivation chemistry has come a long way since acid and dimethyldichlorosilane (DMDCS) deactivation. With more choices available, how do you choose one deactivation chemistry over another? Deactivation of the inlet liner is critical in the introduction of the sample to the column, because the liner is the first point of contact for the sample in the inlet system. If the liner is not properly deactivated, adsorption or breakdown of the sample can occur and result in poor quantitation or misidentification of compounds (Figure 3). For the majority of analyses, liner deactivation is



necessary to ensure complete, sample transfer to the capillary column. Deactivation is especially critical for analysis of certain pesticides, herbicides, amines, acids, and drugs.

Not all deactivations are alike—different types of chemicals and processes are used to deactivate the surface of the glass. There are several types of liner deactivations available:

#### Pinpoint Deactivation

This is the most widely used deactivation technique for liners and typically uses DMDCS deactivation. It is good for most non-critical analyses, analysis of polyaromatic hydrocarbons (PAHs), highly concentrated samples, and non-active sample matrices. This deactivation has very low resistance to sample degradation before re-deactivation or liner replacement is needed.

#### Polymeric Deactivation

A polymeric deactivation provides total surface coverage. There are no exposed active sites as there are with pinpoint deactivation. Polymeric deactivation has a high resistance to sample degradation and shows increased response for low concentration samples and highly active samples, such as endrin, DDT, and drugs. Endrin breakdown of less than 2% is standard when using a polymeric deactivation.

Longer liner lifetimes are provided because the deactivation is bonded to the surface of the glass and is more durable than pinpoint deactivation.

#### Base or Amine Deactivation

This is a special deactivation for the analysis of bases and amines. This deactivation provides superior response and sample repeatability for the analysis of trace amine compounds (Figure 4).

When choosing a liner for your analysis, match the liner geometry and deactivation to the analysis you will be performing. The liner geometry and deactivation are as important as the choice of column for the analysis of special compounds. Increased performance and more accurate analysis will be the results of a thoughtful decision.

Request Restek's  
handy pocket reference guide  
**Inlet Supplies**

(lit. cat. #59980), or visit our web site at [www.restekcorp.com](http://www.restekcorp.com). The Inlet Supplies pocket guide is an at-your-fingertips reference packed with liner selection information, inlet supplies, and a complete product listing.

Figure 3

### Deactivated Liners Significantly Decrease Endrin Breakdown

The untreated inlet liner exhibits 62% endrin breakdown.

The deactivated inlet liner exhibits 1% endrin breakdown.

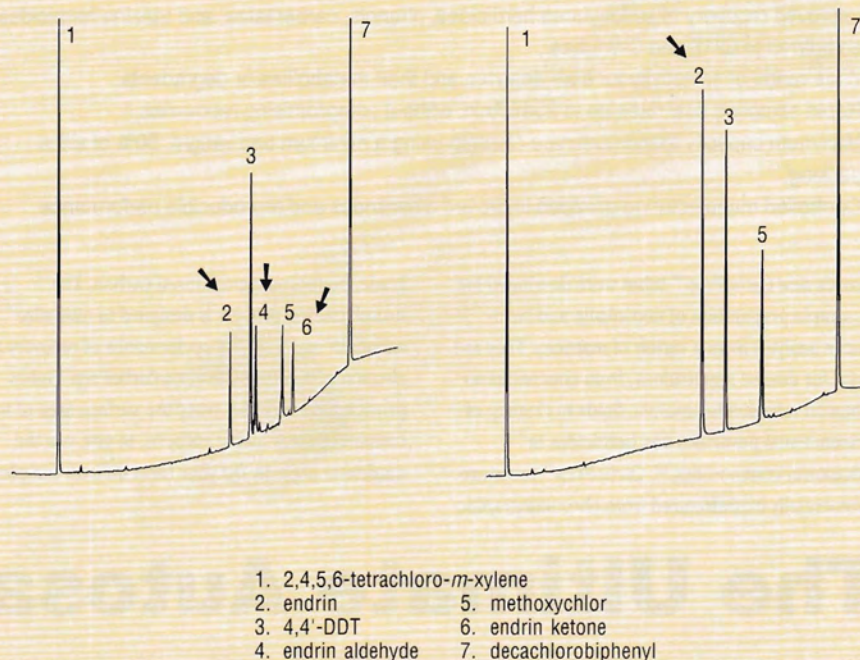


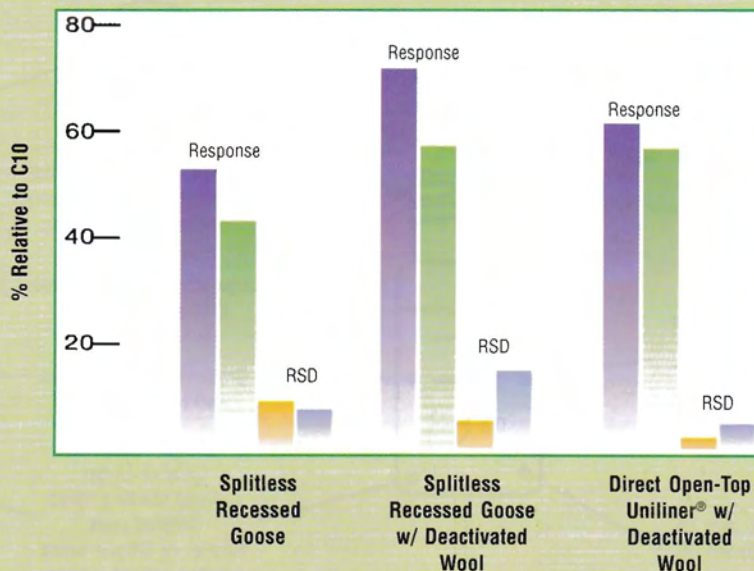
Figure 4

### Response Comparison of Amine Deactivation vs. Polymeric Deactivation.

■ Amine Deactivation

■ Polymeric Deactivation

Column: 30m, 0.53mm ID, 3.0µm, Rtx®-5 Amine; Diethanolamine on-column concentration: 15ng; Injections/sleeve: 5; Inj./det. temp: 250°C/285°C Each sleeve conditioned @ 285°C for 1 hour prior to injections. HP 5890II Plus with HP 7673 Autosampler.





# New CarboPrep™ SPE Tubes



**We are pleased to announce the addition of CarboPrep™ SPE tubes to our growing line of Resprep™ products!**

by Lydia Nolan, Sample Preparation Product Line Manager

- Improved recovery of sulfonylurea herbicides, phenols, carbamates, and triazine herbicides compared to C-18 and C-8 tubes.
- Wide range of selectivity for both analytes and their metabolites or degradents.
- Rapid sampling flow rates up to 20mL/min. without compromising recoveries.
- Maximum capacity for contaminate cleanup using a minimum bed weight, 50% of silica packing.
- Controlled manufacturing provides improved cleanliness and reproducible performance.

Restek's new CarboPrep™ tubes were developed for the sample preparation of nonvolatile and semi-volatile analytes from a variety of matrices. They are useful for diverse applications, from concentration of human estrogen in amniotic fluids to cleanup of environmental pesticides and herbicides in agricultural commodities.<sup>1,2</sup> CarboPrep™ tubes are commercially manufactured from chromatographic-

grade, nonporous, graphitized carbon. The manufacturing process is designed to minimize variability and improve performance of recovery and cleanup procedures. Restek's CarboPrep™ tubes provide a carbon with twice the surface area of most commercially available carbons, to give you the maximum capacity for your most difficult samples.

To experience the many benefits of CarboPrep™ tubes for yourself, call 800-356-1688, ext. 3, or contact your local Restek representative for a sample pack and *Get Prepped* today!

#### References

1. F. Andreolini, C. Borra, F. Caccamo, A. DiCorcia, and R. Samperi. *Analytical Chemistry*, 1987, 59, pp. 1720-1725.
2. J. Fillion and L. Nolan, *Today's Chemist*, 1996, pp. 14-24.

cat. #	
CarboPrep™ 3mL, 250mg 26088	(50-pk.)
CarboPrep™ 6mL, 500mg 26087	(30-pk.)

Custom sizes available, please call for details.

## The Ultimate Autosampler Vials

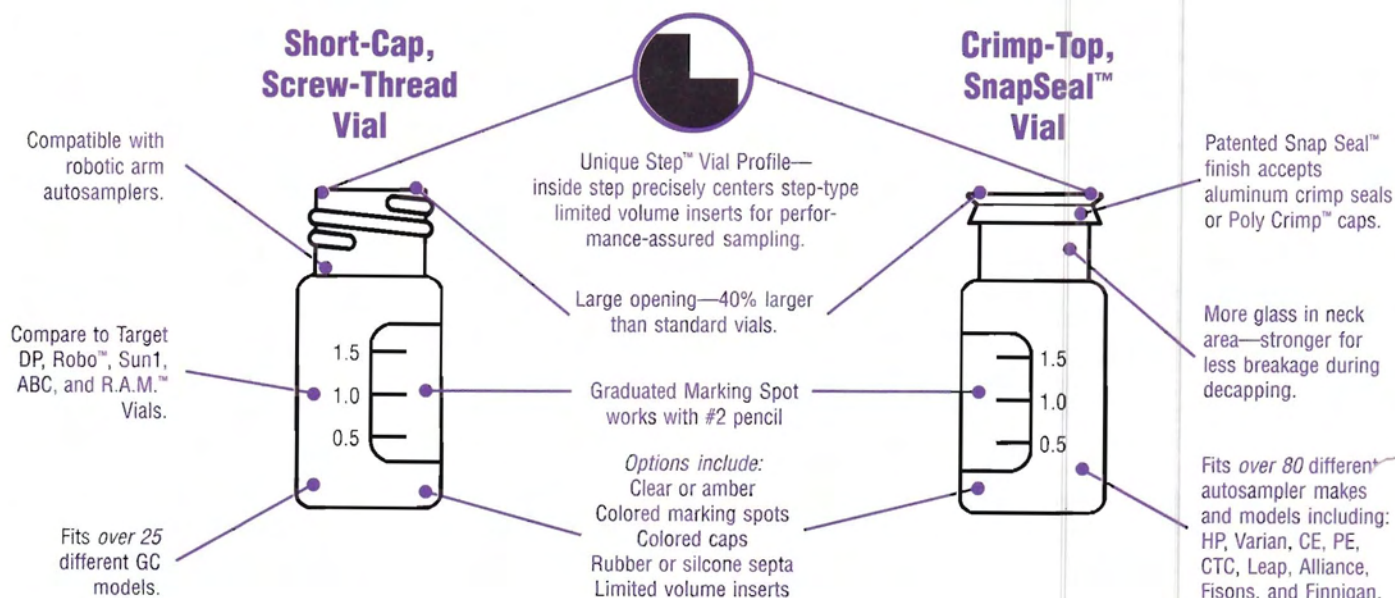
Restek has assembled what may be the ultimate line of autosampler vial products. These vials offer more features—such as large openings, step insert profiles, and graduated marking spots—all at the same low price as other manufacturers' standard plain vials. For a complete listing of Restek's extensive line of vials for any instrument or application, see the *1999 Product Guide*.

**For a free sample vial kit, call 800-356-1688, ext. 3, or contact your local Restek representative.**

✓ **Type I, 33 expansion borosilicate glass offers the least pH shift (lowest leaching characteristics) of any glass type.**

✓ **Manufactured in compliance with ISO 9002 guidelines for assured, consistent quality.**

✓ **Restek's exclusive Silcote™ CL7, silicon polymer-based silanization ensures sample integrity.**



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• 14 •

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# Peak Performers

by Doug Elliott, GC Accessories Product Line Manager

## Flexible Graphite Ferrules

### New Technology Means Longer Ferrule Life

Our fused silica quality assurance (QA) analysts are reporting twice the number of re-use cycles with the new Restek Flexible Graphite Ferrules. In addition to having lower bleed and improved sealing performance, they can be re-used 4 or 5 times! Our analysts are very happy with this high level of performance. Let us know how well they work in your applications.



For a complete listing of ferrules, please see the Restek 1999 Product Guide.

### Graphite Capillary Ferrules

(for 1/16" compression-type fittings)

Ferrule ID	Fits Column ID	10-pack
0.3mm	0.20mm	20233
0.4mm	0.25mm	20200
0.5mm	0.32mm	20201
0.8mm	0.53mm	20202

### 50-pack

0.4mm	0.25mm	20227
0.5mm	0.32mm	20228
0.8mm	0.53mm	20224

### Compact Graphite Ferrules for HP GCs

(for capillary injection ports)

Ferrule ID	Fits Column ID	10-pk.
0.4/0.5mm	0.25–0.32mm	20250
0.8mm	0.53mm	20252

### 50-pk.

0.4/0.5mm	0.25–0.32mm	20251
0.8mm	0.53mm	20253

## Cross Disk Inlet Seal

### Replacement 0.8mm ID Inlet Seal, for HP 6890 GCs

Restek now has the 0.8mm ID cross-type inlet seals for HP 6890 injectors. Deactivations include our gold-plating process, which has been optimized to be stable and uniform. The legendary Silcosteel®-treated seals are as inert as gold, but more rugged. Try them for yourself!



Washers included!

### 0.8mm ID Cross Disk Inlet Seal for HP GCs

(Similar to HP part #5182-9652)

Inlet Seal Type	2-pk.	10-pk.
Gold-Plated	20477	20476
Silcosteel®-Treated	20475	20474

For a complete listing of inlet seals, please see the 1999 Product Guide.

## Air Diverter

### for 5890/6890 GCs



- Diverts GC exhaust heat away from the lab bench.
  - Reduces oven cycle time.
  - Improves retention time precision.
  - Easy to install—no tools required.
- (Similar to HP part # 19247-60510)  
Cat.# 22076, (ea.)

## Restek Leak Detective™

### Electronic Leak Detector



- Compact, lightweight, hand-held design.
  - Contamination-free leak detection.
  - Detects helium or hydrogen trace leaks at  $\geq 3 \times 10^{-4}$  cc/sec or  $\geq 200$ ppm.
  - Pays for itself in the first leak found.
  - Battery or AC line adaptor
- 110v:** Cat. #21607, **220v:** Cat. # 21609, (ea.)

## Clean & Green

### Instrument-Grade Tubing



We know how important clean tubing for plumbing is to the successful delivery of pure gas to your instrument. We also know that we have a responsibility to protect the environment and our employees. For those reasons, Restek has developed a new cleaning process that uses biodegradable detergents, solubilizers, penetrants, organic acids, and non-toxic solvents for superior surface cleaning—leaving no film or residue.



Now even better!

When you want the cleanest tubing for plumbing your analytical instrument system, and you also are concerned about the quality of the environment and worker safety, choose Restek Instrument-Grade Tubing. See the 1999 Product Guide for details.



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the

# RESTEK Advantage

Innovators of High Resolution Chromatography Products

## Siltek™ Deactivation— The Next Generation

### Outstanding Performance for Chlorinated Pesticide Analyses

by Dr. David Smith, R&D Chemist, and Deb Salabsky, Applications Chemist

- ★ Maximizes the inertness of sample pathway.
- ★ Minimizes breakdown.
- ★ Low bleed.
- ★ Thermally stable.
- ★ "Clean and green"—manufactured without the use of harmful organic solvents.



**First** Restek developed a 100% polymeric, high-temperature silanization process for inlet liners. Restek's polymeric silanization is the deactivation of choice, resulting in low endrin breakdown and inertness for compounds containing active functional groups like phenols, diols, and acids.

**Next** Restek developed a surface deactivation for handling basic compounds, like those found in drugs, azo-dyes, and amines. Our base-deactivated glass accessories provide excellent recovery of trace-level active basic compounds.

#### Now Restek Introduces the Next Generation of Deactivation... Siltek™.

The Siltek™ deactivation process (patent pending) produces a highly-inert glass surface, which features high temperature stability, extreme durability, and low bleed. Try Siltek™-deactivated liners, guard columns, wool, and connectors for minimized breakdown and better recovery of sample analytes.

#### Siltek™-Deactivated Inlet Liners

Gas chromatographic (GC) analysis of chlorinated pesticides presents unique challenges to environmental laboratories because these compounds often are analyzed at trace levels and are susceptible to decomposition caused by reactive sites in the analytical system. Pesticide methods, such as the US Environmental Protection Agency (EPA) Methods 8081 and 608, have stringent breakdown criteria. The two pesticide compounds used to monitor system inertness are notorious for exhibiting breakdown—endrin, which breaks down into endrin aldehyde and endrin ketone, and DDT, which breaks down into DDE and DDD. The breakdown of these compounds most often occurs in the GC injection port. (cont. on page 2)

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Tubes**  
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**Integra-Guard™ Guard Columns**  
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**Restek-on-the-Road—New  
Comprehensive GC Seminar**  
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Rtx®-CLPesticides Columns**  
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OLM 04.1 Reference Materials**  
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# Siltek™ Deactivation—The Next Generation

(continued from pg. 1)

Routine maintenance of the injection port and GC columns is essential to minimize compound breakdown.

To illustrate the importance of proper surface deactivation for endrin and DDT analysis, a 50pg/μL test mix was injected on an undeactivated direct injection glass inlet liner. Endrin breakdown was 62% and DDT breakdown was below detection limits, as shown in Figure 1.

Next, the raw liner was removed and replaced with a Siltek™-deactivated direct injection liner. The results of this injection are shown in Figure 2. Endrin breakdown measured less than 1% and DDT breakdown again was below detection limits. The results not only confirm the necessity for inlet liner deactivation in pesticides analysis, but also show the inherent inertness of Siltek™ deactivation and its ability to improve the accuracy of pesticides analysis.

## Siltek™-Deactivated Guard Columns

Guard columns are commonly used in the analysis of chlorinated pesticides. Many analysts use them as a way to divide a sample equally onto two different analytical columns by way of a Press-Tight® 'Y' connector. This configuration allows a primary and a confirmational analysis using one injection. Guard columns also make routine maintenance easier by allowing removal of the first meter of column. This eliminates non-volatile contamination, without affecting the analytical column(s). Siltek™-deactivated guard columns and Siltek™-deactivated connectors provide an inert sample introduction pathway that is ideal for chlorinated pesticide analysis.

## Siltek™ Deactivation—The Complete Solution

The analysis of US EPA Method 8081 calibration standard is shown in Figure 3. This chromatogram was generated using a Siltek™-deactivated inlet liner and guard column, and an Rtx®-CLPesticides analytical column.

The best protection against endrin and DDT breakdown for chlorinated pesticide analysis is to outfit your GC with Siltek™-treated products. For a highly inert pathway and fast GC cycle times, use Rtx®-CLPesticides and Rtx®-CLPesticides2 analytical columns in combination with Siltek™-deactivated liners and guard columns.

**for more info**

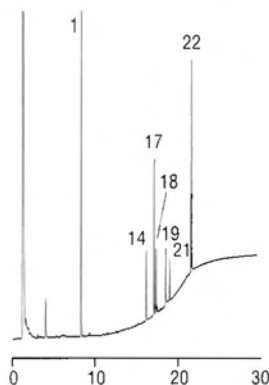
For more information on Siltek™ deactivation, request "Siltek™ Deactivation Benefits Brochure" (lit. cat.# 59803).

Call 800-356-1688, ext. 5, or contact your local Restek representative.

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**Figure 1**

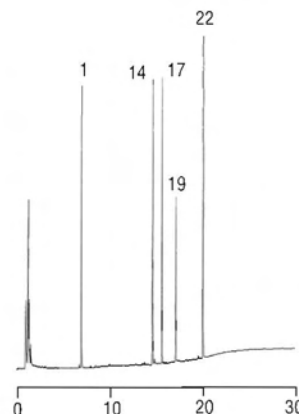
Direct injection Uniliner® without deactivation exhibits 62% endrin breakdown.



**Figure 2**

A Siltek™-deactivated liner exhibits approximately 1% endrin breakdown.

30m, 0.53mm ID, 0.42μm  
Rtx-CLPesticides2 (cat.#11340)  
with open-top Uniliner®  
(cat.# 20843-214.1 for Siltek™  
deactivation)  
Inj.: 1μL of 50pg/μL standard of  
tetrachloro-meta-xylene (IS),  
endrin, 4,4'-DDT, methoxychlor,  
and decachlorobiphenyl (IS)  
Oven temp.: 120°C (hold 1 min.)  
to 300°C @ 9°C/min.  
(hold 10 min.)  
Inj. temp.: 250°C  
Det.: ECD, 300°C  
Carrier gas: helium



**Figure 3**

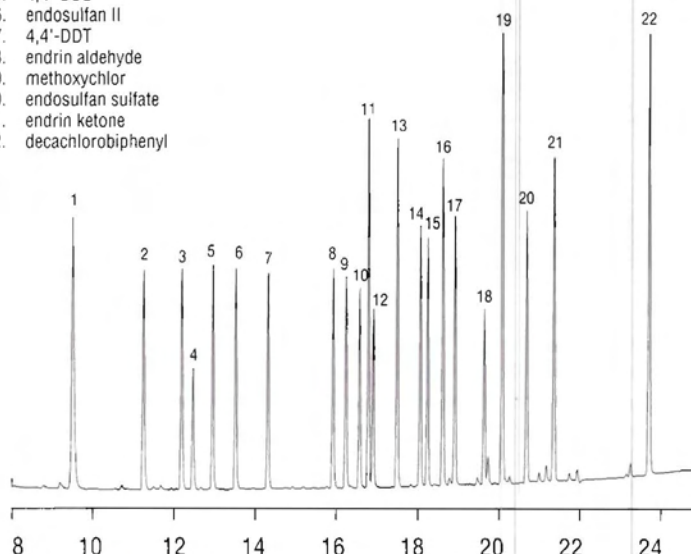
Analysis of US EPA Method 8081 with a Siltek™-deactivated Uniliner® and guard column shows resolution of all 22 chlorinated pesticides.

## Peak List for Figures 1, 2, and 3

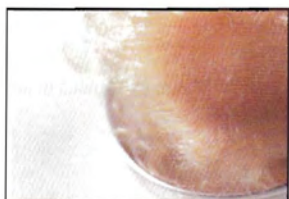
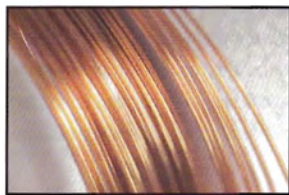
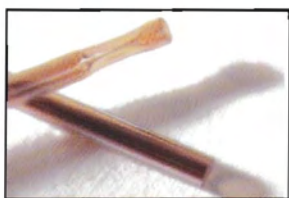
- 2,4,5,6-tetrachloro-m-xylene
- α-BHC
- γ-BHC
- β-BHC
- δ-BHC
- heptachlor
- aldrin
- heptachlor epoxide
- γ-chlordane
- α-chlordane
- 4,4'-DDE
- endosulfan I
- dieldrin
- endrin
- 4,4'-DDD
- endosulfan II
- 4,4'-DDT
- endrin aldehyde
- methoxychlor
- endosulfan sulfate
- endrin ketone
- decachlorobiphenyl

30m, 0.32mm ID, 0.5μm (cat.# 11139) Rtx®-CLPesticides  
with a 5m, 0.32mm ID Siltek™-deactivated guard column  
(cat.# 10027) and a Siltek™-deactivated gooseneck liner  
(cat.# 20798-214.1)

On-column conc.: 16-160pg  
Oven temp.: 120°C (hold 1 min.) to 300°C @ 9°C/min.  
(hold 10 min.)  
Inj. temp.: 250°C, splitless (hold for 0.75 min.)  
Det.: ECD, 300°C with Anode purge  
Carrier gas: helium at 31cm/sec. linear velocity







For Siltek™-deactivated inlet liners, add the corresponding suffix number to your liner catalog number.

Siltek™-Deactivated Inlet Liners			
qty.	Siltek™	Siltek™ with Siltek™-deactivated wool	Siltek™ with CarboFrit™
each	-214.1	-213.1	-216.1
5-pk.	-214.5	-213.5	-216.5

Siltek™-Deactivated Press-Tight™ Connectors*		
type	qty.	cat.#
straight	25-pk.	20449
angled "Y"	3-pk.	20469
<i>Other types of Press-Tight™ connectors can be ordered on a custom basis by adding the suffix -266.</i>		
other	each	-266

Siltek™-Deactivated Guard Columns			
nominal ID	nominal OD	5-meter	10-meter
0.25mm	0.37 ±0.04mm	10026	10036
0.32mm	0.45 ±0.04mm	10027	10037
0.53mm	0.69 ±0.04mm	10028	10038

Siltek™-Deactivated Borosilicate Wool	
qty.	cat.#
10 g	21100

for **moreinfo**

See page 10 for an article on US EPA Method 8081A Chlorinated Pesticide Analysis.

## CarboPrep™ SPE Tubes

For Improved Recovery

by Gary Stidsen, Sample Preparation Product Line Manager

- \* Excellent for cleanup of pesticide residue extracts.
- \* Maximum capacity for contaminate cleanup using a minimum bed weight.
- \* Improved recovery of sulfonylurea herbicides, phenols, carbamates, and triazine herbicides compared to C-18 and C-8 tubes.
- \* Wide range of selectivity for both analytes and their metabolites or degradents.
- \* Rapid sampling flow rates up to 20mL/min. without compromising recoveries.

Restek's CarboPrep™ tubes were developed for the sample preparation of nonvolatile and semi-volatile analytes from a variety of matrices. They are useful for diverse applications, from concentration of human estrogen in amniotic fluids to cleanup of environmental pesticides and herbicides in agricultural commodities.<sup>1,2</sup> CarboPrep™ tubes are commercially manufactured from chromatographic-grade, nonporous, graphitized carbon. The manufacturing process is designed to minimize

variability and improve performance of recovery and cleanup procedures. Restek's CarboPrep™ tubes provide a carbon with twice the surface area of most commercially available carbons, to give you the maximum capacity for your most difficult samples.

To experience the many benefits of CarboPrep™ tubes for yourself, call 800-356-1688, ext. 3, or contact your local Restek representative for a sample pack and *Get Prepped* today!

cat. #
CarboPrep™ 3mL, 250mg 26088 (50-pk.)
CarboPrep™ 6mL, 500mg 26087 (30-pk.)

*Custom sizes available, please call for details.*

#### References

1. E. Andreolini, C. Borra, F. Caccamo, A. DiCorcia, and R. Samperi., *Analytical Chemistry*. 1987. 59. pp. 1720-1725.
2. J. Fillion and L. Nolan, *Today's Chemist*. 1996. pp. 14-24.

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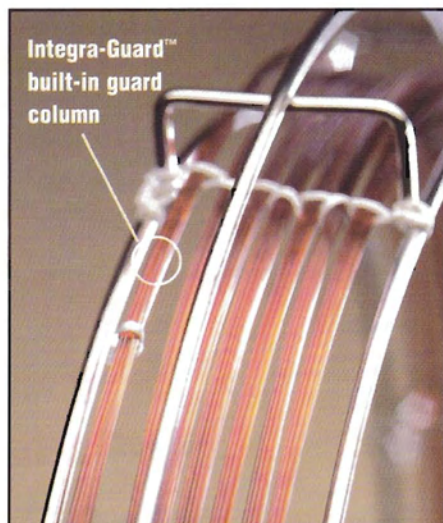
• 3 •



# Integra-Guard™ Columns

## Get the Protection Without the Connection!

by Rick Crago, Fused Silica Columns Product Line Manager



**Figure 1**

The best connection is no connection at all!



**Table I**

Phases currently available  
with an Integra-Guard™ guard column.

Rtx®-1  
Rtx®-1MS  
Rtx®-5  
Rtx®-5MS  
XTI®-5  
Rtx®-1301  
Rtx®-624  
Rtx®-1701  
Rtx®-Volatiles  
Rtx®-20  
Rtx®-35  
Rtx®-BAC 1  
Rtx®-BAC 2  
Stabilwax®  
Stabilwax®-DA  
Stabilwax®-DB

Some people swear by press-fit connectors, and others swear at them. For many analysts, the art of attaching a guard column to an analytical column is a mystery. Restek's chemists have discovered the solution to this mystery—the most reliable connection is no connection at all! No guard column system is more permanent than one continuous length of tubing containing both the guard column and the analytical column.

Restek pioneered this innovative approach to guard columns, and our Integra-Guard™ column is engineered to be worry-free for the analyst. The transition area between the guard and analytical column is the point at which the guard column ends and the analytical column begins. High-temperature string is used to tie the guard column in a separate loop, making it easy to distinguish from the analytical column. The entire setup is suspended in Restek's unique "crush-free" cage, which prevents the column from coming in contact with anything that could damage it. We offer Integra-Guard™ columns in a wide variety of phases (Table I).

### Guard columns are beneficial because they:

- \* Increase the lifetime of a column by trapping non-volatile contamination on the first few meters.
- \* Allow for system maintenance without sacrificing separation.
- \* Function as "retention gaps," allowing components with different volatilities to be focused in the same band on the analytical column.

### Problems that conventional guard columns can create:

- \* Difficult and time consuming to make a proper connection.
- \* Leaks may develop during an analysis, destroying the columns.
- \* Active sites and dead volume from tubing connectors may cause peak tailing and loss of resolution.

### Integra-Guard™ Columns provide the solution:

- \* The most reliable connection between a guard column and an analytical column is pictured at the left NO CONNECTION AT ALL (Figure 1).
- \* Integra-guard technology eliminates the need for a connector by incorporating the guard column into the actual analytical column.

If you are currently using a guard column or considering one for the future, call Restek today and ask about our Integra-Guard™ columns.

Integra-Guard™ Guard Columns*		
mm ID	length	suffix#
0.25	5m	-124
	10m	-127
0.32	5m	-125
	10m	-128
0.53	5m	-126
	10m	-129

\*Add suffix # and price to the order of your column.



# Allure™ Acidix HPLC Column

## Specialized Columns for LC/MS Analysis of Acids and Amino Acids

by Keith Duff, Dave Bell, and Vernon Bartlett



- ★ Improved LC/MS sensitivity.
- ★ Maximum retention of acidic compounds.
- ★ High selectivity and reproducibility.
- ★ Analysis of amino acids without derivatization.

Restek's Allure™ phases are engineered to provide maximum sensitivity for liquid chromatography/mass spectrometry (LC/MS) separations. The Allure™ Acidix phase retains and exhibits good peak shape for compounds that contain carboxylic acid, sulfonic acid, phosphoric acid, or other acidic functional groups. Maximum retention occurs at high percentages of organic modifier, which results in a desirable increase in the signal-to-noise ratio for LC/MS analyses.

Use of the Allure™ Acidix column allows chromatographers to generate high-throughput acid and amino acid analyses by eliminating the need for derivatization of these analytes. Typically, amino acids require derivatization prior to separation by HPLC. This adds much cost and time to the method, and should be avoided whenever possible. In addition, incomplete derivatization can distort quantitative measurements. This is a problem particularly when more than one moiety on an amino acid can be derivatized. Until now, underivatized amino acids only could be run using ion exchange, but many of the buffers required in these methods are not LC/MS compatible. The Allure™ Acidix column retains and separates amino acids, achieving good peak shape using simple isocratic mobile phases (Figure 1). Volatile buffers and high organic content can be used in the mobile phase to assist in the LC/MS ionization process for good MS sensitivity.

However, most of the advantages exhibited by the Allure™ Acidix are not limited to LC/MS separations. Fast analyses, excellent peak shape and retention, high reproducibility, and the elimination of the need for derivatization apply to other HPLC systems as well. Polar acids (e.g., ascorbic acid) that are difficult to retain by traditional C18 columns are easily retained by the Allure™ Acidix phase (Figure 2). Larger molecular weight acidic compounds such

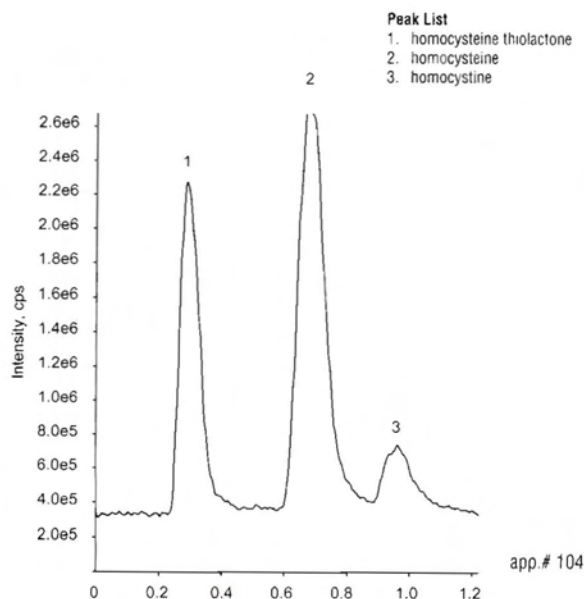
as pharmaceuticals also show excellent retention and peak shape (Figure 3). Additionally, salicylic acid and aspirin are cleanly and easily separated on the Allure™ Acidix column (Figure 4).

The Allure™ Acidix column is a breakthrough in HPLC stationary phase development that permits the analysis of amino acids without derivatization. Furthermore, it provides the highest sensitivity

available for LC/MS detection of acidic compounds, and it retains most acids that C18 phases cannot. For samples with solubility limitations, the Allure™ Acidix column can be used at both the high and the low end of percent organic in the mobile phase. Most importantly, it displays the reproducibility and reliability chromatographers have come to expect from Restek chromatography products.

**Figure 1**

**Allure™ Acidix column retains and separates un-derivitized amino acids, achieving good peak shape using isocratic mobile phases and LC/MS.**



<b>Column:</b>	Allure™ Acidix
<b>Catalog#:</b>	9162552
<b>Dimensions:</b>	50 x 2.1mm
<b>Particle Size:</b>	5µm
<b>Pore Size:</b>	60Å
<b>Conditions:</b>	
<b>Mobile Phase:</b>	water pH to 3.0 with formic acid:acetonitrile (40:60)
<b>Flow:</b>	0.2mL/min.
<b>Temperature:</b>	ambient
<b>Detection:</b>	PE/SCIEX API 150 EX, TURBO IONSpray <sup>®</sup>
<b>Ionspray voltage:</b>	+3000V
<b>Temperature:</b>	350°C
<b>Orifice:</b>	10V
<b>Ring:</b>	90V
<b>Auxiliary gas:</b>	6000cc/min.
<b>Sample:</b>	
<b>Inj.:</b>	5µL
<b>Conc.:</b>	10µg/mL
<b>Solvent:</b>	mobile phase

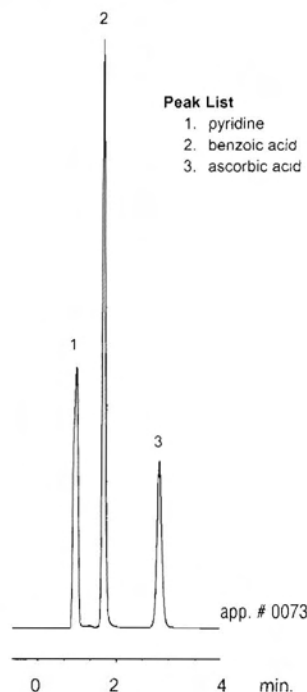


# Allure™ Acidix HPLC Column

(continued from pg. 5)

**Figure 2**

Polar acids that are difficult to retain on traditional C18 columns are easily retained on the Allure™ Acidix column.



**Column:** Allure™ Acidix  
**Catalog#:** 9162565  
**Dimensions:** 150 x 4.6mm  
**Partical Size:** 5µm  
**Pore Size:** 60Å

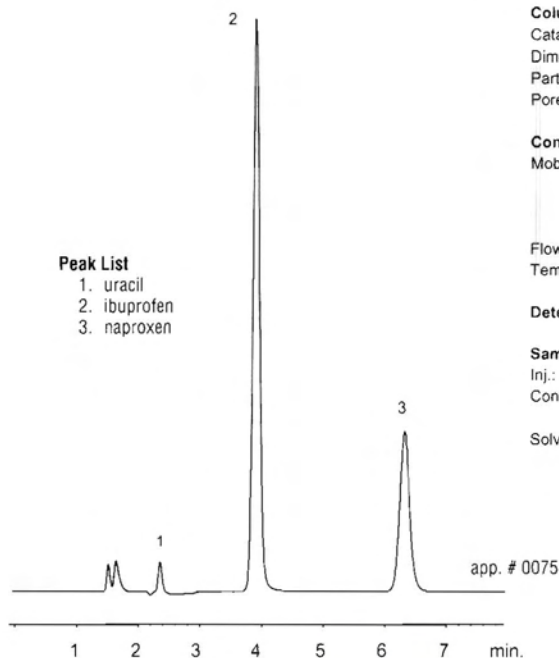
**Conditions:**  
**Mobile Phase:** 10mM potassium phosphate  
 pH 2.6: acetonitrile (60:40 v/v)  
**Flow:** 1.2mL/min.  
**Temperature:** 27°C

**Detection:** UV @ 230nm

**Sample:**  
**Inj.:** 25µL  
**Conc. (acids):** 250µg/mL  
**Solvent:** water:acetonitrile (99:1, v/v)

**Figure 3**

Pharmaceuticals show excellent retention and peak shape on the Allure™ Acidix column.



**Column:** Allure™ Acidix  
**Catalog#:** 9162565  
**Dimensions:** 150 x 4.6mm  
**Particle Size:** 5µm  
**Pore Size:** 60Å

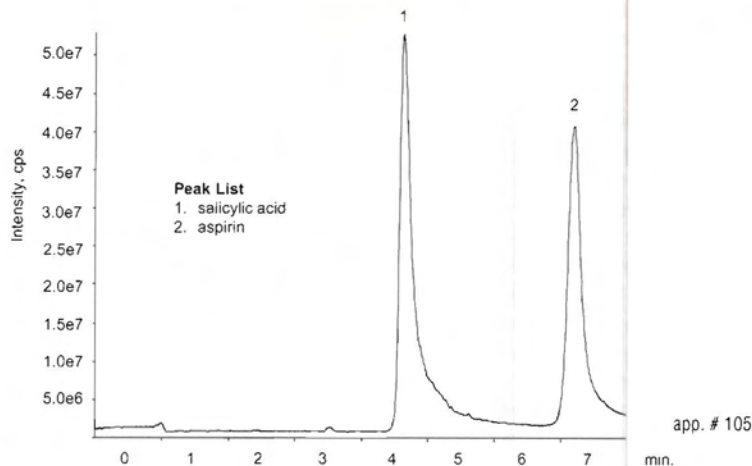
**Conditions:**  
**Mobile Phase:** 20mM ammonium  
 acetate pH 4.5:  
 acetonitrile  
 (30:70, v/v)  
**Flow:** 1.0mL/min.  
**Temp.:** ambient

**Detection:** UV@220nm

**Sample:**  
**Inj.:** 10µL  
**Conc.:** 100µg/mL (ibuprofen  
 and naproxen)  
**Solvent:** ammonium acetate  
 buffer:acetonitrile  
 (1:1, v/v)

**Figure 4**

Salicylic acid and aspirin are cleanly and easily separated on the Allure™ Acidix column.



**Column:** Allure™ Acidix  
**Catalog#:** 9162565  
**Dimensions:** 150 x 4.6mm  
**Partical Size:** 5µm  
**Pore Size:** 60Å

**Conditions:**  
**Mobile Phase:** 20mM ammonium  
 formate, pH 4.5:  
 acetonitrile (20:80, v/v)  
**Flow:** 1mL/min  
**Temperature:** ambient

**Detection:** PE/SCIEX API 150 EX,  
 Heated Nebulizer interface  
 in negative ion mode

**Needle current:** 3µA  
**Temperature:** 350°C  
**Orifice:** -5V  
**Ring:** -30V  
**Auxillary gas:** 6000cc/min.

**Sample:**  
**Inj.:** 5µL  
**Conc.:** 50µg/mL  
**Solvent:** water:methanol (1:1, v/v)

## Restek Survival Kit for HPLC



cat.# 25322

The Restek Survival Kit is an invaluable analytical spare-parts kit that contains the essential tools and supplies to maintain and set-up your solvent delivery system.

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• 6 •



## Product Listing

Allure™ Acidix Columns				
Particle Size: 5µm	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	9162531	9162532	9162533	9162535
50mm length	9162551	9162552	9162553	9162555
100mm length	9162511	9162512	9162513	9162515
150mm length	9162561	9162562	9162563	9162565
200mm length	9162521	9162522	9162523	9162525
250mm length	9162571	9162572	9162573	9162575

for **moreinfo**

For more information on Allure™ HPLC columns, request the **LC/MS Columns Flyer** (lit. cat.# 59735).

### Free HPLC wall chart and LC/MS Catalog

To receive your free wall chart (lit. cat.# 59894) and catalog (lit. cat.# 59607) call our Literature Request Hotline at **800-356-1688, ext. 5**; or visit us online at **www.restekcorp.com**.



# Silcosteel®-Treated Sample Cylinders

## New SCAQMD Standard for Sulfur Analyses

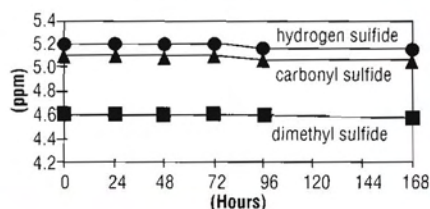
by Gary Barone, MPG Product Line Manager, and Dave Shelow, Air Monitoring Products Product Line Manager

visit us at  
**Gulf Coast Conference**  
booth #223



**Figure 1**

Sulfur compound (~5.0ppm) storage in a Silcosteel®-treated stainless steel cylinder over seven days shows no significant compound loss.



Results of a recent study by South Coast Air Quality Management District (Rule 1118 Ad Hoc Working Group) determined that Silcosteel®-treated sample cylinders are accepted for Method 307-91, *Determination of Sulfur in a Gaseous Mixture*.

The use of stainless steel high-pressure cylinders has greatly increased the accuracy and efficiency of gas sampling. Tedlar® bags, which previously had been recommended, required a large pressure reduction and possible loss of sample through the permeable wall. Silcosteel® treatment is a thin-film passivation treatment for stainless steel, resulting in a rugged and inert coating that decreases sample loss, and the stainless steel cylinder reduces pressure problems.

During the last 12 years, Silcosteel® treatment has been used for many applications in analytical chemistry. Most recently, Silcosteel® sample cylinders have become the standard for gas sampling in the petroleum industry.

This Silcosteel® coating is ideal for applications involving the transfer of extremely low-level organics and corrosives (see Figure 1). Unlike fluoropolymer surface coverings, Silcosteel® coating is incorporated into the lattice of the steel, which results in very high stability and durability—to maintain the integrity of your sample. Always use Silcosteel®-treated sample pathways when peak performance is required for sulfur-related work.

### Silcosteel®-Treated Sample Canisters

size	cat.#	size	cat.#
75cc	24271	500cc	24274
150cc	24272	1000cc	24275
300cc	24273		

### Silcosteel®-Treated Hoke Sample Cylinder Valves

description	metal stem tip cat.#	Kel-F stem tip cat.#
1/4" male NPT exit	24276	24278
1/4" compression exit	24277	24279

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## Restek Survival Kit for HPLC

*For start-up and standard use in all HPLC-systems*

The Restek Survival Kit is an invaluable analytical spare part kit that contains the essential tools and supplies for your solvent delivery needs.

cat.# 25322

### *Restek HPLC Survival Kit Contents:*

Item	Qty.
PEEK Column Connector	10
PEEK Tubing, 1/16" OD x 0.005" ID	3m
PEEK Tubing, 1/16" OD x 0.007" ID	3m
PEEK Tubing, 1/16" OD x 0.010" ID	3m
PEEK Tubing, Elbow 90	5
PEEK Tubing, Elbow 180	5
Teflon Tubing, 1/8" OD x 0.063"	3m
Teflon Tubing, 1/8" OD x 0.094"	3m
Tubing Clip	5
Rheotool Wrench	1
1/4" x 5/16" Open-End Wrench	1
Clean-Cut™ Tubing Cutter	1
Replacement Blades for Cutter	1
PEEK Union 1/16"	2
Inlet Filter, 2µm	1
Inlet Filter, 10µm	1

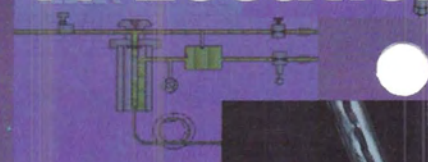
for **moreinfo**

For more information on the HPLC Survival Kit and other HPLC accessories, request the flyer "HPLC Mobile Phase Accessories" (lit. cat.# 59728-INT).

# Restek-on-the Compro GC S

## Coming to Near

### 41 Location





# oad presents ensive minar

Location  
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*Restek offers expert, hands-on training in many aspects of chromatography. For information on training topics or to discuss bringing training to your facility or area, call 800-356-1688, ext. 2174, or contact your local Restek representative.*

## Register Today!

**Call** us at 800-356-1688, ext. 3

**Fax** this form to 814-353-1309

**Mail** this form to Attn: Seminar Coordinator, 110 Benner Circle, Bellefonte, PA 16823

Register **electronically** at [www.restekcorp.com/seminars/register.htm](http://www.restekcorp.com/seminars/register.htm)

### Registration Form

Seminar Location: \_\_\_\_\_

Seminar Date: \_\_\_\_\_ Seminar# \_\_\_\_\_

Company: \_\_\_\_\_

Names of attendees:

**Attendee #1** \_\_\_\_\_

**Attendee #2** \_\_\_\_\_

**Attendee #3** \_\_\_\_\_

**Attendee #4** \_\_\_\_\_

**Attendee #5** \_\_\_\_\_

### only \$199 per person, Lunch included

Method of Payment: ☐ Check ☐ PO ☐ Visa ☐ American Express  
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*The seminar confirmation letter(s) will be sent to the billing address unless otherwise indicated below:*

Address \_\_\_\_\_

City \_\_\_\_\_ State \_\_\_\_\_ Zip \_\_\_\_\_

What is your application area? \_\_\_\_\_

What type of injection do you use? [e.g., split, direct] \_\_\_\_\_

What detectors do you commonly use? \_\_\_\_\_

How many years of GC experience do you have? \_\_\_\_\_

What's your toughest chromatography challenge? \_\_\_\_\_

Referred by / Salesperson: \_\_\_\_\_



# US EPA Method 8081A

## Excellent Separation of Chlorinated Pesticides Using Rtx<sup>®</sup>-CLPesticides and Rtx<sup>®</sup>-CLPesticides2 Columns

by Dr. Frank Dorman, Applications Group Leader

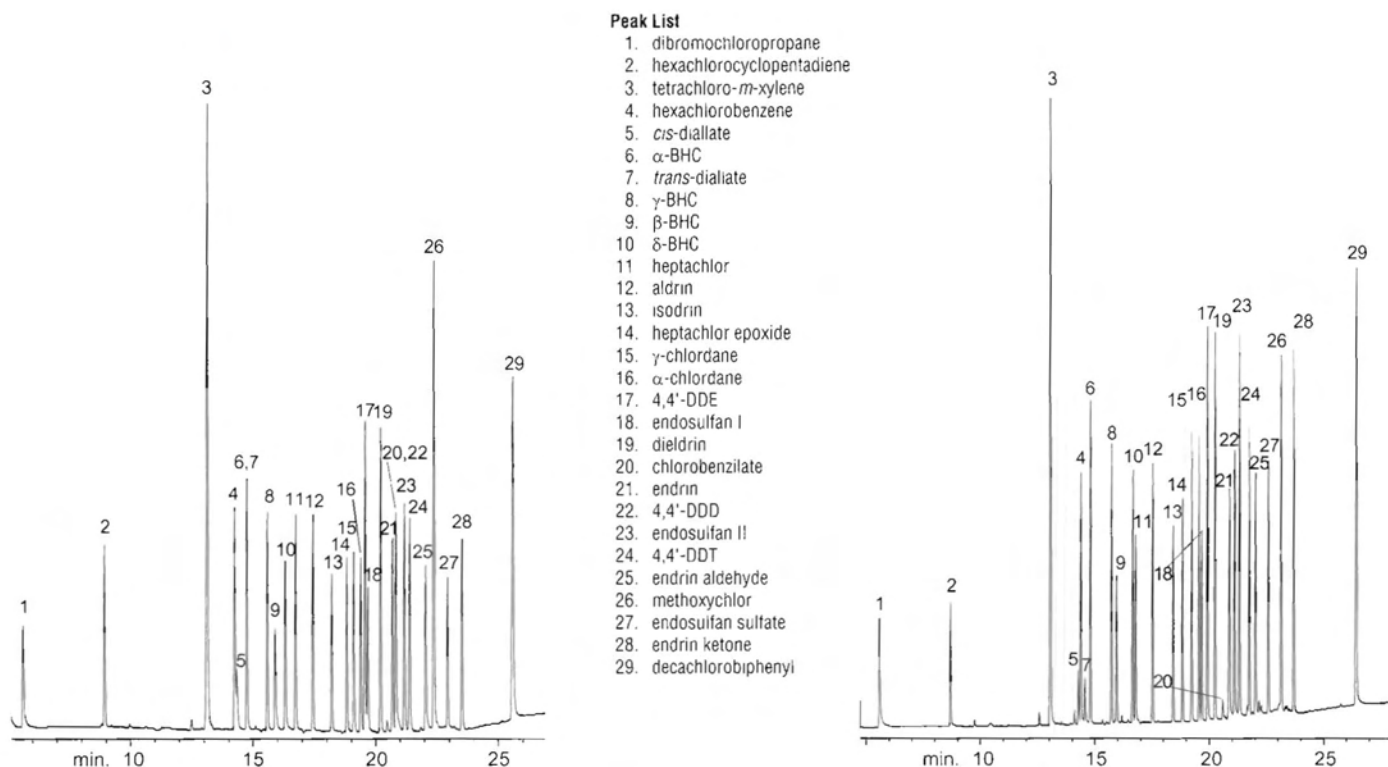
For environmental laboratories, the methods to analyze chlorinated pesticides often are the most challenging to perform. Analysts struggle with linearity, breakdown, and lengthy calibrations; as well as column bleed, column reactivity, and poor separation. Restek has addressed all of these issues with the development of the Rtx<sup>®</sup>-CLPesticides and the Rtx<sup>®</sup>-CLPesticides2 capillary columns. These columns were designed specifically for the separation of chlorinated pesticides, to be used in parallel for simultaneous quantitation and confirmation by gas chromatog-

raphy/electron capture detection (GC/ECD). We have shown the performance and separation that can be achieved with these columns for EPA Method 608, 8080, and 8081. Now many laboratories are dealing with the latest version of the chlorinated pesticides method—SW-846, 8081A. This method adds new target analytes to the 20 common single-component pesticides contained in earlier versions.

Because the Rtx<sup>®</sup>-CLPesticides and Rtx<sup>®</sup>-CLPesticides2 columns were designed with

selectivity for neutral, halogenated compounds, they are easily adapted for the analysis of the extended list of pesticides in Method 8081A. Figure 1 shows the separation of the 22 chlorinated pesticides listed in both US EPA Method 8081 and 8081A, plus the seven additional single-component compounds also listed in Method 8081A. Using a guard column and splitting the flow into the two columns with a glass 'Y' Press-Tight<sup>®</sup> connector, the chromatograms were acquired simultaneously. These two columns also have a high maximum operating

**Figure 1** Achieve full separation of US EPA Method 8081A chlorinated pesticides using the Restek Rtx<sup>®</sup>-CLPesticides and Rtx<sup>®</sup>-CLPesticides2 columns.



30m, 0.32mm ID, 0.50 $\mu$ m Rtx<sup>®</sup>-CLPesticides and 30m, 0.32mm ID, 0.25 $\mu$ m Rtx<sup>®</sup>-CLPesticides2 columns (cat.#'s 11139 & 11324).

On-column concentration: 16-160pg; Oven temp.: 80°C (hold 1 min.) to 300°C @ 10°C/min. (hold 15 min.);

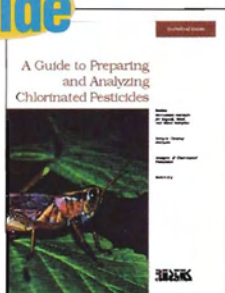
Inj. port: Direct, Uniliner<sup>®</sup> sleeve (cat.# 20335); Detector: ECD, 300°C with Anode Purge;  
Dead time: 1.9 min.; Head pressure: 8.7psi (constant); Flow rate: 1.3mL/min. @ 120°C, helium.



temperature, excellent inertness, low bleed, and can be used under the same flow and temperature conditions. This is beneficial for installing them as a column pair into a single injection port, thereby minimizing injector maintenance concerns.

The combination of the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns provides unsurpassed performance for the analysis of chlorinated pesticides. They can be baked-out at the end of each analysis to remove high-boiling contaminants, without degrading the stationary phase. They do not have the problems associated with cyanopropyl phases (1701) such as on-column methoxychlor and DDT breakdown, and low maximum temperature. The Rtx®-CLPesticides and Rtx®-CLPesticides2 columns are the best choice for improving resolution and capacity for the analysis of dirty extracts, and for increasing throughput for chlorinated pesticide samples.

**free  
guide**



Restek's 24-page technical guide, **A Guide to Preparing and Analyzing Chlorinated Pesticides** (lit. cat.# 59892), covers analytical details for the preparation and analysis of chlorinated pesticides. The guide discusses specific extraction methods for liquid, solid, and biota samples, in addition to sample cleanup methods and the actual analyses themselves.

To request your free copy, call our literature request hotline at 800-356-1688, ext. 5. or contact your local Restek representative.

For the most inert injection system with the lowest endrin breakdown, see page 3 for **Siltek™-deactivated** inlet liners, guard columns, and Press-Tight® connectors.

## Product Listing

### Rtx®-CLPesticides Columns

ID	df (µm)	stable to	10m	20m
0.18mm	0.18	340°C	42101	42102
ID	df (µm)	stable to	15m	30m
0.25mm	0.25	340°C	11120	11123
0.32mm	0.50	340°C	11136	11139
0.53mm	0.50	340°C	11137	11140

### Rtx®-CLPesticides2 Columns

ID	df (µm)	stable to	10m	20m
0.18mm	0.14	340°C	42301	42302
ID	df (µm)	stable to	15m	30m
0.25mm	0.20	340°C	11320	11323
0.32mm	0.25	340°C	11321	11324
0.53mm	0.42	340°C	11337	11340

### Rtx®-CLPesticides Column Kits

These kits include both a CLPesticides and CLPesticides2 column, a Universal Angled 'Y' Press-Tight® Connector, and a 5m guard column. (Note: Columns are not preconnected in these kits.)

Description	cat.#
0.53mm ID Rtx®-CLPesticides Kit	11197
0.32mm ID Rtx®-CLPesticides Kit	11198
0.25mm ID Rtx®-CLPesticides Kit	11199

### Organochlorine Pesticide Mix AB #2

aldrin	8µg/mL	dieldrin	16µg/mL
α-BHC	8	endosulfan I	8
β-BHC	8	endosulfan II	16
δ-BHC	8	endosulfan sulfate	16
γ-BHC (lindane)	8	endrin	16
α-chlordane	8	endrin aldehyde	16
γ-chlordane	8	endrin ketone	16
4,4'-DDD	16	heptachlor	8
4,4'-DDE	16	heptachlor epoxide (B)	8
4,4'-DDT	16	methoxychlor	80

In hexane/toluene (1:1), 1mL/ampul.

	each	5-pack	10-pack
	32292	32292-510	—
w/data pack	32292-500	32292-520	32392

### Organochlorine Pesticide Mix C #2

chlorobenzilate	32µg/mL	hexachlorobenzene	8µg/mL
diallate (cis & trans)	80	hexachlorocyclopentadiene	8
1,2-dibromo-3-chloropropane	8	isodrin	8

In hexane/toluene (1:1), 1mL/ampul.

	each	5-pack	10-pack
	32295	32295-510	—
w/data pack	32295-500	32295-520	32395





# CLP Semi-Volatiles OLM 04.1

## New Reference Materials Available

by Eric Steindl, Analytical Reference Materials Product Line Manager

### Standards for the EPA Superfund Contract Lab Program (CLP):

- ✓ Meets latest Statement of Work.
- ✓ Fewest number of calibration solutions possible.
- ✓ Maximum shelf life.

The latest revision to the EPA Superfund Contract Lab Program (CLP) Methods for semi-volatile compounds have necessitated changes in the calibration standards. Restek has developed new mixtures that satisfy all requirements of the OLM 04.1 Methods. These high-quality reference materials are manufactured with raw materials that have been thoroughly tested for purity.

#### CLP 04.1 B/N Matrix Spike Mix

1000µg/mL each in methanol, 1mL/ampul  
acenaphthene  
2,4-dinitrotoluene  
*N*-nitroso-di-*n*-propylamine  
pyrene

each	5-pk.	10-pk.
31492	31492-510	—
w/ data pack		
31492-500	31492-520	31592

#### CLP 04.1 BNA Surrogate Mix

In methylene chloride, 1mL/ampul  
2-chlorophenol-d<sub>4</sub> 1500µg/mL  
1,2-dichlorobenzene-d<sub>4</sub> 1000  
2-fluorobiphenyl 1000  
2-fluorophenol 1500  
nitrobenzene-d<sub>5</sub> 1000  
phenol-d<sub>6</sub> 1500  
*p*-terphenyl-d<sub>14</sub> 1000  
2,4,6-tribromophenol 1500

each	5-pk.	10-pk.
31493	31493-510	—
w/ data pack		
31493-500	31493-520	31593

#### CLP 04.1 Phenols Calibration Mix

2000µg/mL each in methylene chloride, 1mL/ampul  
4-chloro-3-methylphenol  
2,4-dichlorophenol  
2,4-dimethylphenol  
2,4-dinitrophenol  
2-chlorophenol  
2-methyl-4,6-dinitrophenol  
2-methylphenol  
4-methylphenol  
2-nitrophenol  
4-nitrophenol  
pentachlorophenol  
phenol  
2,4,5-trichlorophenol  
2,4,6-trichlorophenol

each	5-pk.	10-pk.
31494	31494-510	—
w/ data pack		
31494-500	31494-520	31594

#### CLP OLM 04.1 SV Kit #1

Contains 1mL each: Restek cat.#  
SV Screening Mix 31000  
SV Tuning Mix 31001  
CLP 04.1 BNA Surrogate Mix 31493  
CLP 04.1 B/N Matrix Spike Mix 31492  
Acid Matrix Spike Mix 31005  
SV Internal Standard Mix 31006  
CLP 04.1 Phenols Calibration Mix 31494  
CLP 04.1 B/N MegaMix 31495  
SV Calibration Mix #6 (pesticides) 31012

each	each w/ data pack
31603	31603-500

#### CLP OLM 04.1 SV Kit #2

Contains 1mL each of: Restek cat.#  
CLP 04.1 Phenols Calibration Mix 31494  
CLP 04.1 B/N MegaMix 31495  
SV Calibration Mix #6 (pesticides) 31012

each	each w/ data pack
31604	31604-500

#### CLP OLM 04.1 SV Kit #3

Contains 1mL each of: Restek cat.#  
CLP 04.1 Phenols Calibration Mix 31494  
CLP 04.1 B/N MegaMix 31495

each	each w/ data pack
31605	31605-500



**New Catalog!**

Restek's new **Purus™ Gas Systems Catalog** details everything to design and maintain your gas delivery system. For your free copy, contact our literature request hotline at 800-356-1688, ext. 5 or contact your local Restek representative. (lit. cat.# 59966)

#### CLP 04.1 B/N MegaMix

1000µg/mL each in methylene chloride/benzene (3:1), 1mL/ampul  
acenaphthene  
acenaphthylene  
acetophenone  
anthracene  
atrazine  
benzaldehyde  
benzo(a)anthracene  
benzo(b)fluoranthene  
benzo(k)fluoranthene  
benzo(ghi)perylene  
benzo(a)pyrene  
biphenyl  
4-bromophenyl phenyl ether  
butyl benzyl phthalate  
di-*n*-butyl phthalate  
caprolactam  
carbazole  
4-chloroaniline  
bis(2-chloroethoxy)methane  
bis(2-chloroethyl)ether  
2-chloronaphthalene  
4-chlorophenyl phenyl ether  
chrysene  
dibenz(a,h)anthracene  
dibenzofuran  
3,3'-dichlorobenzidine  
diethyl phthalate  
dimethyl phthalate  
2,4-dinitrotoluene  
2,6-dinitrotoluene  
bis(2-ethylhexyl)phthalate  
fluoranthene  
fluorene  
hexachlorobenzene  
hexachlorobutadiene  
hexachlorocyclopentadiene  
hexachloroethane  
indeno(1,2,3-cd)pyrene  
isophorone  
2-methylnaphthalene  
naphthalene  
2-nitroaniline  
3-nitroaniline  
4-nitroaniline  
nitrobenzene  
*N*-nitroso-di-*n*-propylamine  
*N*-nitrosodiphenylamine  
di-*n*-octyl phthalate  
2,2'-oxybis-(1-chloropropane)  
phenanthrene  
pyrene

each	5-pk.	10-pk.
31495	31495-510	—
w/ data pack		
31495-500	31495-520	31595



# New High-Performance Molecular Sieve 5A & 13X Packings

## For Improved Gas Separations

by Barry Burger, R&D Chemist, and Michael Feeney, R&D Manager

Molecular sieves, a synthetic form of Zeolite, have been used since the 1950's for separation of light gases (e.g., oxygen, nitrogen, methane, carbon monoxide), and inert gases (e.g., helium, argon, neon, krypton, and xenon). Restek offers the two most common molecular sieves 5A and 13X, which are used in a wide range of packed and PLOT column gas chromatographic analyses. Molecular sieves have become a standard for gas separations, yet until now there have been few changes or improvements in their basic composition or performance. As part of Restek's strategy in developing innovative, high-quality products, we are introducing high-purity, Molecular Sieve 5A and 13X packings.

### Quality Assurance Testing to Ensure Reproducibility

Research of the existing packed column market indicated that chromatographers have experienced problems with the current suppliers of molecular sieve packings and packed columns. Restek goal was to introduce a high-performance molecular sieve packing featuring inertness and reproducibility. This solution requires purification of each lot of Zeolite, to remove metals and other trace contaminants that cause adsorption of trace gases such as carbon monoxide. Each batch of material is then carefully classified to exact mesh ranges to ensure reproducible efficiency, column back pressure, and retention times. Furthermore, our research determined that the thermal conditioning process is critical in determining the relative retention of methane/carbon monoxide and for producing excellent peak symmetry for active compounds such as carbon monoxide. As a result, each lot of molecular sieve is precisely conditioned and quality assurance tested for efficiency, back pressure, peak symmetry and relative retention time using a permanent gas mixture.

### Molecular Sieve 5A or 13X with Silcosteel® Tubing for Permanent Gas Analysis

Restek offers both Molecular Sieve 5A and 13X. These materials differ in pore size and composition, which results in differences in retention and selectivity for many gases. The 5A packing has greater retention, which aids in separation of argon, oxygen, and nitrogen. Therefore, it is a better choice for analysis of trace impurities in inert gases (helium, hydrogen, argon, and nitrogen) used in chromatography or the semiconductor industry. (cont. on page 14)

Figure 1

Molecular Sieve 5A column provides excellent resolution of oxygen, nitrogen, methane, and carbon monoxide.

1m x 1/8" x 2mm ID Silcosmooth™ tubing  
Molecular Sieve 5A 80/100 mesh  
(cat # 80440-800)  
10µL gas sample with 5-10% each component in helium.

Oven temp.: 50°C  
Inj./det. temp.: 150°C/200°C  
Flow: 30mL/min., helium

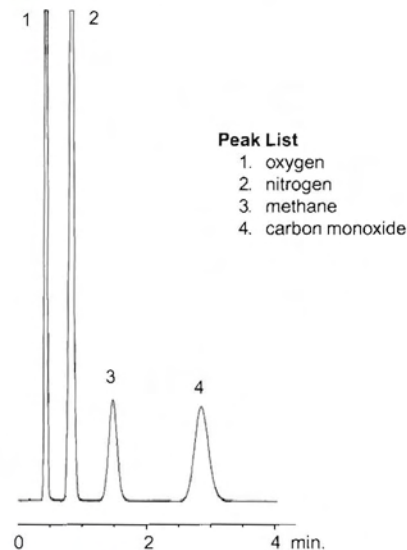
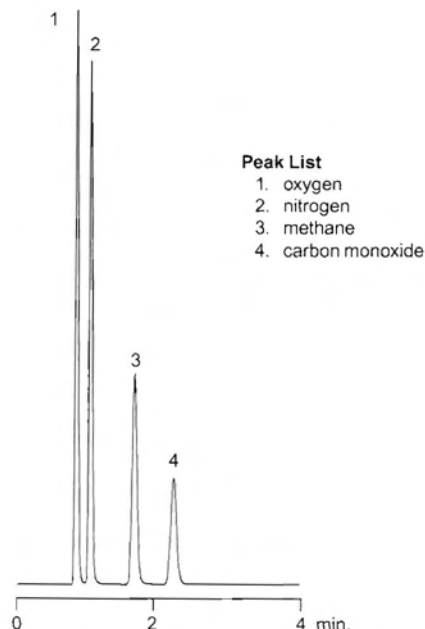


Figure 2

Molecular Sieve 13X column provides rapid analysis and excellent peak symmetry for carbon monoxide.

2m x 1/8" x 2mm (ID) Silcosmooth™ tubing  
Molecular Sieve 13X 80/100 mesh  
(cat # 80439-800)  
10µL gas sample with 5-10% each component in helium.

Oven temp.: 50 °C  
Inj./det. temp.: 150 °C/200°C  
Flow: 30 mL/min., helium





# Peak Performers

by Doug Elliott, GC Accessories Product Line Manager

## Thermolite® Septa

*Achieve the Lowest Bleed at the Most Affordable Prices*

- ✓ Tested for low bleed—Ensures optimum performance.
- ✓ Excellent puncturability—Septa fragments will not interfere with data.
- ✓ Packaged in non-contaminating tins—Ensures product integrity with every shipment.
- ✓ Usable to 340°C inlet temperatures—Analysis of high molecular weight compounds.
- ✓ Preconditioned and ready to use—Saves time.

**free  
septa**

To request a **FREE** sample of **Thermolite®** or **IceBlue™** septa, call 800-356-1688, ext. 3, or contact your local Restek representative.



Thermolite® Septa			
Septum Diameter	25-pk.	50-pk.	100-pk.
5mm (3/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9.5mm (3/8")	20359	20360	20361
10mm	20378	20379	20380
11mm (7/16")	20363	20364	20365
12.5mm (1/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374



**free  
vial  
pack**

Restek has assembled a free trial pack filled with vials, caps, and inserts for you to evaluate. Contact us at 800-356-1688, ext. 5, or contact your local Restek representative to request your free vial pack (lit. cat.# 53076).

## New High-Performance Molecular Sieve 5A & 13X Packings *(continued from pg. 13)*

Molecular Sieve 13X often is preferred for analysis of carbon monoxide, particularly at trace concentrations, because the lower retention results in a sharper chromatographic peak and improved detection limits. Figures 1 and 2 (page 13) compare the same gas mixture analyzed using a 1-meter high-performance Molecular Sieve 5A column with a 2-meter high-performance Molecular Sieve 13X column. Notice the excellent peak symmetry for carbon monoxide, indicating that the packing and column tubing are extremely inert. Combining high-quality molecular sieves with Restek's Silcosmooth™ tubing produces a packed column optimized for trace analysis of the difficult components oxygen and carbon monoxide.

### Restek Offers a Wide Range of Packed Columns and Packings

In addition to offering a wide range of standard packings and packed columns, Restek offers micropacked columns and many unique bonded GC packings. The new high-performance Molecular Sieve 5A and 13X packings are the latest examples of Restek's innovation in providing unique improvements and solutions to solving problems in chromatography.

### Silcosmooth™ Tubing

1/8"OD x 2mm ID: cat.# 21596 (ft.)

3/16"OD x 4mm ID: cat.# 21595 (ft.)

### Molecular Sieve 5A Packings

mesh	cat.#	qty. (g)
45/60	25600	50
60/80	25601	50
80/100	25602	50
100/120	25603	50

### Molecular Sieve 13X Packings

mesh	cat.#	qty. (g)
45/60	25604	50
60/80	25605	50
80/100	25606	50
100/120	25607	50

[www.restekcorp.com](http://www.restekcorp.com)

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# Peak Performers

by Doug Elliott, GC Accessories Product Line Manager

## FastPack™ Inlet Maintenance Kits

For HP GCs

- ✓ **Clean**—Mylar® bag is sealed factory-clean; no aging or contamination from weeks in the lab.
- ✓ **Convenient**—The parts you use in one bag means no hunting for individual parts.
- ✓ **Economical**—Cost less than the quantity catalog price for the individual parts.

FastPack™ Inlet Maintenance Kits are a great way to make performing routine maintenance on your HP inlet easy! The kit includes your preferred deactivated liner with a Viton® O-ring, a 0.8mm ID gold-plated inlet seal, a washer, and an 11mm Thermolite® septa—all sealed in a factory-clean Mylar® bag.



### FastPack™ Inlet Maintenance Kits for HP GCs

1 pack includes 5 maintenance kits

Kit includes: o-ring, inlet seal, washer, septa, & liner  
(liner dimensions: 4mm ID, 6.5mm OD, 78.5mm)

pack of  
5 kits

5 or more  
packs

20 or more  
packs

4mm Splitless cat.# 21101

4mm Splitless Gooseneck cat.# 21102

4mm Splitless Double Gooseneck cat.# 21103

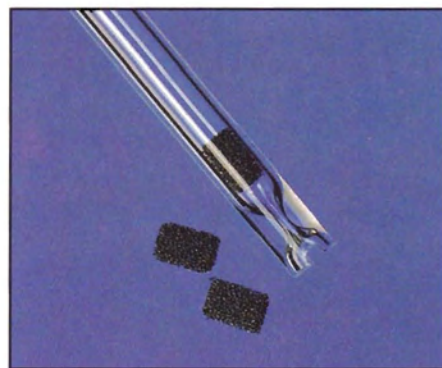
4mm Split with F.S. Wool\* cat.# 21104

\* The 4mm split liner with fused silica wool dimensions are 4mm ID, 6.3mm OD, 78.5mm.

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Frit for liner size ID

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# the Advantage

## Faster, More Accurate Volatile Analysis

### Using 0.45mm ID Rtx®-624 & Rtx®-1 Capillary GC Columns

by Christopher English

There are over a dozen acceptable gas chromatographic (GC) columns for volatile compound analyses on the market today. Selecting a column that truly meets your needs can be difficult and frustrating. Restek has been in the process of developing new stationary phases to resolve a broad range of volatile compounds. During the development, however, it became apparent that existing volatile columns could be optimized for faster analyses and better resolution. By reducing the column ID of our

Rtx®-624 and Rtx®-1 capillary GC columns to 0.45mm, we can decrease analysis time and improve resolution of traditionally coeluting compounds, without sacrificing capacity and carrier gas flow requirements.

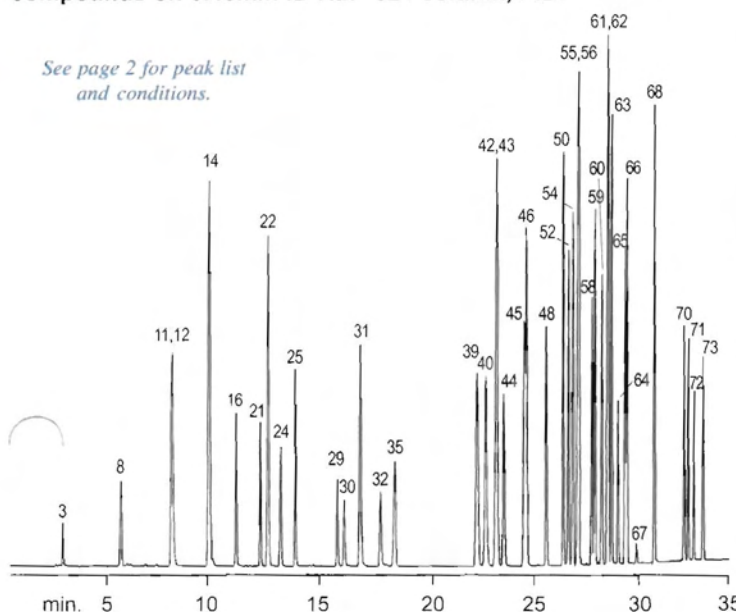
**The Importance of Resolution**  
Volatile analysis methods such as US Environmental Protection Agency (EPA) Method 8021 require use of a photoionization detector (PID) and an electrolytic conductivity detector (ELCD) in series. The most problematic of these two detectors is the ELCD because of the characteristic tailing peaks. Sensitivity can be increased, but not without a sacrifice in peak shape, and the ELCD can exaggerate poor resolution for some compounds of interest.

Most environmental laboratories follow US Environmental Protection Agency (EPA) Method 502.2 for GC analysis of drinking water. The best column must address the compounds that are most commonly found in your samples. For example, water that has been disinfected will have measurable amounts of trihalomethanes (THMs). The four THMs are chloroform, bromodichloromethane,

Figure 1:

Faster analysis and better resolution of volatile organic compounds on 0.45mm ID Rtx®-624 column, PID.

See page 2 for peak list and conditions.



## in this issue

### Faster, More Accurate Volatile Analysis

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### Specialized SPE for Wastewater Extractions

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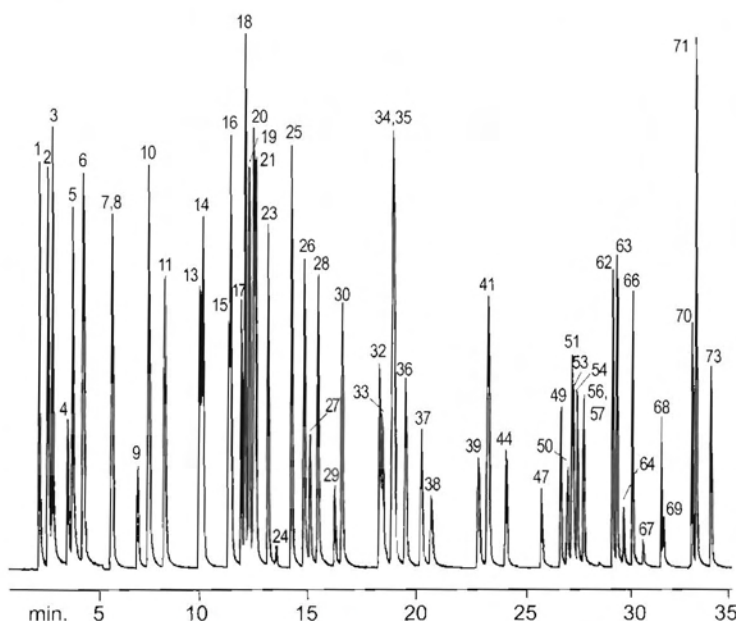
# Faster, More Accurate Volatile Analysis (continued from page 1)

dibromochloromethane, and bromoform (listed in descending order of concentrations typically found in municipal drinking water). For quantitation, these compounds must be well resolved from the other 60 analytes listed in US EPA Method 502.2.

Most states in the US require the analysis of a performance evaluation (PE) sample prior to running drinking water samples for regulatory purposes. Columns that cannot resolve THMs from other target compounds in the calibration will produce poor quantitation and ultimately result in failure of the PE. Acceptable resolution for THMs is obtained when the valley between a pair of peaks is at least 25% of the average of the two peak heights. Quantitative analysis must be done using a column that can meet this criteria. Laboratories should test their own proficiency by analyzing a check standard with the surrogates and THMs, following a passing calibration with the full target list. Values for the check standard calculated from the curve must be within 20% of their "true" value. Currently, there are no GC columns that can offer a 25% valley separation for all compounds in US EPA Methods 8021A, 8021B, and 502.2, however, finding a column that addresses the target compounds most common in your real-world samples is the key to obtaining accurate data.

**Figure 2:**

**Good selectivity for THMs using an 0.45mm ID Rtx®-624 Column, ELCD.**



## Run Conditions for Figures 1-3

<b>Concentrator:</b>	Tekmar® LSC-3000 Purge and Trap
<b>Trap:</b>	VOCARB™ 3000
<b>Purge:</b>	11 min. @ 40mL/min.
<b>Dry purge:</b>	1 min. @ 40mL/min. (Moisture Control System OFF)
<b>Desorb preheat:</b>	245°C
<b>Desorb:</b>	250°C for 2 min.
<b>Bake:</b>	260°C for 8 min.
<b>Interface:</b>	direct
<b>Transfer line:</b>	0.45mm ID fused silica (5-Meter Length, cat.# 10071, \$60; 10-Meter Length, cat.# 10072, \$120)
<b>GC:</b>	Finnigan 9001
<b>Oven temp:</b>	35°C (hold 9 min.) to 90°C @ 17°C/min. (hold 10 min.), 220°C @ 14°C/min. (hold 5 min.)
<b>Carrier gas:</b>	helium
<b>Flow rate:</b>	9mL/min.
<b>µGold Tandem PID:</b>	Makeup: 7mL/min.; Purge: 7mL/min., set @ 0.35mV base temp 200°C.
<b>Hall 2000 ELCD:</b>	Reaction gas: hydrogen, 25mL/min.;
<b>Reactor temp.:</b>	940°C; Propanol flow: 470µL/min.

## The New 0.45mm ID Columns Improve Resolution and Save Time for Volatiles Analyses

At Restek, we have optimized dimensions and analysis conditions for columns currently used for volatile analysis: the Rtx®-624 and the Rtx®-1 capillary GC columns. In Figures 1 through 3 you will find chromatograms using optimized analysis conditions and column parameters. The 0.45mm internal diameter (ID) of these columns enhances resolution and decreases the analysis time. Desorb flow rates of 9mL/min. effectively sweep the volatile compounds off of the trap, and help maintain tight bands for the gases and other early eluting compounds. Analysis time has been decreased by almost 20 minutes compared to using the same stationary phase on a 0.53mm ID column.

The PID and the ELCD were run in series to obtain two chromatograms (Figures 1 and 2). Figure 1 shows an analysis on the Rtx®-624 column (75m, 0.45mm ID, 2.55µm) using a PID. The greatest disadvantage of the 624 phase has been the coelution of o-xylene and styrene. Using the conditions and column dimensions shown for Figures 1-3, resolution is possible between these compounds (Figure 1, peaks 45 and 46). This 0.45mm ID column offers an excellent alternative to other GC phases for volatile analysis. Figure 2 demonstrates good selectivity using an ELCD for the THMs in EPA Method 8021A, as well as the compounds listed in the 8021B.

Volatile compounds also can be found as subsets of Method

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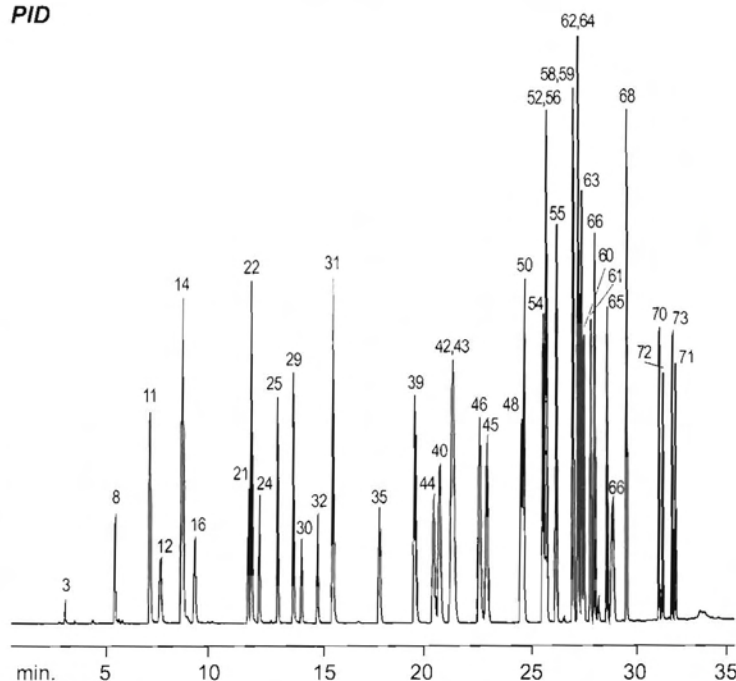


38021 in the following EPA Methods: 501, 504, 502.2, 601, 602, 8010, and 8020. The five surrogates suggested by these methods were added to the compound list. The best surrogate compounds under the conditions shown are fluorobenzene, 1-chloro-2-fluorobenzene, and 1,4-dichlorobutane. Customer feedback resulted in the addition of Freon® 113 and methyl-tert-butyl-ether (MTBE). These compounds are commonly analyzed in addition to the method-specific target analytes. Figure 3 shows confirmation analysis on the Rtx®-1 column (75m, 0.45mm ID, 2.55µm), which provides a different selectivity, using PID/ELCD under the same conditions as the Rtx®-624 chromatograms in Figures 1 and 2. The recently optimized column dimensions and GC conditions for the Rtx®-624 and Rtx®-1 capillary GC columns achieve decreased analysis time and improved resolution for volatile compound analyses.

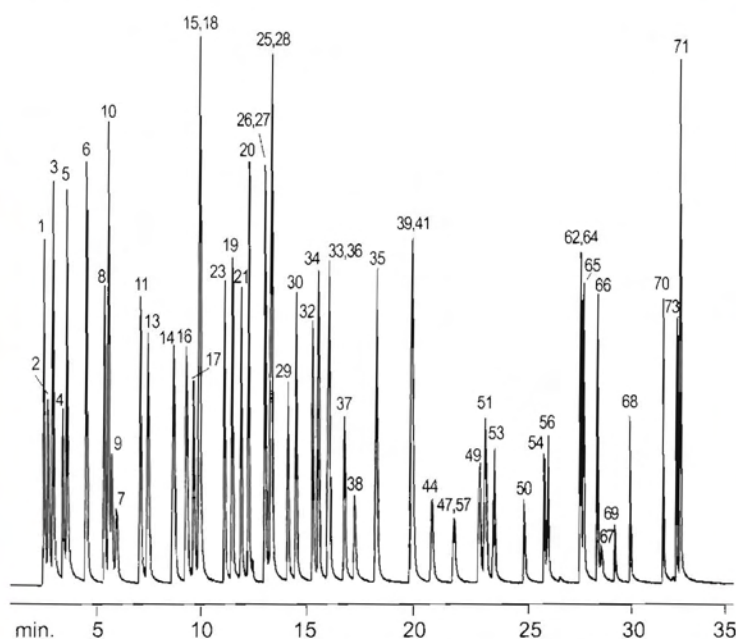
**Figure 3:**

The different selectivity provided by the Rtx®-1 column makes it an excellent confirmation to the Rtx®-624 column.

**PID**



**ELCD**



# **Peak List for Figures 1-3**

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. Freon® 113
8. 1,1-dichloroethene
9. allyl chloride
10. methylene chloride
11. trans-1,2-dichloroethene
12. methyl-tert-butyl-ether
13. 1,1-dichloroethane
14. chloroprene
15. 2,2-dichloropropane
16. cis-1,2-dichloroethene
17. bromochloromethane
18. chloroform
19. 1,1,1-trichloroethane
20. carbon tetrachloride
21. 1,1-dichloropropene
22. benzene
23. 1,2-dichloroethane
24. fluorobenzene
25. trichloroethene
26. 1,2-dichloropropane
27. dibromomethane
28. bromodichloromethane
29. 2-chloroethylvinyl ether
30. cis-1,3-dichloropropene
31. toluene
32. trans-1,3-dichloropropene
33. 2-bromo-1-chloropropane
34. 1,1,2-trichloroethane
35. tetrachloroethene
36. 1,3-dichloropropane
37. dibromochloromethane
38. 1,2-dibromoethane
39. chlorobenzene
40. ethyl benzene
41. 1,1,1,2-tetrachloroethane
42. m-xylene
43. p-xylene
44. 1-chloro-2-fluorobenzene
45. o-xylene
46. styrene
47. bromoform
48. isopropyl benzene
49. 1,4-dichlorobutane
50. bromobenzene
51. 1,1,2,2-tetrachloroethane
52. n-propyl benzene
53. 1,2,3-trichloropropane
54. 2-chlorotoluene
55. 1,3,5-trimethylbenzene
56. 4-chlorotoluene
57. 1,3-dichloro-2-propanol (40ppb)
58. tert-butylbenzene
59. 1,2,4-trimethylbenzene
60. sec-butylbenzene
61. p-isopropyl toluene
62. 1,3-dichlorobenzene
63. 1,4-dichlorobenzene
64. benzyl chloride
65. n-butylbenzene
66. 1,2-dichlorobenzene
67. bis-(2-chloroisopropyl) ether (40ppb)
68. 4-bromo-1-chlorobenzene
69. 1,2-dibromo-3-chloropropane
70. 1,2,4-trichlorobenzene
71. hexachlorobutadiene
72. naphthalene
73. 1,2,3-trichlorobenzene

## **Product Listing:**

### **Column Description**

cat.#	price
Rtx®-624	
75m, 0.45mm ID, 2.55µm	
cat #10982	\$900
Rtx®-1	
75m, 0.45mm ID, 2.55µm	
cat #10992	\$900
<b>Transfer Line</b>	
cat.#	price
5m, 0.45mm ID	
cat #10071	\$60
10m, 0.45mm ID	
cat #10072	\$60

Restek also offers a full line of volatile component standards, and custom mixes are available. Please contact our Technical Service chromatographers.

### **Acknowledgement**

Finnigan 9001 GC, µGold Tandem Photoionization Detector and Hall 2000 Detector provided through courtesy of ThermoQuest/CE Instruments, 2215 Grand Avenue Parkway, Austin Texas 78728.

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# Optimizing the Massachusetts Extractable Petroleum Hydrocarbon Method

by Christopher English

Soil and water contamination as a result of petroleum spills are the primary sources of environmental pollution in the United States. Measures to identify and cleanup these sites have proven difficult. Massachusetts is one of the leading states in total petroleum hydrocarbon (TPH) regulation with their recent publication of two methods for the identification of gasoline contamination: extractable petroleum hydrocarbons (EPH) and volatile petroleum hydrocarbons (VPH). The EPH method focuses on areas that early methods have failed to address such as weathering of the sample, identifying specific compounds, and allowing estimates of overall toxicity of diesel range organics (DRO).

## Sample Preparation and Analysis

EPH samples are extracted in methylene chloride, dried with sodium sulfate, and solvent-exchanged into hexane. A Kuderna-Danish (KD) apparatus is used to concentrate the sample

prior to cleanup. Silica gel tubes are used for cleanup, and the sample is fractionated into aliphatic and aromatic extracts. These two extracts then are analyzed using a flame ionization detector (FID). The aliphatic range of compounds are collectively calculated between the ranges of C9 to C18 and C19 to C36 (Figure 1), while the aromatic range is from C11 to C36 and allows the identification of specific polyaromatic hydrocarbons (Figure 2).

Samples are fractionated using solid phase extraction (SPE) silica gel tubes. In this method, silica gel is used to separate petroleum fractions into aliphatic and aromatic extracts. The most difficult part of EPH analysis is consistency and attention to detail during this fractionation step. Surrogates are added prior to extraction to monitor the efficiency of the fractionation procedure (Figure 3). The method recommends using a commercially prepared tube to avoid introducing

variables to the extraction procedure. Contamination (i.e., moisture in the tubes) will adversely effect the fractionation of the two extracts. Other factors that may prevent proper quantitation of field samples include: sample matrixes, amount of hexane used, variability of lab-packed columns, inconsistent pre-rinsing, silica gel variability, and phthalate contamination.

## Contamination

To prevent phthalate contamination, rinse glassware thoroughly and install carbon traps at the end of gas lines. Silica gel tubes suspected of having contaminants should be rinsed with methylene chloride, followed by hexane, prior to the addition of the extract. Restek offers SPE tubes specifically designed for the EPH section of the method. These tubes have exceptionally low background and provide excellent reproducibility.

## Column Selection

The EPH method recommends using the Restek Rtx®-5 column

(30m, 0.32mm ID, 0.25µm). Because the method can be used to identify specific polyaromatic hydrocarbons (PAHs), compound separation is critical. Closely eluting compound pairs such as benzo(b)fluoranthene/benzo(k)fluoranthene and indeno(1,2,3-cd)pyrene/dibenzo(a,h)anthracene must have 50% to baseline separation in order to meet the criteria set by the method. When using optimized flows, Restek's Rtx®-5 column provides better than 80% resolution of the benzo(b)fluoranthene/benzo(k)fluoranthene pair—the most difficult separation for the EPH method (Figure 4). Column bleed is minimal, resulting in excellent quantitation of the late eluting compounds. Using the appropriate sample preparation procedure and optimized analysis conditions described here, combined with using an Rtx®-5 column, will ensure a trouble-free EPH analysis and result in a high degree of accuracy.

Figure 1:

The Rtx®-5 column provides excellent separation of the EPH aliphatic compounds and minimal bleed.

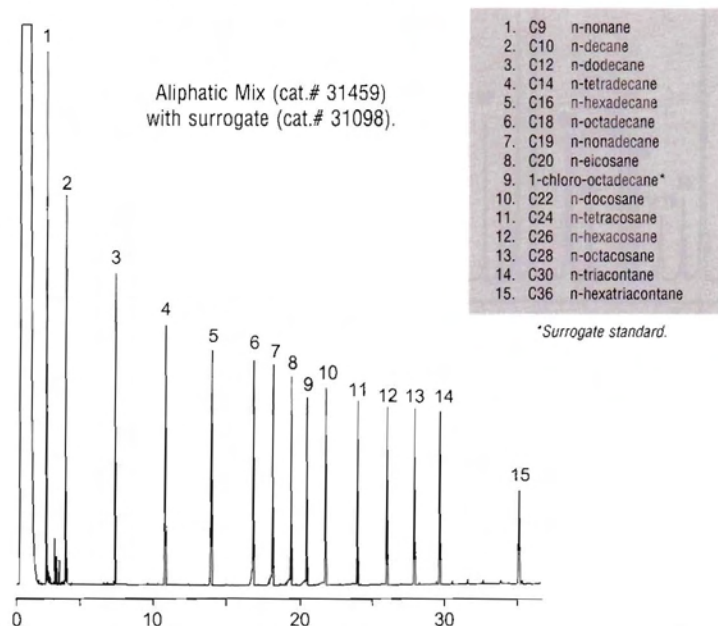
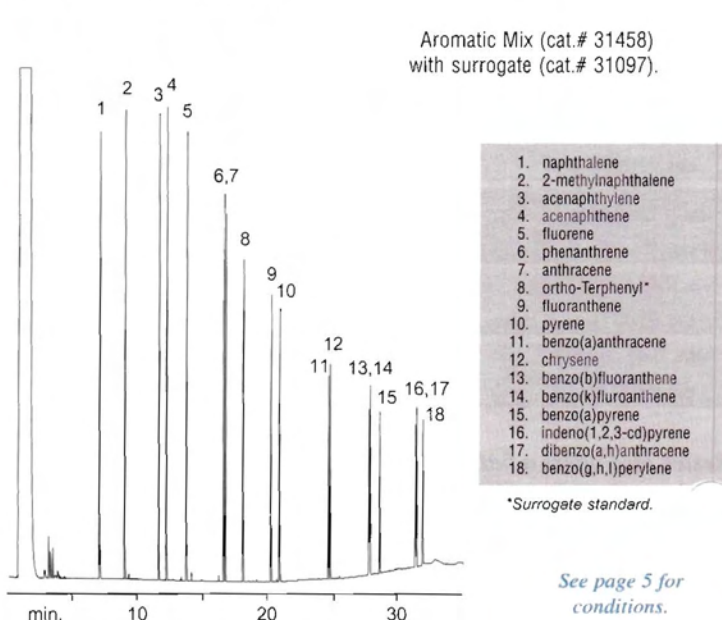


Figure 2:

The Rtx®-5 column provides good resolution of the aromatic calibration compounds.



See page 5 for conditions.

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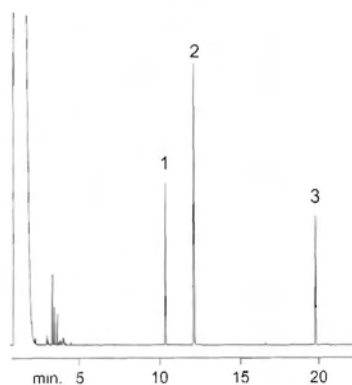
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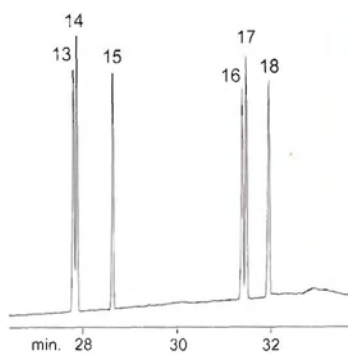


Efficiency of the fractionation standards (cat.# 31480) are monitored using an internal standard (cat.# 31065).



1. 2-fluorobiphenyl  
2. 2-bromonaphthalene  
3. 5-alpha-androstane

The Rtx®-5 column provides better than 80% resolution of benzo(b) and benzo(k).



For peak IDs, see Figure 2.

#### Conditions for Figures 1-4

30m, 0.32mm ID, 0.25µm Rtx®-5 (cat.# 10224). **Concentration:** 100ng/µL on-column; **Injection:** manual, solvent flush technique, 1µL, direct. **Oven temp:** 60°C (hold 1 min.) to 305°C @ 8°C/min. (hold 4 min.); **Inj./det. temp.:** 285°C/315°C; **Carrier gas:** helium; **FID sensitivity:** 2.56 x 10E-10 AFS; **Linear velocity:** 76.9cm/sec. @ 60°C.

#### Product Listing:

Rtx®-5 Capillary GC Columns	cat. #	price
30m, 0.25mm ID, 0.25µm	10223	\$385
30m, 0.32mm ID, 0.25µm	10224	\$415
30m, 0.53mm ID, 0.50µm	10240	\$465
<b>Uniliner® Direct Injection Sleeve for HP GCs*</b>		
Fits 0.32-0.53mm ID, each	20335	\$54
Fits 0.32-0.53mm ID, 5-pk.	20336	\$198
<b>MA TPH SPE Tubes</b>		
20mL, 5g Silica, 20-pk.	26065	\$95
<b>MA Fractionation Check Mix</b>		
each	5-pk.	10-pk.
31481 \$40	31481-510 \$180	----
w/data pack 31481-500 \$50	31481-520 \$200	31481 \$360

\*For a complete list of Uniliner® inlet sleeves, see the annual Product Guide.

For a complete list of standards for underground storage tank (UST) analyses, please request Lit. Cat. #59617.

## Specialized SPE Products for Environmental Wastewater Extractions

### Resprep™ Oil & Grease Extraction Disks

by Lydia Nolan

- Reduce the use of dangerous & expensive CFCs
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Increasing environmental concerns about the effects of released chlorofluorocarbons (CFCs) in the atmosphere, and in an effort to reduce discharges during routine laboratory procedures, the US Environmental Protection Agency (EPA) has mandated the elimination of Freon® (CFC-113) extraction solvent for industrial waste samples. This has recently resulted in the release of an alternative procedure for US EPA Method 413.1—the US EPA Method 1664, Oil and Grease Analysis in Wastewater. The updated 1664 method describes an n-hexane liquid-liquid extraction and allows use of alternative extraction and concentration

techniques such as SPE if the performance specifications of the method are not met.

The n-hexane extraction process described in the method has several difficulties. As with all liquid-liquid extractions, emulsions are problematic, but with industrial discharge samples this problem may become extreme, resulting in poor partitioning or samples too viscous to process at all. In addition, the n-hexane is lighter than water, making routine separatory funnel processes difficult to manage. Some of these difficulties may be overcome using an SPE procedure.

By decanting the supernatant from the sample first and using an appropriate prefilter, most complex samples can be extracted in less than an hour. The SPE system also can be automated using vacuum manifolds, which allow simulta-

neous processing of up to six samples, thereby improving sample throughput and lab efficiency.

For a complete description of the SPE extraction method, please request lit. cat.# 59800.

#### Product Listing:

Description	cat. #	price
Oil & Grease SPE Disk, 47mm (20-pk.)	26022	\$75
Flow Filter, 47mm (20-pk.)	26024	\$14
<b>Maxi-Manifold</b>		
Complete Manifold (6 positions)	26026	\$3250
1 Station (1 position)	26025	\$1045
Individual Stations (add-on)	26027	\$500
<b>Dry Prep, Sodium Sulfate Drying Tube</b>		
Dry Prep I, small (50-pk.)	26075	\$80
Dry Prep II, large (50-pk.)	26076	\$85
25mm Syringe Tip Filter, PTFE, 0.45µm (50-pk.)	26073	\$120
US EPA Method 1664 Oil & Grease Mix	31457	\$25
w/Data Packet (5-pk.)	31457-510	\$112.50

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# GC/MS Analysis of Phthalate Ester Endocrine Disrupters\*

by Frank Dorman, Ph.D., & Christopher Cox

*\*Part of a series on endocrine disrupters. See the Fall 1997 Restek Advantage for analysis of organo tin compounds, and the Fall 1998 Restek Advantage for analysis of alkyl phenols and bisphenol A compounds.<sup>1,2</sup>*

This article will address the analysis of a series of phthalate ester endocrine disrupters using gas chromatography/mass spectrometry (GC/MS) and a benchtop quadrupole detector. We can achieve the lowest reasonable detection limit using common instrumentation.

Several countries are actively investigating the effects of endocrine disrupters, and beginning to research potential methods of preparation and analysis.<sup>3,4</sup> In addition, these compounds have become a focal point of several recent chemistry conventions, including the 1998 American Chemical Society national meeting. However, there are no accepted or standard methods of analysis, and there is still considerable disagreement as to which specific compounds should be studied and in what order they should be prioritized.

Phthalate compounds have long been used in the plastics industry and, until very recently, were not monitored except in a very few cases. Since the discovery that these compounds can exhibit estrogenic activity, they have become of great interest. Additionally, because some of these compounds were not regulated, they often are found in the environment, which helps to further intensify the interest with the public.<sup>5</sup> We will demonstrate a method for the analysis of phthalate esters

that achieves a reasonably low limit of detection, and is adaptable to common laboratory instrumentation and preparation techniques.

Although no US Environmental Protection Agency (EPA) method exists for phthalate esters at these detection limits, it is possible to perform the preparation and analysis with equipment commonly found in an environmental laboratory. The compounds can be extracted under neutral conditions using US EPA Method 3510 (separatory funnel) or 3520 (liquid-liquid) for water samples, and US EPA Method 3540 (soxhlet) or 3550 (sonication) for solid matrices. These methods produce a methylene chloride extract, which is dried using granular sodium sulfate and then concentrated to a final volume. For biota or soil samples with lipid or other high molecular weight contaminants, gel permeation chromatography (GPC) should be used to clean the extracts prior to final concentration.

Because all of the phthalate compounds share a common mass-to-charge ion of 149, except dimethyl phthalate at 163, the best detection limit will be obtained by operating the MS in single ion monitoring (SIM) mode. This will typically yield a 10-fold or greater increase in sensitivity over a full scan acquisition.

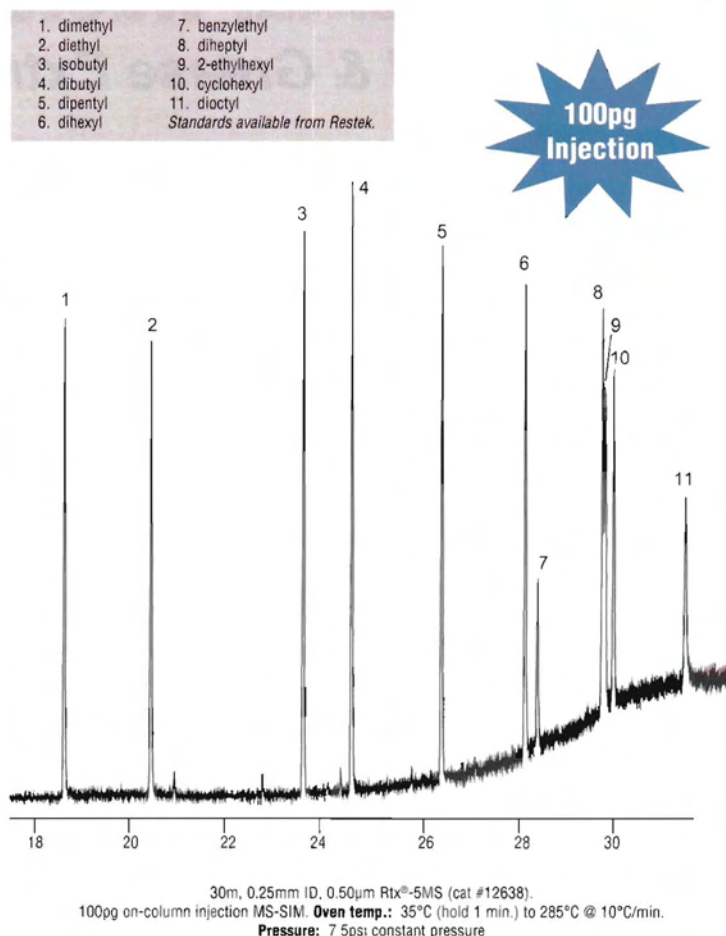
Figure 1 shows the GC/MS analysis obtained from a 100pg injection of the phthalate esters. Mass-to-charge ion 163 is monitored until 19 minutes, after which mass-to-charge 149 is monitored for the duration of the run. It should be noted that this represents a factor of 200 lower concentration than would typically be analyzed by US EPA Method 8270.

Figure 2 shows a GC/MS analysis obtained from a 20pg

injection of the same compounds. This represents nearly a 1000-fold lower concentration than US EPA Method 8270! This concentration level was used to obtain method detection limits (MDLs) for these compounds. Seven replicate analyses are performed, and the resulting MDL was calculated to be less than 10pg for each compound, with the later-eluting compounds producing a higher MDL due to splitless injection port discrimination. These analyses were

**Figure 1:**

**Rtx®-5MS column and optimized conditions analyze a concentration 200-times lower than typical US EPA Method 8270.**



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performed using an HP 5890 GC and a 5971A MS. Limits of detection should improve with a 5973 MS or an ion-trap, because these newer detectors offer increased sensitivity.

For additional information, please contact Restek's

technical service at 1-800-356-1688, ext 4. Restek also offers the analytical standards for these compounds at concentrations to suit your application. For information, please ask for our chemical standards group.

#### Product Listing:

Description	cat. #	price
Rtx®-5MS (30m, 0.25mm ID, 0.50µm)	12638	\$415

For custom standards, please call 800-356-1688, ext. 3, or contact your local Restek representative.

#### References

1. *The Restek Advantage*, Fall 1997.
  2. *The Restek Advantage*, Fall 1998.
  3. Special Report on Environmental Endocrine Disruption: An Effects Assessment and Analysis, EPA/630/R-96/012, Feb. 1997.
  4. JAIMA 1998, Tokyo, Japan.
  5. *The Boston Globe*, Vol. 255, # 58, p. 1, 1998.
- References 3, 4, and 5 not available from Restek.

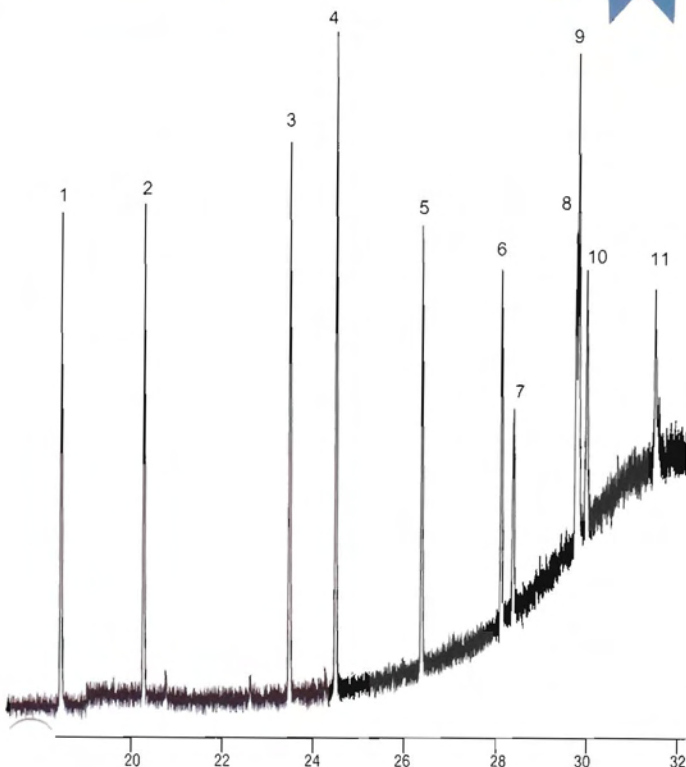
Figure 2:

Rtx®-5MS column and optimized conditions analyze a concentration 1000-times lower than typical US EPA Method 8270.

1. dimethyl	7. butylbenzyl
2. diethyl	8. diheptyl
3. diisobutyl	9. bis-2-ethylhexyl
4. dibutyl	10. dicyclohexyl
5. dipentyl	11. dioctyl
6. dihexyl	

Standards available from Restek

20pg  
Injection



30m, 0.25mm ID, 0.50µm Rtx®-5MS (cat #12638). 20pg on-column injection MS-SIM  
Oven temp.: 35°C (hold 1 min.) to 285°C @ 10°C/min.  
Pressure: 7.5psi constant pressure

## Instrument-Grade Stainless Steel and Copper Tubing

Meets or exceeds oxygen service cleanliness guidelines for CGA Method G-4.1

Now even better!



No chlorinated solvents.

When you want the cleanest tubing for plumbing your analytical instrument system, and you also are concerned about the quality of the environment and worker safety, choose Restek Instrument-Grade Tubing.

We know how important clean tubing for plumbing is to the successful delivery of pure gas to your instrument. We also know that we have a responsibility to protect the environment and our employees. For those reasons, Restek has developed a new cleaning process that uses biodegradable detergents, solubilizers, penetrants, organic acids, and non-toxic solvents for superior surface cleaning—leaving no film or residue. The cleaning performance of this process equals that of chlorinated solvents without the well-publicized hazards to our environment.



Purus® Gas Systems from Restek

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## Two chromatographers put Restek's Rtx®-CLPesticides

## A Low-Cost, High-Resolution Solution for Chlorinated Pesticides Analysis.

Jeff Westerlund, Sound Analytical Services, Tacoma, WA

Jeff Westerlund recently has begun using Restek's Rtx®-CLPesticides and Rtx®-CLPesticides2 columns for US Environmental Protection Agency (EPA) Method 8081 chlorinated pesticide analysis. He also set up his gas chromatograph (GC) using nitrogen as a carrier gas. He chose nitrogen because it is available at high-purity levels, but is much less expensive than helium. Nitrogen is not typically recommended as a carrier for GCs because of its low efficiency. Because the Rtx®-CLPesticides stationary phases were designed with a high selectivity for chlorinated pesticides, carrier gas efficiency may be sacrificed without greatly affecting separation.

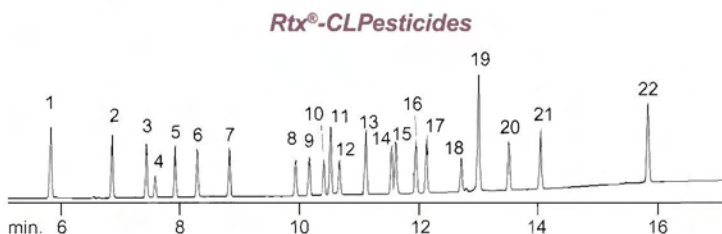
*"In my 11 years of experience as a chromatographer working in the environmental industry, I have concentrated on method improvements while keeping a focus on controlling or lowering costs. It is important that in the process of lowering costs, you get approval from auditing agencies, or in other words, you don't sacrifice data quality. I have been interested in the use of nitrogen as a carrier gas for some time because it is available at low cost and high purity, but the sacrifice in separation using previous columns limited its use.*

*When I found the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns from Restek, I was excited to learn that they had been designed specifically for the selectivity of chlorinated pesticides. I tried the columns and immediately thought they might show promise for use with nitrogen carrier gas. I plumbed my GC (Varian 3400) with nitrogen and, after some development work, produced the following chromatograms (Figure 1).*

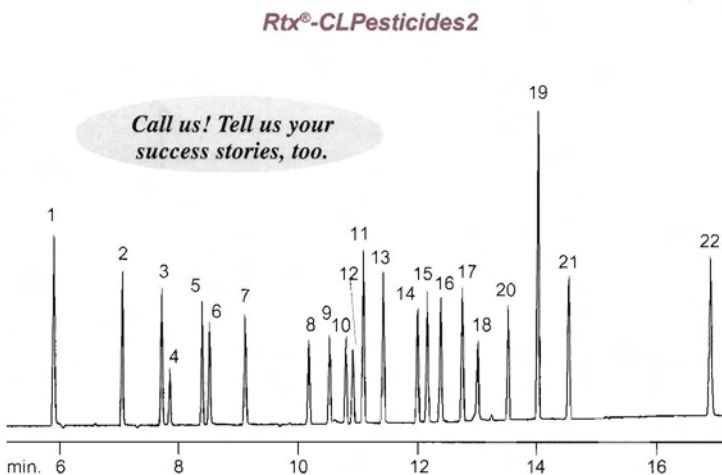
*These chromatograms show that my total run time was less than 17 minutes for decachlorobiphenyl to elute on*

Figure 1:

Fast, high resolution of chlorinated pesticides on 0.32mm ID Rtx®-CLPesticides and Rtx®-CLPesticides2 columns.



- |                                 |                       |                        |
|---------------------------------|-----------------------|------------------------|
| 1. 2,4,5,6-tetrachloro-m-xylene | 8. heptachlor epoxide | 16. endosulfan II      |
| 2. α-BHC (a-HCH)                | 9. γ-chlordane        | 17. 4,4'-DDT           |
| 3. γ-BHC (lindane)              | 10. α-chlordane       | 18. endrin aldehyde    |
| 4. β-BHC (b-HCH)                | 11. 4,4'-DDE          | 19. methoxychlor       |
| 5. δ-BHC (d-HCH)                | 12. endosulfan I      | 20. endosulfan sulfate |
| 6. heptachlor                   | 13. dieldrin          | 21. endrin ketone      |
| 7. aldrin                       | 14. endrin            | 22. decachlorobiphenyl |
|                                 | 15. 4,4'-DDD          |                        |



Chromatogram courtesy of Sound Analytical Services.  
Conditions are proprietary.

both columns. Also, baseline separation of all the target compounds was maintained with the exception of greater than 80% between endrin and DDD on the Rtx®-CLPesticides column. We currently are using this instrumental setup for the analysis of samples for US EPA Methods 8081 and 8082, and

have been very happy with the performance of this system. This analysis also would have advantages for application to field GC instruments, as you would only have to carry nitrogen for the instrument. This would eliminate at least one tank or generator, while minimizing cost."



Jeff Westerlund, Sound Analytical

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# 9 s into Reality

## Columns & Rtx<sup>®</sup>-CLPesticides2 columns to the test!

The Dioxin & Toxic Organics Section of the Ontario Ministry of the Environment (MOE) has been investigating decreasing analysis time by fast gas chromatography (GC) through the use of narrow bore columns (0.18 and 0.10 mm ID). Reducing analysis time increases sample throughput and decreases costs. The limiting factor in reducing analytical run times is maintaining good analyte separation. Conventional organochlorine pesticide analysis uses 5% phenyl/50% phenyl columns and the separation requires an analysis time of about one hour.

MOE has developed a fast GC method for organochlorine pesticides using the Rtx<sup>®</sup>-CLPesticides and Rtx<sup>®</sup>-CLPesticides2 columns. The previous analysis that took 55 minutes is now completed in just over 10 minutes, with an improvement in resolution of the target compounds. Another advantage of the Rtx<sup>®</sup>-CLPesticides and Rtx<sup>®</sup>-CLPesticides2 columns is their thermal stability.

Higher thermal stability allows use of higher final oven temperatures, which drives off any high-boiling contaminants that may make it through our rigorous cleanup process. There also is virtually no column bleed. This, along with the thermal stability, indicate the lifetime of these columns are longer than those used previously. Over 2000 soil, biota, and vegetation samples have been analyzed on these columns without a reduction in performance.

The use of Fast GC and analyte-specific columns has reduced analysis times for organochlorine pesticides to about 10 minutes, or a factor of 5 in time savings. This also translates into an increase in sample throughput by 5-fold, without adding any additional instrumentation to the lab.

If you would like additional information on the Restek Rtx<sup>®</sup>-CLPesticides and Rtx<sup>®</sup>-CLPesticides2 columns, please contact Restek's Technical Service Department or your local Restek Distributor; or visit our web site at [www.restekcorp.com](http://www.restekcorp.com).

Request the new 24-page "Guide to Preparing and Analyzing Chlorinated Pesticides" (Lit. Cat.# 59892).

## Fast GC Analysis of Organochlorine Pesticides Using Analyte-Specific Columns

*Eric Reiner, Ontario Ministry of the Environment, Ontario, Canada*

### Product Listing:

Rtx <sup>®</sup> -CLPesticides Columns	15-Meter cat.#/price	30-Meter cat.#/price
0.18mm ID, 0.18µm	42101*/\$265	42102**/\$385
0.25mm ID, 0.25µm	11120/\$290	11123/\$445
0.32mm ID, 0.50µm	11136/\$310	11139/\$475
0.53mm ID, 0.50µm	11137/\$325	11140/\$525
Rtx <sup>®</sup> -CLPesticides2 Columns	15-Meter cat.#/price	30-Meter cat.#/price
0.18mm ID, 0.18µm	42301*/\$265	42302**/\$385
0.25mm ID, 0.20µm	11320/\$290	11323/\$445
0.32mm ID, 0.25µm	11321/\$310	11324/\$475
0.53mm ID, 0.42µm	11337/\$325	11340/\$525

\*0.18mm ID Columns are 10 meters in length.

\*\*0.18mm ID Columns are 20 meters in length.

### CLP Pesticide Standards

#### Organochlorine Pesticide Mix AB #2

aldrin	8µg/mL	dieldrin	16µg/mL
α-BHC	8	endosulfan I	8
β-BHC	8	endosulfan II	16
δ-BHC	8	endosulfan sulfate	16
γ-BHC (lindane)	8	endrin	16
α-chlordane	8	endrin aldehyde	16
γ-chlordane	8	endrin ketone	16
4,4'-DDD	16	heptachlor	8
4,4'-DDE	16	heptachlor epoxide (B)	8
4,4'-DDT	16	methoxychlor	80

At concentration listed in hexane/toluene (1:1), 1mL/loop

	Each	5-pk.	10-pk.
	32292 \$25	32292-510 \$112.50	—
w/data pack	32292-500 \$35	32292-520 \$125	32392 \$225



*Eric Reiner, Ontario Ministry of the Environment*

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# OPN Res-Sil™ C Packing

## Restek's Newest Bonded Phase for Packed Column GC

by Barry Burger

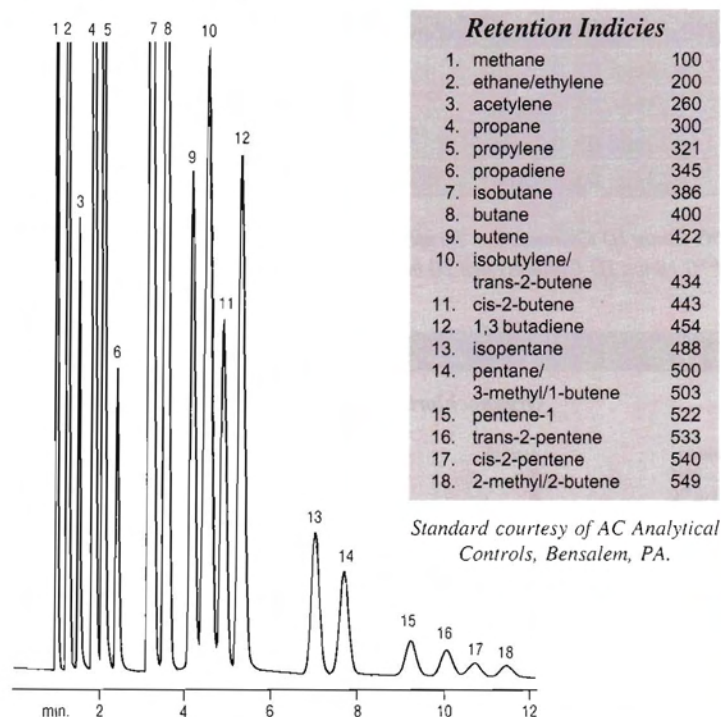
- Unique separation of saturated and unsaturated hydrocarbons
- Innovative bonding chemistry for batch-to-batch reproducibility, excellent thermal stability, and long life
- Wide range of bonded phases available
- Equivalent to Waters Durapak® packings

For over 25 years the process GC and petrochemical industries have used bonded silica packings such as Waters Durapak® packing for analysis of C1 to C4 hydrocarbons. These phases provide unique selectivity by modifying silica with a covalent attachment of either n-octane or cyano-propyl (OPN) functional groups. These phases have many advantages over conventional gas liquid chromatography packings because they

yield faster separations, higher thermal stability, shorter conditioning times, and longer lifetimes. In the past, these packings had inconsistent reproducibility and limited availability. Restek's research team has solved these age old problems by developing new Res-Sil™ C packings for consistent batch-to-batch performance and immediate delivery.

Figure 1:

OPN on Res-Sil™ C packing demonstrates unique selectivity for cis-2-butene and 1,3-butadiene.



Standard courtesy of AC Analytical Controls, Bensalem, PA.

12' x 2mm ID x 1/8" OD Silcosteel® column packed with OPN on Res-Sil™ C 80/100 mesh.  
20µL on-column injection of refinery gas

Oven Temp.: 50°C Inj. temp.: 200°C Det. temp.: 200°C Flow rate: 30mL/min., He

### Unique Selectivity for Process GC and High-Speed Analysis of Petrochemicals

Speed of analysis is crucial for process GC, and in laboratory gas analyzers using multiple columns and valve switching for separation of complex gas mixtures. The Res-Sil™ C bonded packings are ideal for resolution of the saturated and unsaturated C4 hydrocarbons that are difficult to separate. The chromatogram in Figure 1 demonstrates the unique selectivity for separation of cis-2-butene before 1,3-butadiene using the OPN on Res-Sil™ C packing. This unique selectivity, when combined with other columns in series, provides petroleum and petrochemical method developers with a powerful tool for fast determination of C1 to C5 hydrocarbons.<sup>1</sup>

### Innovative Research and Stringent QA Provide Batch-to-Batch Consistency

Historically, one of the problems with bonded phases such as Carbowax®, n-octane, and OPN on Porasil® packing has been batch-to-batch variations in the amount of liquid stationary phase added to solid silica support. Restek's product development team pulled together chemists with experience in GC packings and HPLC phase development to innovate a new synthesis procedure. Utilizing new synthesis pathways, the amount of bonded liquid phase is precisely

controlled in every batch, resulting in reproducible retention times and separations. Each production batch of Res-Sil™ C packing is quality assurance tested with a complex hydrocarbon mixture to meet demanding retention time and retention index specifications. Column bleed also is evaluated at the recommended maximum temperature of 150°C, as part of the QA test to ensure that retention shifts and high baselines are not observed.

### OPN on Res-Sil™ C Packing is the Latest of a Full Line of Bonded GC Phases

Restek now offers a wide range of bonded packings for packed GC columns, including Rtx®-1 and Stabilwax® phases, Carbowax® and n-octane phases on Res-Sil™ C packing, and the new OPN on Res-Sil™ C packing. Each of these packings have low bleed, conditioning times of less than 30 minutes, long lifetime, and consistent batch-to-batch reproducibility. Every batch of Restek's bonded phases is tested for bleed, efficiency, retention index and retention time reproducibility. In addition, Restek offers a full range of packed and micro-packed GC columns, available with specially-deactivated Silcosteel® tubing for improved inertness and efficiency.

1. N.C Saha, S.K. Jain, and R.K. Dua. *J. of Chromat. Sci.* 1978: 16, pp.323-328.

Reference not available from Restek.

### Product Listing:

Description	cat. #	price/g*
n-Octane on Res-Sil™ C, 80/100 Mesh	25030	\$30/g*
OPN on Res-Sil™ C, 80/100 Mesh	25042	\$30/g*
Res-Sil™ C, 80/100 Mesh	25028	\$24/g
2 % Carbowax 1540 on Res-Sil™ C, 80/100 Mesh	25044	\$30/g*

\*Minimum order 10g

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# Analysis and Stability of Low-Level Sulfur-Containing Samples Using Silcosteel®-Treated Products

by Gary Stidsen and Jingzhen Xu

Low-level air analyses of reduced sulfur compounds, such as hydrogen sulfide and methyl mercaptan, have been very difficult to perform due to adsorption of the compounds on metal surfaces. These difficulties are compounded by the short holding times of collected air samples. Restek has conducted research to improve sulfur analysis and has developed an analytical technique to allow for low concentration (ppbv) analysis of reduced sulfur compounds using a Silcosteel®-treated stainless steel system.

Restek's Silcosteel® passivation technique bonds an inert silica layer to the surface of stainless steel. The Silcosteel®-treated surface acts as a barrier to active compounds that can react or be adsorbed on stainless steel. This provides a very inert surface for the storage and transfer of reactive sulfur compounds. Most stainless steel products, including tubing, can be treated with the Silcosteel® process. Because the Silcosteel® layer is incorporated into the structure of the stainless steel, it is highly flexible. Therefore, Silcosteel®

tubing can be bent without affecting the inertness characteristics.

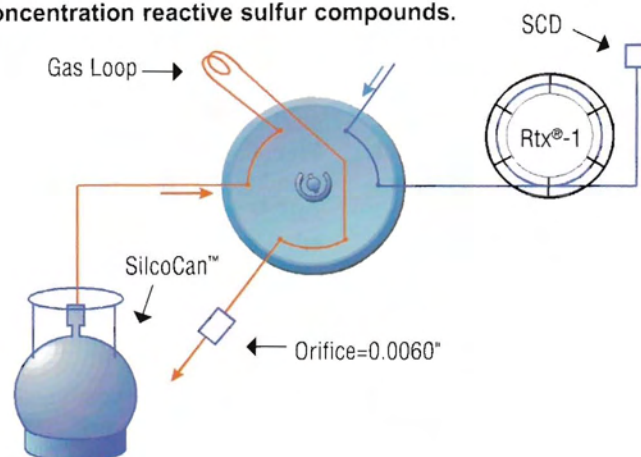
Holding time data of sulfur compounds using Silcosteel®-treated sample canisters with low ppmv concentrations were previously published in *The Restek Advantage*, Fall 1997. As an expansion of this project and to assist chemists in the field of sulfur analysis, we developed an analytical technique to demonstrate the effects of using Silcosteel®-treated transfer lines, sample loops, and sample storage canisters to work with low ppbv concentration of reactive sulfur compounds.

The research process was completed in three steps:

The **first step** was to select a mix of sulfur compounds for the stability tests (Table I). Some of these compounds will react with stainless steel surfaces and some will not. The reactive compounds include hydrogen sulfide, methyl mercaptan, and ethyl mercaptan. Carbonyl sulfide, dimethyl sulfide, and dimethyl disulfide are typically stable and do not

**Figure 1:**

**Silcosteel®-treated system ensures excellent analysis of low-concentration reactive sulfur compounds.**



react with stainless steel. Therefore, dimethyl sulfide was chosen as an internal standard.

The **second step** was to configure the analytical system so that the 60ppbv standard could be detected with enough of a signal-to-noise ratio so adsorption of compounds could be detected up to 80% loss. The analytical system was designed with a 1mL Silcosteel®-treated sample loop, Silcosteel®-treated Valco® valve, and 1/16" Silcosteel®-treated transfer lines (Figure 1). The analytical column was directly connected to the Valco® valve.

In order to consistently purge the 1mL sample loop with the sample, an orifice was attached to the exit of the sample loop allowing 60-90mL/min. of sample to flow through the gas loop during sample loading. The analytical column was an Rtx®-1 column (60m x 0.53mm ID, 7.0µm) and the detector was a Sievers Model 355 sulfur

chemiluminescence detector (SCD).

To introduce the sample to the GC column, the Silcosteel®-treated canister was pressurized to 26psig, and the sample loop was flushed with the sample for 90 seconds. After 90 seconds, the canister valve was closed and the pressurized sample loop was vented to atmospheric pressure. Once the sample loop was equilibrated to atmospheric pressure, the Valco® valve was switched to introduce the sample from the loop to the analytical column, then the GC analysis was initiated.

The SCD is very sensitive to sulfur-containing compounds, with a detection limit of about 5ppbv for a 1mL injection. The compounds first enter the burner, where they are combusted to SO\* and other products. The compounds then are transferred to a reaction cell, where the SO reacts with ozone resulting in SO<sub>2</sub> + O<sub>2</sub>. This chemiluminescence

**Table I—Sulfur Compounds**

Compound	Formula	Stock Conc. (ppmv)	Standard Conc. (ppbv)	Standard Conc. as S (ppbv)
Hydrogen sulfide	H <sub>2</sub> S	100	60	56
Carbonyl sulfide	COS	100	60	30
Methyl mercaptan	CH <sub>3</sub> SH	100	60	40
Ethyl mercaptan	CH <sub>3</sub> CH <sub>2</sub> SH	100	60	20
Dimethyl sulfide	CH <sub>3</sub> SCH <sub>3</sub>	100	60	30
Dimethyl disulfide	CH <sub>3</sub> SSCH <sub>3</sub>	100	60	40



12 reaction is detected at 300-400nm. Figure 2 shows a chromatogram of the 60ppb sulfur standard acquired using the gas loop setup. The standard exhibited very good signal-to-noise ratio without using sample focusing.

The *last step* was to test the stability of the sulfur compounds in the Silcosteel®-treated canisters over a three day period. The test compared the stability of the sulfur compounds in four Silcosteel®-treated canisters to a stainless steel canister. The standards were prepared by adding 10mL (gas) of 1000ppbv to a 6-liter canister, which then was pressurized to 26psig using nitrogen. The standards were made "dry," meaning no water was added to the canisters during preparation. A dry standard prevents any possible reaction with water and allows the effects of the sulfur compounds to appear directly on the Silcosteel®-treated surface. Dimethyl sulfide was used as the internal standard to ensure accuracy over the 74-hour testing period.

## Results

The results of the analysis indicated that the stability of sulfur compounds was very good over the 74-hour test period. The Silcosteel®-treated canisters showed excellent stability, with greater than 80% recovery of all sulfur compounds. The stainless steel canister showed significant decrease in recovery of hydrogen sulfide, methyl mercaptan, and ethyl mercaptan as time elapsed. Carbonyl sulfide and dimethyl disulfide showed no loss in the stainless steel canister. The graphs in Figure 3 show the results of the stability tests of hydrogen sulfide, methyl mercaptan, and ethyl mercaptan. Notice how quickly the drop in recovery of the sulfur compounds occurs in the stainless steel cylinder.

In conclusion, the combined usage of the Silcosteel®-treated transfer lines, gas loops, Valco® valve, and canisters provides excellent inert surfaces for analyzing reactive sulfur compounds down to 60ppbv.

Figure 2:

Excellent signal-to-noise ratio achieved without sample focusing.

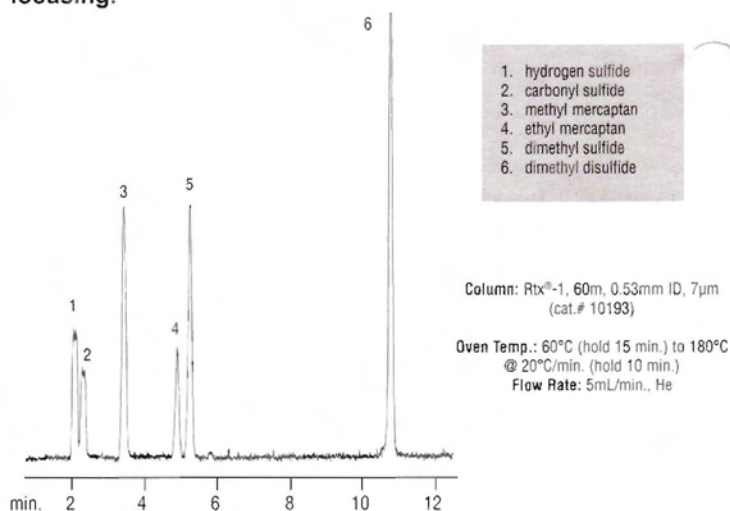
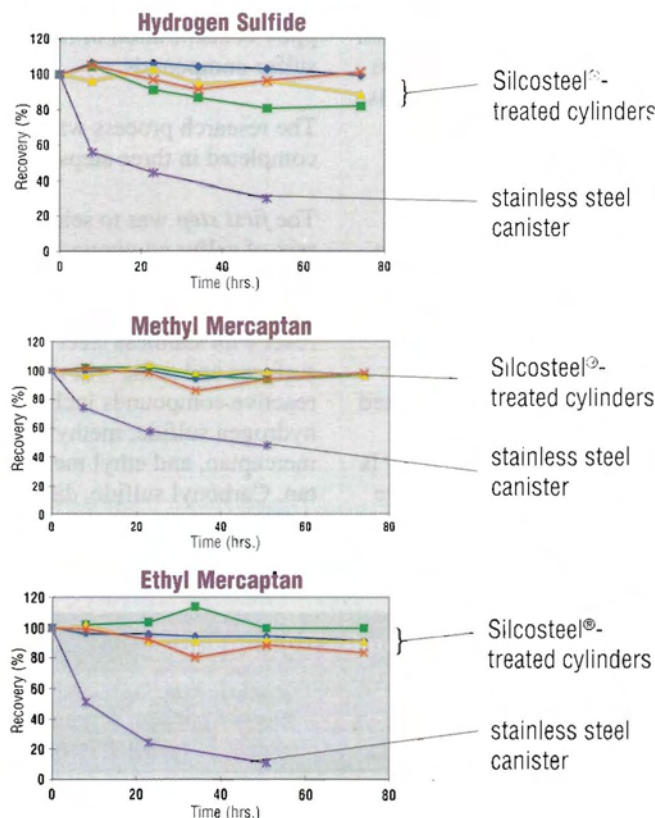


Figure 3:

Final results proved stability of sulfur compounds.



## Product Listing:

SilcoCan™ Canisters, with a Silcosteel®-Treated Valve*					
	cat.#	price			
6.0 Liter	24200-650	\$550			
Silcosteel®-Treated Sample Canisters					
75cc	24271	\$125			
150cc	24272	\$140			
300cc	24273	\$150			
500cc	24274	\$180			
1000cc	24275	\$320			
Silcosteel®-Treated Hoke Sample Cylinder Valves					
KEL-F® stem tip, 1/4" male NPT exit	24278	\$130			
KEL-F® stem tip, 1/4" compression exit	24279	\$130			
Silcosteel® Sample Loops for GC			Silcosteel®-Treated Transfer Lines		
size	cat.#	price	6-ft. Lengths	cat.#	price
250µL	22803	\$80	1/16" OD x 0.010" ID	20550	\$50
500µL	22804	\$82	1/16" OD x 0.020" ID	20524	\$50
1mL	22801	\$86	1/16" OD x 0.030" ID	20530	\$50
5mL	22802	\$86	1/16" OD x 0.040" ID	20538	\$50

\*Please call Restek's Customer Service at 800-356-1688 or 814-353-1300, ext. 3 for availability of SilcoCan™ canisters.

If you are wondering if Silcosteel® coating is right for your application, call us at 800-356-1688, ext. 4, or contact your local Restek Representative for a custom Silcosteel® information survey.



# New Flexible Graphite Ferrule Technology

by Doug Elliott and Brad Righnour

- **Conforms to challenging spaces with virtually no ferrule creep.**
- **Purer graphite eliminates detector interferences.**
- **Smoother surface and cleaner edges.**



Restek has been searching for a superior graphite ferrule, and we have found it! This new ferrule has been made from material developed by UCAR Carbon Co., who for over 100 years has dedicated research to developing graphite products that are in the forefront of fluid sealing technology. The finished product now offered by Restek has preferred performance characteristics including heat resistance, natural lubricity, chemical inertness, and high thermal conductivity.

The finest ferrule now available, Restek's New Flexible Graphite Ferrule starts with the most pure, natural graphite. The flexibility of the new ferrule is apparent by the ease with which the ferrule conforms to challenging spaces while maintaining its superior resilience. Pits, gouges, and even microscopic irregularities are filled under only moderate fitting tightening, to create leak-tight seals. In addition, this high-purity graphite exhibits virtually no ferrule creep relaxation for long-term fitting tightness.

These ferrules are manufactured by a process that eliminates the need for organic or inorganic binders, fillers, or other potentially off-gassing or absorptive ingredients. The resulting material is over 98% pure carbon, with the remainder being inert inorganic material. Sulfur and chloride-containing components are reduced to trace amounts in the ferrule composition to eliminate detector interference.

The unique flexibility properties are evident when the fitting is loosened—the ferrule will recover or spring back to an intermediate thickness with proportional reduction of density from the under-load conditions. This means that these ferrules may be resealed several times before they need to be replaced.

Even more amazing is that these new flexible graphite ferrules can be run continuously at up to 400°C gas chromatography (GC) tempera-

tures with no degradation of their sealing ability.

Restek offers both flexible graphite and VESPEL®/graphite ferrules to use with capillary columns. We have found that 60% VESPEL®/40% graphite give the best combination of sealing performance with ease of workability. They seal with minimal torque, are reusable, and are preferred for vacuum and high-pressure uses. Both ferrule types are stable to 400°C.

*For a complete listing of ferrules, please see the Restek 1999 Product Guide.*

## Capillary Ferrules

(for 1/16" compression-type fittings)

Ferrule ID	Fits Column ID	Graphite	VESPEL®/Graphite
<b>10-pack</b>			
0.3mm	0.20mm	20233 \$30	— —
0.4mm	0.25mm	20200 \$25	20211 \$30
0.5mm	0.32mm	20201 \$25	20212 \$30
0.6mm	0.28mm	— —	20232 \$40
0.8mm	0.53mm	2020 \$25	20213 \$30
<b>50-pack</b>			
0.4mm	0.25mm	20227 \$100	20229 \$120
0.5mm	0.32mm	20228 \$100	20231 \$120
0.8mm	0.53mm	20224 \$100	20230 \$120

## Compact Graphite Ferrules for HP GCs

(for capillary injection ports)

Ferrule ID	Fits Column ID	Graphite 10-pk.	Graphite 50-pk.
0.4/0.5mm	0.25–0.32mm	20250 \$25	20251 \$100
0.8mm	0.53mm	20252 \$25	20253 \$100

## Compact VESPEL®/Graphite Ferrules for HP GCs

Ferrule ID	Fits Column ID	VESPEL®/Graphite 10-pack	VESPEL®/Graphite 50-pack
0.4mm	0.25mm	20238 \$45	20239 \$180
0.5mm	0.32mm	20248 \$45	20249 \$180
0.8mm	0.53mm	20263 \$45	20264 \$180

## Hey HP Users!

Restek has produced a new 32-page catalog, *Genuine Restek Replacement Parts for HP GCs*, detailing our full line of products for HP GCs. It covers everything from the injector to the detector and includes tech tips and useful reference materials.

To request your copy, call our literature request hotline at 800-356-1688, ext. 5, or contact your local Restek representative.

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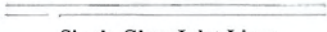




# Peak Performers

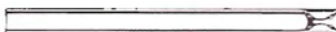


## New Inlet Liners from Restek

All of Restek's Liners  
are Deactivated


### for HP GCs with Gerstel injectors

Liners for HP CIS4 and PTV GCs	Benefits/Uses:	ID**/OD & Length (mm)	cat.#/price 10-pk.	Similar to HP part #
 Single Glass Inlet Liner	general use	2.0 ID 3.0 OD x 71	21157 \$71	5181-2036
 Baffled Glass Inlet Liner	active compounds, drugs, pesticides	1.5 ID 3.0 OD x 71	21704 \$149	5183-2037
 Glass Inlet Liner with Wool*	large volume injections	2.0 ID 3.0 OD x 71	21156 \$173	5183-2039


### for Varian GCs

Liners for Varian 1078/1079 GCs	Benefits/Uses:	ID**/OD & length (mm)	ea.	cat.#/price 5-pk.	25-pk.	Similar to Varian Part #
 1078/1079 Split—No Frit	active samples	3.4 ID 5.0 OD x 54	20859 \$32	20901 \$140	— —	03-918464-0 <sup>n</sup>
 Open 0.75mm ID	trace, low volume samples	0.75 ID 5.0 OD x 54	21714 \$22	21715 \$91	21716 \$370	03-925330-00
 1078/1079 Split/Splitless SilcoSleeve™	universal, unbreakable	3.8 ID 5.0 OD x 54	— —	20711 \$88	— —	—

### for Perkin-Elmer GCs

Splitless Liners for Perkin-Elmer GCs	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.#/price 5-pk.	Similar to PE Part #
 Auto SYS XL Split/Splitless	most common analyses	2.0 ID 4.0 OD x 81.2	21717 \$37	21718 \$148	N612-1004

### for Shimadzu GCs

Direct Injection Liners for Shimadzu GCs (0.32/0.53mm ID)	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.#/price 5-pk.	Similar to Shimadzu Part #
 94mm Uniliner® with Wool*	trace, dirty, high MW active samples, high recovery & linearity	3.0 ID 5.0 OD x 94	21713 \$45	21719 \$180	—

\*This sleeve is prepacked with fused silica wool. To order glass wool instead, add the suffix "-202" to the sleeve's catalog number.

\*\* Nominal ID at syringe needle expulsion point.



# 15 Direct Replacement Parts for HP GCs

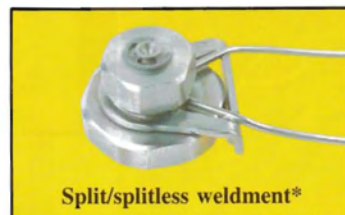
## Direct Replacement Split/Splitless Injection Port for HP 5890 GCs

Restek now offers a **direct replacement** split/splitless injector for the HP 5890 GC. The injector is manufactured from high-quality stainless steel and meets or exceeds HP original equipment specifications.

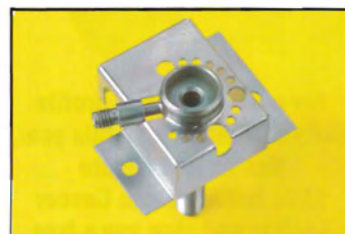
Would you like even better performance from your injector? Restek's proprietary Silcosteel®-coated version of the split/splitless injector is what you are looking for. Silcosteel® treatment passivates the metal surface to ensure an inert pathway for the sample, delivering increased performance.

\* Septum nut not included. Order separately.

Replacement Split/Splitless Injection Port for HP 5890 GCs			
Product	cat.#	price	
Replacement Split/Splitless Weldment Similar to HP part# 19251-60575	20265	\$200	
Replacement Shell Weldment Similar to HP part# 19251-80570	20266	\$178	
Septum Nut	21309	\$39	
Silcosteel®-Treated Split/Splitless Weldment	20267	\$272	
Silcosteel®-Treated Shell Weldment	20268	\$250	



**SILCOSTEEL®**  
version available



## Replacement Chemical Trap for HP 5880 GCs

(Similar to HP part #19362-60500.): cat.# 21158, \$82



## 1/4" CD Adaptor Fitting

(Similar to HP part #19802-80020.): cat.# 20269, \$99



## Gerstel GRAPHPACK® Conectors

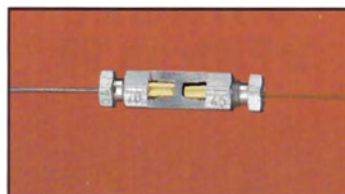
GRAPHPACK® technology results in a complete system that quickly and reliably makes truly leak-free, low-dead-volume connections. The central component is a metal-jacketed graphite ferrule—the ideal sealant for gas chromatography applications. These GRAPHPACK® ferrules eliminate all the disadvantages and shortcomings associated with connecting MXT® tubing to fused silica tubing.

GRAPHPACK® 3D/2 Connector\*: (0.25mm to 0.32 mm);  
cat.# 20272 (ea.), \$97

GRAPHPACK® 3D/2 Connector\*: (0.7mm to 0.45 mm);  
cat.# 20273 (ea.), \$97

\*Use only with GRAPHPACK® 3D/2 ferrules.

GRAPHPACK® 3D/2 Ferrules			
Ferrule ID	Fits Column ID	cat.# (10-pk.)	
0.4mm	0.25mm	20271	\$139
0.5mm	0.32mm	20270	\$139
0.8mm	0.53mm	20274	\$139



## Replacement 0.8mm ID Cross Disk Inlet Seal for HP 6890 GCs

(Similar to HP part #5182-9652.)

0.8mm ID Cross Disk Inlet Seal for HP GCs				
Inlet Seal Type	2-pk.		10-pk.	
Gold-Plated	20477	\$62	20476	\$280
Silcosteel®-Treated	20475	\$62	20474	\$280

**New**  
**CROSS DISKS**

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# 16 Behind the Scenes RESTEK

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High Resolution  
Chromatography  
Products



## Will you be in Orlando March 7-12? Check out Booth #6348 – The Hottest Booth at PittCon '99.

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Making a  
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Restek Corporation, innovators in chromatography products, will be offering several technical presentations at this year's Pittsburgh Conference. Visit our booth (#6348) for up-to-date information on these presentations, new products, what's "hot" in the industry, and employment opportunities. See you there—stop by for your free gift!

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Please direct comments & suggestions on this publication to my attention: Kristin Dick, Ext. 2313, or e-mail to [kristind@restekcorp.com](mailto:kristind@restekcorp.com)

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# the **RESTEK** Advantage

Innovators of High Resolution Chromatography Products

The US Clean Air Act of 1990 mandated the addition of oxygenates in 30% of America's gasoline supply to improve combustion of gasoline and decrease polluting emissions. Oxygen-containing compounds most commonly added to gasoline are methanol, ethanol, *tert*-butanol, methyl-*tert*-butylether (MTBE), diisopropylether (DIPE), ethyl-*tert*-butylether (ETBE), and *tert*-amyl-methylether (TAME). Of these compounds, MTBE is the primary additive. The combination of its relatively low manufacturing cost and non-corrosive nature made it a clear choice for the petroleum industry. Other oxygenates are added at much lower concentrations.

- ✓ *Oxygenates, including alcohols, resolved on one column*
- ✓ *Fewer coelutions than other columns, to prevent misidentifications*
- ✓ *Excellent capacity for ethanol and other polar compounds*

Now, however, we know that the ethers pose a threat to human health. These compounds have vapor pressures that range from 68mm Hg for TAME to 250mm Hg for the most volatile, MTBE. The high vapor pressure of MTBE threatens air quality at gasoline pumps. The oxygenates also are soluble in water, which contaminates ground water. In fact, an estimated 9,000 community drinking water wells now have detectable levels of MTBE contamination. Therefore, the US Environmental Protection Agency (EPA) has moved to ban MTBE use in gasoline. Other ethers can be used as additives, but they are more expensive and pose similar health risks.

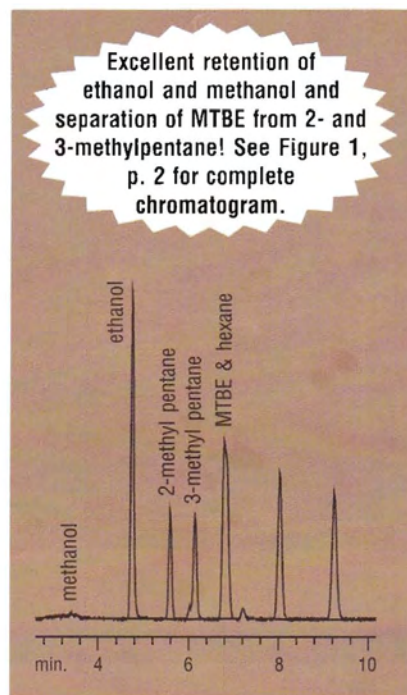
Corn-based ethanol is the proposed gasoline additive. Ethanol has a lower toxicity, lower volatility, and is not environmentally persistent. A variety of methods has been used for the capillary gas chromatographic (GC) analysis of oxygenates in gasoline. Success of these methods is based on the established ability of the GC capillary columns to resolve oxygenates from early-eluting alkanes such as 2-methylpentane and 3-methylpentane. Because of the possibility of widespread corn-based ethanol use, the environmental chemist now must find a GC column appropriate for the analysis of oxygenates including the alcohols.

The new Rtx®-VGC column retains the alcohols to allow quantitation of ethanol without interference from methanol. Methanol is commonly used in preparing VOA standards and may be added to gasoline as well. The Rtx®-VGC column is more polar than other capillary columns commonly used for gasoline range organic (GRO) analysis. This column is designed to exhibit greater retention and

## Rtx®-VGC Column

### *Improved GC separation of MTBE*

by Christopher English, Applications Chemist



elution order is different compared to other more non-polar stationary phases. MTBE elutes after 2- and 3-methylpentane. These are the most commonly misidentified compounds when using PID/FID for GRO analysis.

Mass spectrometry (MS) can be used to increase the level of confidence in your analysis. Column selection for MS must ensure that compounds sharing ions will not coelute. Gasoline samples may

in this issue

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**Fall 2000**  
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## Rtx®-VGC Column, cont.

Ethanol analysis using purge-and-trap concentration and MS detection has proved successful with Rtx®-VGC columns (Figure 1). Benzene, toluene, ethylbenzene, xylene (BTEX);

selectivity and fewer coelutions provided by the Rtx®-VGC column decreases high bias.

Ethanol analysis using purge-and-trap

surrogates; retention time markers; and other branched aromatics were added, along with the oxygenates. Because ethanol does not purge well, the concentration of ethanol in the calibration mix was added at 100 times that of the ethers. The sample was analyzed using the Rtx®-VGC column and was detected using an MS system. Environmental laboratories have reported linear calibrations of ethanol and detection limits of 2.5ppm in 5mL of water using this method.

Whether using PID, FID or MS, the Rtx®-VGC column will ensure excellent selectivity and improve your laboratory's data quality for MTBE, ethanol, and other oxygenate analytes.

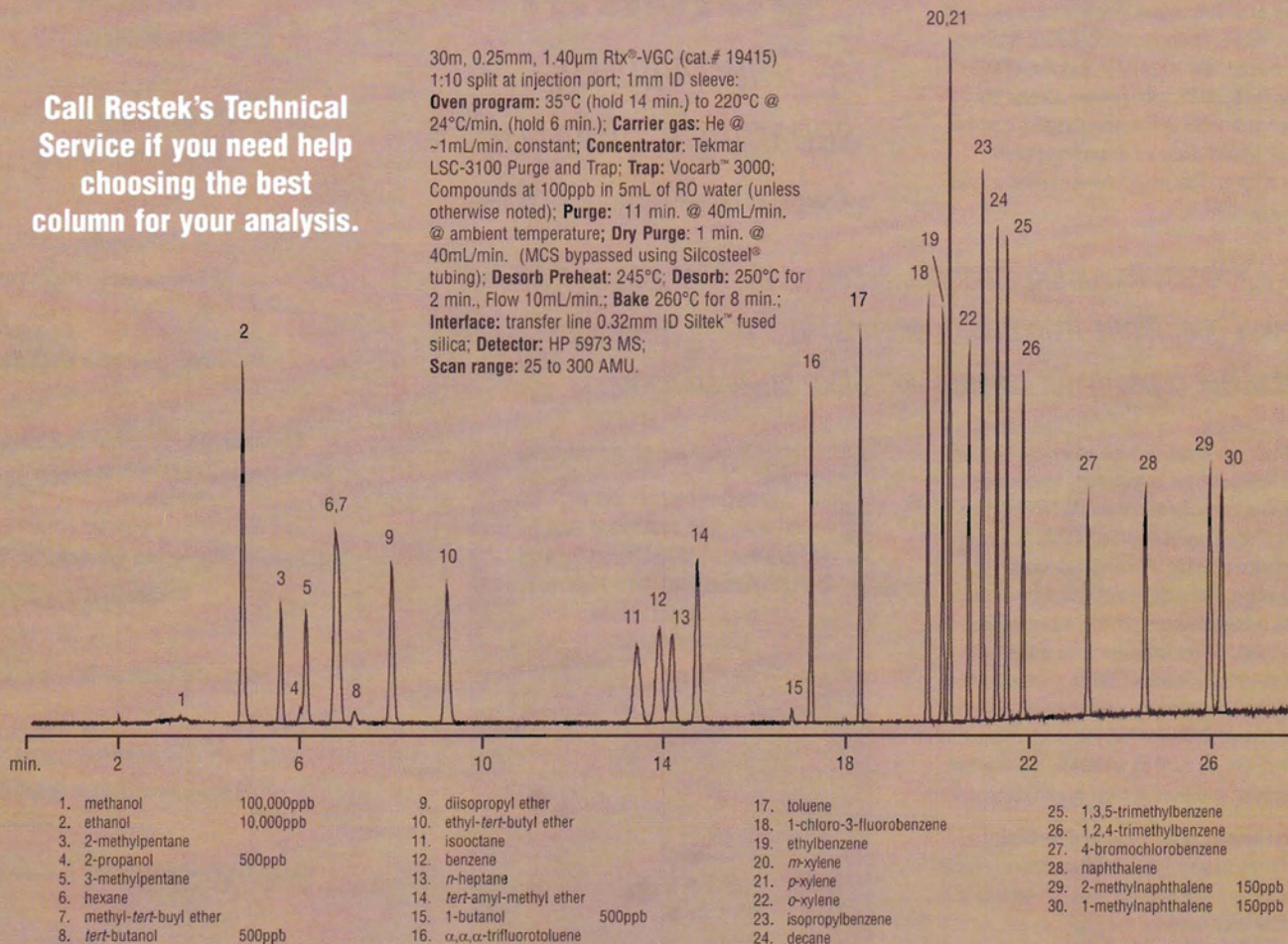
The Rtx®-VGC column has a programmable temperature limit of 260°C and exhibits exceptionally low bleed at common operating temperatures of 220°C.

**Figure 1**

The Rtx®-VGC column resolves gasoline components and commonly used oxygenate additives.

Call Restek's Technical Service if you need help choosing the best column for your analysis.

30m, 0.25mm, 1.40µm Rtx®-VGC (cat.# 19415)  
1:10 split at injection port; 1mm ID sleeve;  
**Oven program:** 35°C (hold 14 min.) to 220°C @ 24°C/min. (hold 6 min.); **Carrier gas:** He @ ~1mL/min. constant; **Concentrator:** Tekmar LSC-3100 Purge and Trap; **Trap:** Vocab™ 3000; Compounds at 100ppb in 5mL of RO water (unless otherwise noted); **Purge:** 11 min. @ 40mL/min. @ ambient temperature; **Dry Purge:** 1 min. @ 40mL/min. (MCS bypassed using Silcosteel® tubing); **Desorb Preheat:** 245°C; **Desorb:** 250°C for 2 min.; Flow 10mL/min.; **Bake** 260°C for 8 min.; **Interface:** transfer line 0.32mm ID Siltek® fused silica; **Detector:** HP 5973 MS; **Scan range:** 25 to 300 AMU.



## Rtx®-VGC (Fused Silica)

ID	df (µm)	Temp. Limits	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	-40 to 240/260°C	19415	19416	—	—
0.32mm	1.80	-40 to 240/260°C	19419	19420	—	—
0.45mm	2.55	-40 to 240/260°C	19408	—	19409	—
0.53mm	3.00	-40 to 240/260°C	19485	19488	19474	19489
ID	df (µm)	Temp. Limits	20-Meter	40-Meter		
0.18mm	1.00	-40 to 240/260°C	49414	49415	—	—

\*American Society for Testing and Materials (ASTM) Method D2887/D4814, and EPA Methods 8015, 8260, and 8020.



Byproducts from incomplete incineration and from impurities in some reaction mixtures—polychlorinated dibenzo dioxins (PCDDs) and polychlorinated dibenzo furans (PCDFs)—are among the most toxic compounds commonly analyzed in the environmental testing field. Exposure to these compounds is connected to an array of health effects.

The dioxin molecule is comprised of two benzene rings connected by two oxygen links; the furans have similar toxicity and structure but only one oxygen link (Figure 1). Depending on the extent and position of chlorine substitution, the congeners have very different toxicity. Substitution at the outermost positions (2, 3, 7, and 8) lead to the most toxic congeners, which are the most important to resolve. US Environmental Protection Agency (EPA) Methods 8290 and 1613 require analysis of all dioxin and furan congeners that have chlorine substitution at the 2, 3, 7 or 8 positions. This requirement, coupled with the similar boiling points of these compounds and the desired low limits of detection, make these separations challenging.

Chemists performing the analysis of dioxins and furans by gas chromatography (GC) with high-resolution mass spectrometry (GC/HRMS) have a difficult time determining the best column for the separation of these target compounds. Many columns are on the market for this analysis, but choosing the right one is critical.

Because a mass spectrometer (MS) is used for detection, many analysts want the lowest bleed columns possible. Some laboratories have begun performing this analysis on silarylene columns (i.e., Rtx®-5Sil MS, DB-5MS®) due to their low GC/MS bleed. It is important to note that these columns do not typically perform the separation required by the analytical methods. For example, silarylene columns can yield a coelution between 2,3,7,8-TCDD and 1,2,3,9-TCDD. Also, silarylene columns are not



## Questions?

Restek's Technical Service Department will go to great lengths to find the answers to your toughest analytical problems. Call us at 800-356-1688 or 814-353-1300, ext. 4. Our regular technical service hours are 8AM to 7PM EST, Monday through Thursday, and 8AM to 5PM EST on Fridays. You also can fax us at 814-353-1309 or email us at support@restekcorp.com.

## Rtx®-5MS

### Choosing the right GC column for dioxin and furan analysis

by Frank Dorman, Ph.D., R&D Chemist

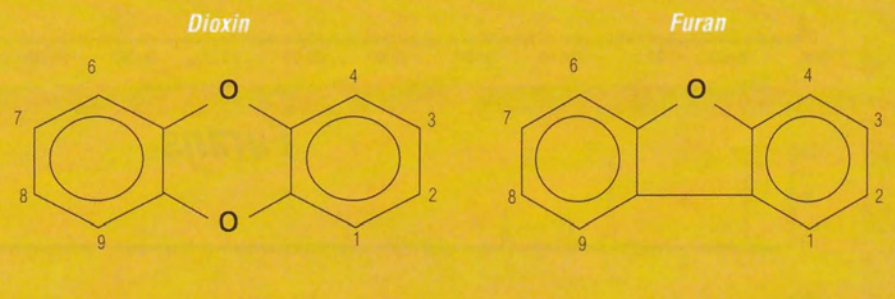
- ✓ Best 2,3,7,8-TCDD separation
- ✓ Columns individually tested to guarantee low GC/MS bleed

equivalent to standard phenyl/methyl columns—elution orders and retention times will be different. This is especially important because the window performance mixtures were designed for use with a 5% diphenyl/95% dimethyl polysiloxane stationary phase.

Restek's 5% diphenyl/95% dimethyl polysiloxane (Rtx®-5MS) columns are better suited to meet the performance standards of this analysis. Notice how the Rtx®-5MS column separates all of the important dioxin and furan congeners (Figure 2, page 4). The key column performance parameter is the separation of 2,3,7,8-TCDD. It must be separated from its nearest eluting neighbor—1,2,3,7-TCDD/1,2,3,8-TCDD—by a 25% valley or better. These columns are individually tested to provide low bleed levels for MS detection. If you would like more information on this application, or choices of confirmation columns, please contact Restek's technical service at 800-356-1688 or 814-353-1300, ext. 4.

Figure 1

The amount and position of chlorine substitution determines the toxicity of the congeners.



## Rtx®-5MS (Fused Silica)

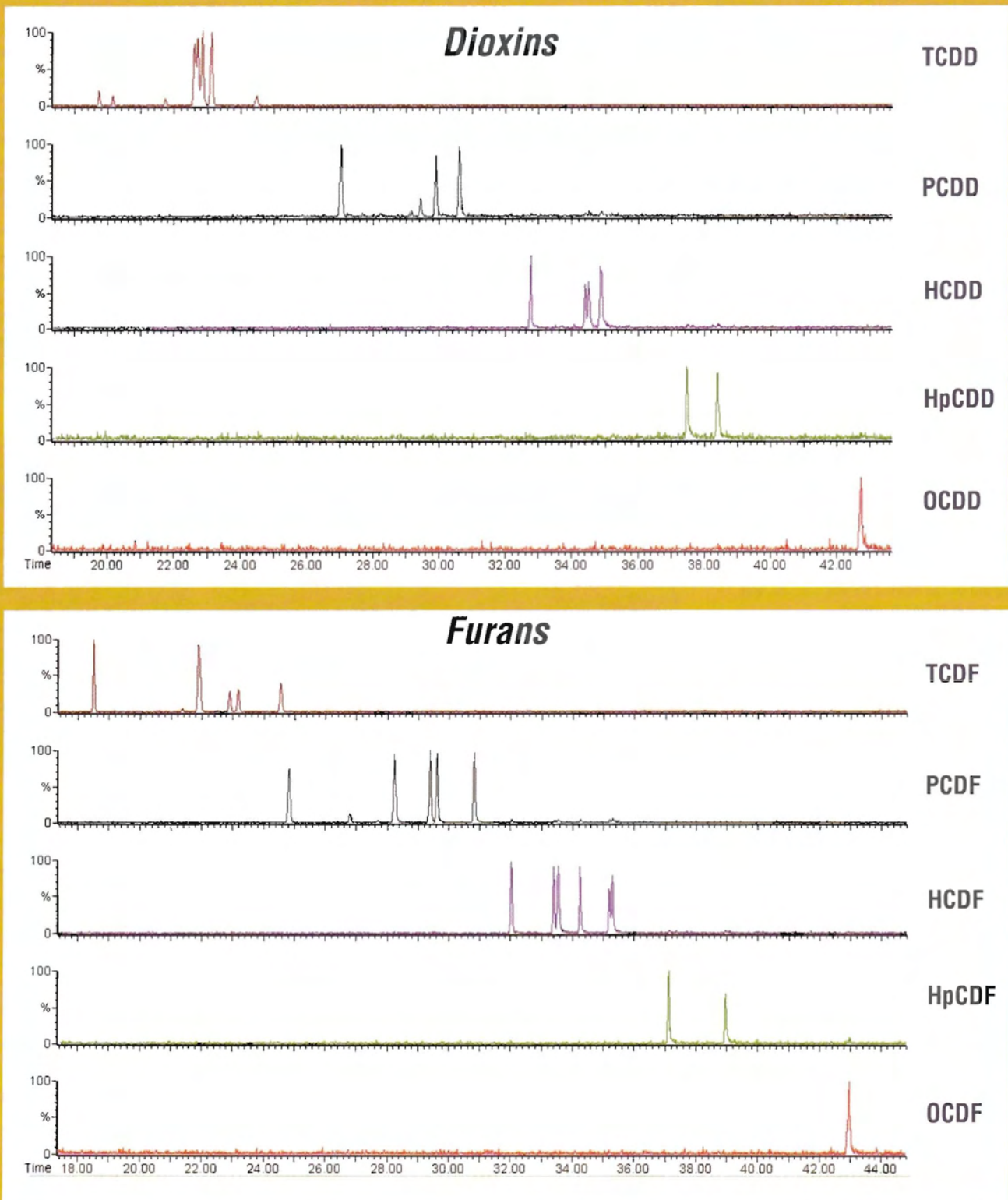
Crossbond® 5% diphenyl/95% dimethyl polysiloxane

ID	df (µm)	Temp. Limits	15-Meter	30-Meter	60-Meter
0.25mm	0.10	-60 to 330/350°C	12605	12608	12611
	0.25	-60 to 330/350°C	12620	12623	12626
	0.50	-60 to 330/350°C	12635	12638	12641
	1.00	-60 to 325/350°C	12650	12653	—
0.32mm	0.10	-60 to 330/350°C	12606	12609	12612
	0.25	-60 to 330/350°C	12621	12624	12627
	0.50	-60 to 330/350°C	12636	12639	12642
	1.00	-60 to 325/350°C	12651	12654	—
0.53mm	0.50	-60 to 320/340°C	12637	12640	—
	1.00	-60 to 320/340°C	12652	12655	—
	1.50	-60 to 310/330°C	12667	12670	—



Figure 2

Rtx®-5MS phenyl/methyl column provides the best separation of dioxin and furan congeners.



60m, 0.25mm ID, 0.25µm Rtx®-5MS (cat.# 12626)

43psi head pressure/constant pressure. Temp.: 100°C (hold 1 min.) to 200°C @ 40°C/min. to 235°C @ 3°C/min. (hold 10 min.) to 300°C @ 6°C/min. (hold 5-10 min.).

Chromatograms courtesy of Karen McPherson, Ontario Ministry of the Environment



For years, a nonbonded Carbowax® phase of nominal 20,000 molecular weight (mean actual molecular weight is 17,000) has been the standard stationary phase for capillary gas chromatography (GC) columns used in the flavor and fragrance industry. Because retention indices of thousands of compounds have been recorded on the Carbowax® phase, analysts are reluctant to use other polyethylene glycol (PEG) columns, which show marked differences in selectivity and compound retention indices. Restek has designed the new Rt-CW20M™ F&F nonbonded column and tests it with a special mix to ensure similar selectivity corresponding to the nonbonded Carbowax® phase for many flavor and fragrance applications.

To ensure that the selectivity of the Rt-CW20M™ F&F polymer matches that of other nonbonded Carbowax® 20M capillary columns, Restek designed a new polar test mix that contains compounds commonly analyzed in flavor or fragrance samples. This new test mix verifies the performance of each Rt-CW20M™ F&F polymer batch, to ensure consistent performance. The selectivity of the Restek phase is evaluated from the retention indices of selected alcohols, aldehydes, ketones, and esters, using methyl esters as marker compounds.

Calculations of Kováts indices for two bonded phase and two nonbonded phase PEG columns reveal similarities and differences in selectivity (Table I). Comparison of the indices confirms the excellent match in selectivity (no difference exceeded 0.02 units) between the two nonbonded phases—the Rt-CW20M™ F&F column and the traditional 20M columns. The two bonded phases—the Rtx®-Wax and Stabilwax® columns—differ by as much as 0.08

# Rt-CW20M™ F&F GC Column

*20M selectivity with  
improved inertness for  
flavor and fragrance  
analysis*

by Sherry Sponsler-Navaroli,  
FFF Applications Manager

units between each other and by as much as 0.13 units from the nonbonded columns

A comparison of an Rt-CW20M™ F&F column and a traditional Carbowax® 20M column, using the new test mix, demonstrates an excellent match in selectivity between the two columns (Figure 1). However, notice the poor response for 2,3-butanediol on the traditional column, suggesting the Rt-CW20M™ F&F column may be more inert.\*

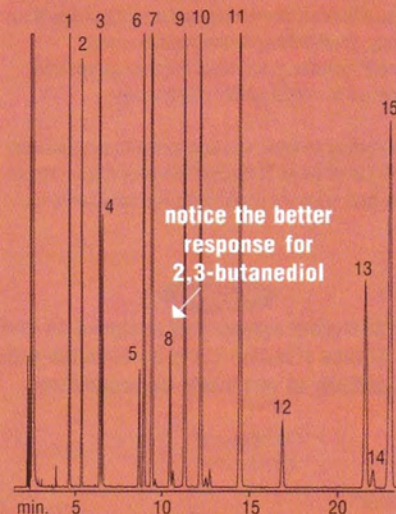
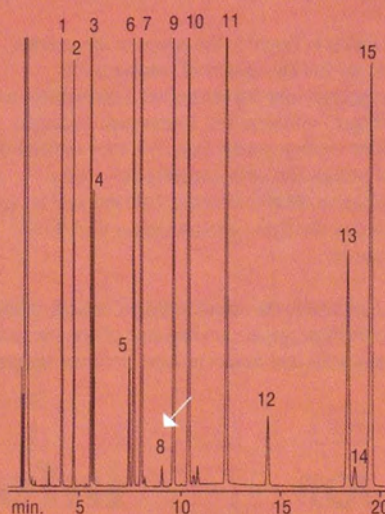
For many flavor and fragrance analyses, the new Rt-CW20M™ F&F column features selectivity equivalent to the traditional nonbonded Carbowax® 20M column. Our new test mixture ensures proper selectivity and product consistency

**Figure 1**

The Rt-CW20M™ F&F column has equivalent selectivity but better inertness, compared to a traditional nonbonded Carbowax® column.

traditional Carbowax® column

Rt-CW20M™ F&F column



notice the better  
response for  
2,3-butanediol

50m, 0.32mm ID, 0.33µm, Rt-CW20M™ and traditional 20M. On-column injection of 5ng to 150ng each compound in methylene chloride, split 10:1. Carrier gas: hydrogen, 40cm/sec.; Inj./det. temperatures: 220°C; Oven temp.: 110°C.

- |                      |                        |
|----------------------|------------------------|
| 1. methyl heptanoate | 9. linalool            |
| 2. hexanol           | 10. linalyl acetate    |
| 3. methyl octanoate  | 11. methyl decanoate   |
| 4. nonanal           | 12. menthol            |
| 5. menthone          | 13. α-terpineol        |
| 6. citronellal       | 14. γ-terpineol        |
| 7. methyl nonanoate  | 15. methyl undecanoate |
| 8. 2,3-butanediol    |                        |

**Table I**

Kováts indices for PEG columns show equivalent selectivity for the nonbonded Rt-CW20M™ F&F and HP-20M columns.

	Rt-CW20M™	HP-20M	Rtx®-Wax	Stabilwax®
methyl heptanoate (ME7)	7.00	7.00	7.00	7.00
hexanol	7.46	7.49	7.50	7.54
methyl octanoate (ME8)	8.00	8.00	8.00	8.00
nonanal	8.06	8.06	8.07	8.09
menthone	8.81	8.80	8.82	8.86
citronellal	8.88	8.88	8.90	8.92
methyl nonanoate (ME9)	9.00	9.00	9.00	9.00
2,3-butanediol	9.26	9.30	9.37	9.45
linalool	9.44	9.44	9.47	9.50
linalyl acetate	9.62	9.62	9.63	9.63
methyl decanoate (ME10)	10.00	10.00	10.00	10.00
menthol	10.34	10.34	10.37	10.42
α-terpineol	10.87	10.87	10.91	10.98
γ-terpineol	10.91	10.91	10.95	10.98
methyl undecanoate (ME11)	11.00	11.00	11.00	11.00

## Rt-CW20M™ F&F

(nonbonded fused Silica) Stable to 220°C

dimensions	cat.#
30m, 0.25mm ID, 0.25µm	12523
50m, 0.32mm ID, 0.33µm	12539

\* Both columns were connected to one inlet using a short piece (<0.25m) of 0.53mm ID Siltek™ guard column (cat.# 10028) and a Y connector (Siltek™ Y Press-Tight® connector cat.# 20469). On-column concentration of 2,3-butanediol is 17.5ng for each column



# Sulfinert™ Sample Cylinders

## Sampling of sulfur compounds at low ppb levels

by Dave Shelow, Environmental Innovations Chemist

- ✓ Inert, for less than 1ppm concentration\*
- ✓ Stability proven for 54 hours of storage

Stainless steel sample cylinders commonly are used in the collection and analysis of refinery and natural gas samples. These samples often contain trace amounts of sulfur-containing compounds (e.g., hydrogen sulfide, mercaptans, and sulfides), which may interfere with reactions or damage catalysts in many petrochemical processes. Because sulfur compounds are quickly adsorbed by the stainless steel surfaces, accurate determination is impossible when using untreated sample cylinders.

Restek's Sulfinert™ passivation technique bonds an inert layer to the surface of stainless steel. This layer acts as a barrier, preventing active compounds from reacting or adsorbing to the stainless steel. Sulfinert™ products are ideal for the storage and transfer of low-level sulfur compounds.

An analytical method was developed to demonstrate the effects of using Sulfinert™ transfer lines, sample loops, and sample cylinders for the analysis of low-

level reactive sulfur compounds (Figure 1). To characterize the Sulfinert™ surface, the stability of sulfur compounds in three Sulfinert™-treated cylinders was tested over a 54-hour period.

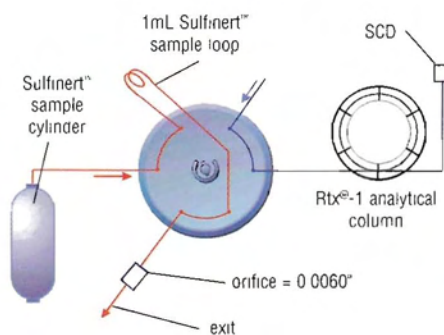
The standards were made by adding 1mL of 100ppm standard into a 500cc sample cylinder and pressurizing to 160psig. No water was added to the standards in order to simulate the petrochemical process. Dimethyl sulfide, which has been shown to be non-reactive in the standard and is not adsorbed by stainless steel, was used as an internal standard for the study.

As shown in Figure 2, the results of the analysis indicate that the stability of reactive sulfur compounds over the test period is remarkable using Sulfinert™ cylinders and accessories. Hydrogen sulfide exhibits greater than 85% recovery over the test period. The other compounds—methyl mercaptan, ethyl mercaptan, carbonyl sulfide, and dimethyl disulfide—exhibit greater than 90% recovery.

In conclusion, the use of Sulfinert™ sample cylinders greatly increases the holding time of reactive sulfur compounds and results in more accurate analyses.

Figure 1

The analytical system was designed so that the 17ppbv standard could be detected with sensitivity to quantitate compound loss.



Sulfinert™ Sample Cylinders	
Size	cat.#
75cc	24130
150cc	24131
300cc	24132
500cc	24133
1000cc	24134

\*Sticsteel® products can be used for concentrations > 1ppm

## for more info

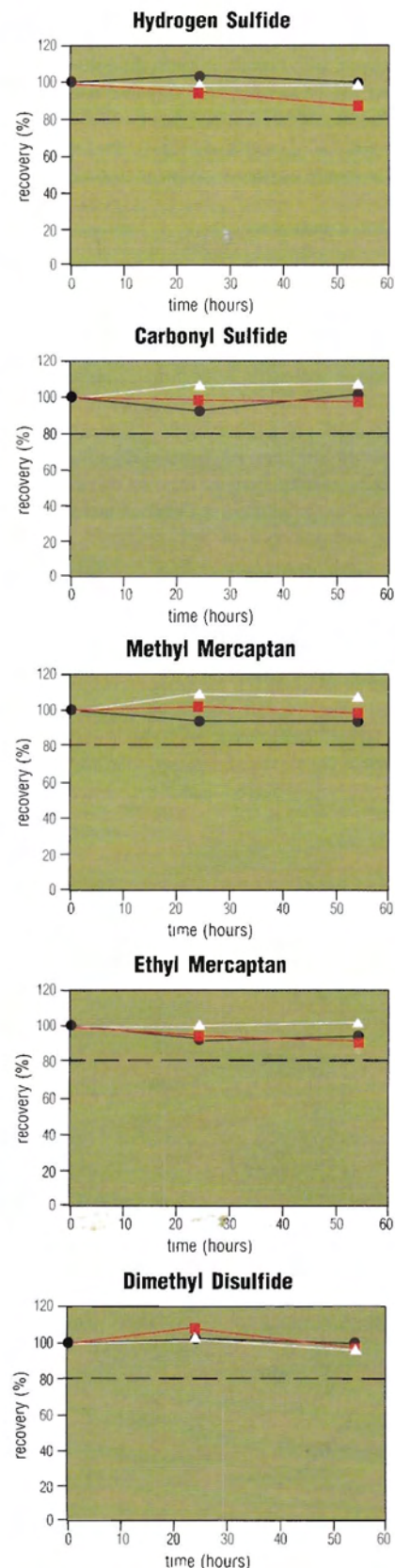
For more information on Sulfinert™, request the flyer lit. cat.# 59203 or visit [www.restekcorp.com/sulfinert.htm](http://www.restekcorp.com/sulfinert.htm).

Sulfinert™ Sample Loops	
Size	cat.#
5µL	22840
10µL	22841
20µL	22842
25µL	22843
50µL	22844
100µL	22845
250µL	22846
500µL	22847
1cc	22848
2cc	22849
5cc	22850

Figure 2

Stability of sulfur compounds is remarkable in Sulfinert™ cylinders.

▲ Sulfinert™ cylinder 1    ● Sulfinert™ cylinder 2    ■ Sulfinert™ cylinder 3





Update IVA of the third edition of SW-846—Test Methods for Evaluating Solid Waste, Physical/Chemical Methods—includes US Environmental Protection Agency (EPA) Method 8270D, the analysis of semivolatile organic pollutants in solid waste, soil, water, and air matrices, using gas chromatography/mass spectrometry (GC/MS). There are no major revisions from EPA Method 8270C.

Restek carefully reviewed EPA Methods 8270C and 8270D and prepared analytical reference materials to include all of the most commonly calibrated compounds. The compounds have been divided to provide flexibility, convenience, and maximum stability. Restek also offers all the required surrogate, internal standard, calibration check, matrix spike, and tuning mixtures currently required for this method. We also can make custom mixtures to meet client-specific compound lists!

#### 8270 Calibration Mix #1

benzoic acid	3-methylphenol ( <i>m</i> -cresol)
4-chloro-3-methylphenol	4-methylphenol ( <i>p</i> -cresol)
2-chlorophenol	2-nitrophenol
2,4-dichlorophenol	4-nitrophenol
2,6-dichlorophenol	pentachlorophenol
2,4-dimethylphenol	phenol
4,6-dinitro-2-methylphenol	2,3,4,6-tetrachlorophenol
2,4-dinitrophenol	2,4,5-trichlorophenol
dinoseb	2,4,6-trichlorophenol
2-methylphenol ( <i>o</i> -cresol)	

2,000µg/mL ea. in methylene chloride. 1mL/ampul

Each	5-pk.	10-pk.
31618	31618-510	
with data pack		
31618-500	31618-520	31718

#### 8270 Calibration Mix #2

aniline	3-nitroaniline
benzidine	4-nitroaniline
4-chloroaniline	N-nitrosodimethylamine
3,3'-dichlorobenzidine	N-nitrosodi- <i>n</i> -propylamine
diphenylamine	pyridine
2-nitroaniline	

2,000µg/mL ea. in methylene chloride. 1mL/ampul

Each	5-pk.	10-pk.
31619	31619-510	
with data pack		
31619-500	31619-520	31719

**for more info**

Request the Fast Facts lit. cat.# 59326 for a complete product listing including more calibration mixtures; calibration check compound, internal standards, surrogate and matrix spiking mixes; and calibration kits.

# Semivolatile Organic Reference Materials

## For US EPA Methods 8270C & 8270D

by Christopher Cox, Senior R&D Chemist

- ✓ Flexibility in calibration
- ✓ Convenience and maximum stability

#### 8270 Calibration Mix #3

aramite	hexachlorobutadiene
bis (2-chloroethyl) ether	hexachlorocyclopentadiene
bis (2-chloroethoxy) methane	hexachloroethane
4-bromophenyl phenyl ether	hexachloropropene
chlorobenzilate	isodrin
2-chloronaphthalene	kepone
4-chlorophenyl phenyl ether	pentachlorobenzene
1,2-dichlorobenzene	pentachloronitrobenzene
1,3-dichlorobenzene	1,2,4,5-tetrachlorobenzene
1,4-dichlorobenzene	1,2,4-trichlorobenzene
1,3-dinitrobenzene	
hexachlorobenzene	

2,000µg/mL ea. in methylene chloride. 1mL/ampul

Each	5-pk.	10-pk.
31620	31620-510	
with data pack		
31620-500	31620-520	31720

#### 8270 Calibration Mix #4

acetophenone	2,6-dinitrotoluene
azobenzene	ethyl methanesulfonate
benzyl alcohol	isophorone
bis (2-ethylhexyl) phthalate	isosafole ( <i>cis</i> & <i>trans</i> )
butyl benzyl phthalate	methyl methanesulfonate
dibenzofuran	1,4-naphthoquinone
diethyl phthalate	nitrobenzene
dimethyl phthalate	4-nitroquinoline-1-oxide
di- <i>n</i> -butyl phthalate	phenacetin
di- <i>n</i> -octyl phthalate	safrole
2,4-dinitrotoluene	

2,000µg/mL ea. in methylene chloride. 1mL/ampul

Each	5-pk.	10-pk.
31621	31621-510	
with data pack		
31621-500	31621-520	31721

#### 8270 Calibration Mix #5

acenaphthene	dibenz(a,h)anthracene
acenaphthylene	fluoranthene
anthracene	fluorene
benzo(a)anthracene	indeno(1,2,3-cd)pyrene
benzo(a)pyrene	3-methylcholanthrene
benzo(b)fluoranthene	1-methylnaphthalene
benzo(g,h,i)perylene	2-methylnaphthalene
benzo(k)fluoranthene	naphthalene
chrysene	phenanthrene
	pyrene

2,000µg/mL ea. in methylene chloride. 1mL/ampul

Each	5-pk.	10-pk.
31622	31622-510	
with data pack		
31622-500	31622-520	31722

#### 8270 Calibration Mix #6

diallate ( <i>cis</i> or <i>trans</i> )	parathion
dimethoate	phorate
disulfoton	pronamide
famphur	thionazine
methyl parathion	O,O,O-trimethyl phosphorothioate

2,000µg/mL ea. in methylene chloride. 1mL/ampul

Each	5-pk.	10-pk.
31623	31623-510	
with data pack		
31623-500	31623-520	31723

#### Organochlorine Pesticide Mix AB #1

aldrin	endosulfan II
α-BHC	endosulfan sulfate
α-chlordane	endrin
β-BHC	endrin aldehyde
4,4'-DDD	endrin ketone
4,4'-DDE	γ-BHC (lindane)
4,4'-DDT	γ-chlordane
δ-BHC	heptachlor
dieldrin	heptachlor epoxide
endosulfan I	methoxychlor

200µg/mL ea. in hexane/toluene (1:1). 1mL/ampul

Each	5-pk.	10-pk.
32291	32291-510	
with data pack		
32291-500	32291-520	32391

#### Appendix IX Mix #1

2-acetylaminofluorene	N-nitrosodiethylamine
4-aminobiphenyl	N-nitrosomethylethylamine
<i>p</i> -dimethylaminoazobenzene	N-nitrosomorpholine
3,3'-dimethylbenzidine	N-nitrosopiperidine
α,α'-dimethylphenethylamine (free base)	
methapyrilene (free base)	N-nitrosopyrrolidine
1-naphthylamine	1,4-phenylenediamine
2-naphthylamine	2-picoline
5-nitro- <i>o</i> -toluidine	<i>o</i> -toluidine
N-nitrosodibutylamine	

2,000µg/mL ea. in methylene chloride. 1mL/ampul

Each	5-pk.	10-pk.
31625	31625-510	
with data pack		
31625-500	31625-520	31725



Volatile organic compounds (VOCs) are some of the most prevalent contaminants found in water supplies. In fact, they are present in one-fifth of the US water supplies. VOCs enter ground water from a variety of sources—from leaking underground fuel tanks to industrial solvent used in septic system cleaners. They may have a variety of harmful health effects including central nervous system depression.

US Environmental Protection Agency (EPA) Method 524.2 was created to monitor the most common contaminants found in the drinking water supply, using gas chromatography/mass spectrometry (GC/MS). The latest revision to this method has introduced 24 additional compounds to the existing list of 60 analytes. Further updates allow alternate sorbents to trap VOCs, provided all quality assurance criteria are met. However, purge time and purge gas flow rates have remained the same as in previous revisions, and cannot be changed.

Restek has introduced the Rtx®-VMS column for the analysis of these contaminants. This column features fast analyses and excellent resolution (Figure 1). The 84 compounds are analyzed on a 0.25mm ID

# Rtx®-VMS 0.25mm ID Column

## Analyzes 84 volatile organic compounds in 11 minutes

By Christopher English, Applications Chemist

- ✓ Excellent resolution of difficult components
- ✓ Tuned selectivity for VOCs in drinking water

column in eleven minutes. The starting temperature is set to 45°C, which is high enough to allow a quick overall oven cycle time without sacrificing gas compound resolution. The total cycle time depends on several factors: the type of GC, the oven start temperature, the final oven temperature, and the temperature of the room in which the analysis

occurs. The limiting factor becomes the purge and trap cycle time, which can vary greatly depending on the dry purge time, bake time, and trap cool-down temperature. The total oven cycle time for this analysis was less than 17 minutes. We used a Vocab™ 3000 trap with a 1 minute dry purge. Changes in dry purge time using this trap did not

Figure 1

Rtx®-VMS column provides fast and excellent resolution of VOCs in drinking water.

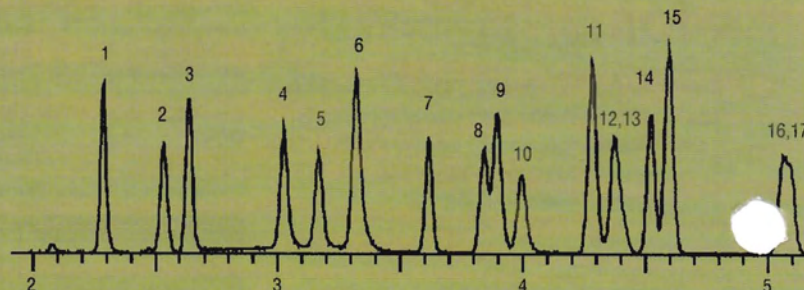
- |                                      |   |
|--------------------------------------|---|
| 1. dichlorofluoromethane             | 44. tetrachloroethene                   |
| 2. chloromethane                     | 45. <i>trans</i> -1,3-dichloropropene   |
| 3. vinyl chloride                    | 46. ethyl methacrylate                  |
| 4. bromomethane                      | 47. 1,1,2-trichloroethane               |
| 5. chloroethane                      | 48. dibromochloromethane                |
| 6. trichlorofluoromethane            | 49. 1,3-dichloropropane                 |
| 7. diethyl ether                     | 50. 1,2-dibromoethane                   |
| 8. 1,1-dichloroethene                | 51. 2-hexanone                          |
| 9. carbon disulfide                  | 52. ethylbenzene                        |
| 10. iodomethane                      | 53. chlorobenzene                       |
| 11. allyl chloride                   | 54. 1,1,1,2-tetrachloroethane           |
| 12. methylene chloride               | 55. <i>m</i> -xylene                    |
| 13. acetone                          | 56. <i>p</i> -xylene                    |
| 14. <i>trans</i> -1,2-dichloroethene | 57. <i>o</i> -xylene                    |
| 15. methyl- <i>tert</i> -butyl-ether | 58. styrene                             |
| 16. 1,1-dichloroethane               | 59. bromoform                           |
| 17. acrylonitrile                    | 60. isopropylbenzene                    |
| 18. <i>cis</i> -1,2-dichloroethene   | 61. 4-bromofluorobenzene                |
| 19. 2,2-dichloropropane              | 62. <i>n</i> -propylbenzene             |
| 20. bromochloromethane               | 63. bromobenzene                        |
| 21. chloroform                       | 64. 1,1,2,2-tetrachloroethane           |
| 22. methyl acrylate                  | 65. 1,3,5-trimethylbenzene              |
| 23. carbon tetrachloride             | 66. 2-chlorotoluene                     |
| 24. tetrahydrofuran                  | 67. 1,2,3-trichloropropane              |
| 25. 1,1,1-trichloroethane            | 68. <i>trans</i> -1,4-dichloro-2-butene |
| 26. 2-butanone                       | 69. 4-chlorotoluene                     |
| 27. 1,1-dichloropropene              | 70. <i>tert</i> -butylbenzene           |
| 28. 1-chlorobutane                   | 71. 1,2,4-trimethylbenzene              |
| 29. benzene                          | 72. pentachloroethane                   |
| 30. propionitrile                    | 73. <i>sec</i> -butylbenzene            |
| 31. 1,2-dichloroethane               | 74. <i>p</i> -isopropyltoluene          |
| 32. fluorobenzene                    | 75. 1,3-dichlorobenzene                 |
| 33. trichloroethene                  | 76. 1,4-dichlorobenzene                 |
| 34. dibromomethane                   | 77. <i>n</i> -butylbenzene              |
| 35. 1,2-dichloropropane              | 78. hexachloroethane                    |
| 36. bromodichloromethane             | 79. 1,2-dichlorobenzene-d4              |
| 37. methyl methacrylate              | 80. 1,2-dichlorobenzene                 |
| 38. <i>cis</i> -1,3-dichloropropene  | 81. 1,2-dibromo-3-chloropropane         |
| 39. toluene                          | 82. nitrobenzene                        |
| 40. chloroacetone                    | 83. hexachlorobutadiene                 |
| 41. 2-nitropropane                   | 84. 1,2,4-trichlorobenzene              |
| 42. 1,1-dichloropropanone            | 85. naphthalene                         |
| 43. 4-methyl-2-pentanone             | 86. 1,2,3-trichlorobenzene              |

30m, 0.25mm ID, 1.4µm Rtx®-VMS (cat.# 19915)

**Linear velocity:** Helium @ ~1.3mL/min. constant pressure;  
**Dead time:** 2.1 min.; **Concentrator:** Tekmar LSC-3000 Purge and Trap; **Oven program:** 45°C (hold 2 min.) to 85°C @ 14°C/min. to 210°C @ 40°C/min. (hold 4 min.); **Trap:** Vocab 3000;  
**GC:** HP6890 Series II Hewlett-Packard 5973 Mass Selective Detector scan range 35 to 300 AMU; **Purge:** 11 min. @ 40mL/min.; **Dry purge:** 1 min. @ 40mL/min. (MCS bypassed);  
**Desorb preheat:** 245°C; **Desorb:** 250°C for 2 min.; **Bake:** 260°C for 8 min.; **Interface:** 1:10 split in port; **Transfer line:** 0.32mm ID Siltek® tubing (cat.# 10027)

### Standards:

20ppb in 5mL of RO water (unless otherwise noted); ketones, alcohols in 40ppb.  
 502.2 Cal Mix #1 (cat.# 30042)  
 502.2 Cal2000 MegaMix™ (cat.# 30431)  
 524 Cal Mix A&B (cat.# 30202)  
 524 Cal Mix #8 (cat.# 30203)  
 524 IS/SS Mix (cat.# 30201)





significantly affect the amount of methanol and water on the column. The "purge ready" temperature was set to 35°C, which increased the concentrator cycle time, but prevented breakthrough of the gases.

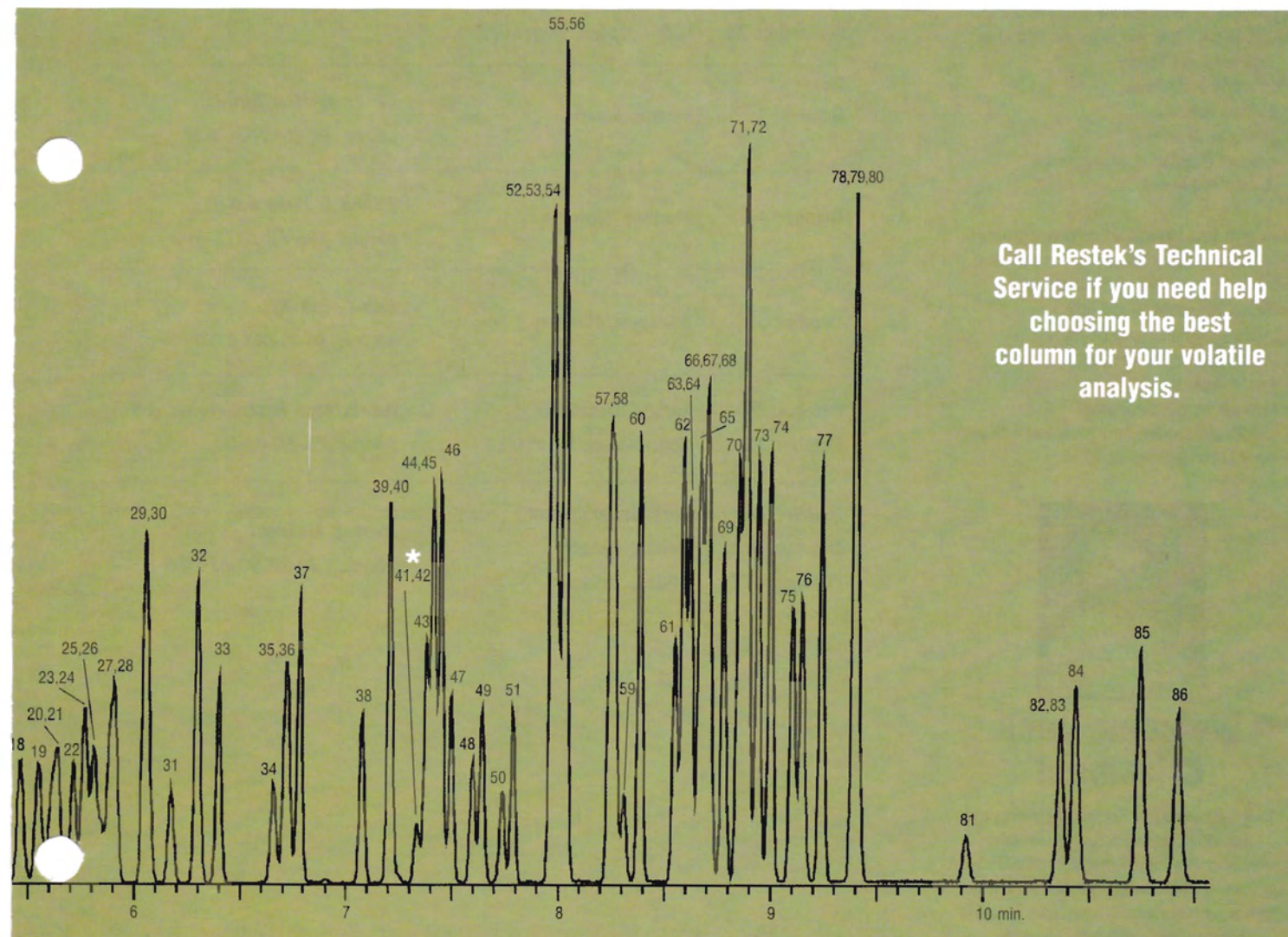
The narrow-bore (0.25mm ID) Rtx®-VMS column provides excellent sensitivity and optimized selectivity for a broad range of halogenated compounds and aromatics. Sensitivity can be increased by using a larger purge volume or a more sensitive MS. The small column ID improves sensitivity through narrower bandwidths. For Figure 1, a 10:1 split injection provided optimum flow to the MS (~1mL/min.) and helped achieve method detection limits (MDLs). The tuned selectivity of the phase prevents problems with closely-eluting compounds that share ions, such as carbon tetrachloride/1,1,1-trichloroethane and tetrahydrofuran/2-butanone. These compounds have similar spectra but are resolved by retention time. Maximum separation of substituted aromatic isomers allows a fast final oven ramp rate, preventing late-eluting compounds from coeluting and affecting quantification over a varied concentration range.

### Rtx®-VMS (Fused Silica)

ID	df (µm)	Temp. Limits	30-Meter	60-Meter
0.25mm	1.40	-40 to 240/260°C	19915	19916
0.32mm	1.80	-40 to 240/260°C	19919	19920
0.45mm	2.55	-40 to 240/260°C	19908	19909
ID	df (µm)	Temp. Limits	20-Meter	40-Meter
0.18mm	1.00	-40 to 240/260°C	49914	49915

### Siltek™ Transfer Lines/Guard Columns

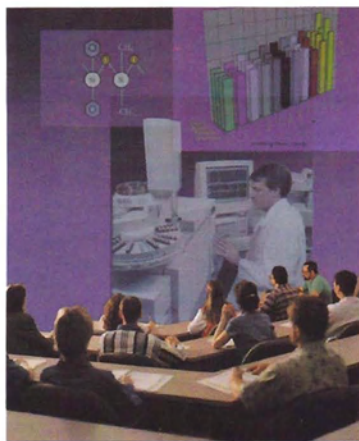
Nominal ID	Nominal OD	5-Meter	10-Meter
0.25mm	0.37 ± 0.04mm	10026	10036
0.32mm	0.45 ± 0.04mm	10027	10037
0.53mm	0.69 ± 0.04mm	10028	10038



Call Restek's Technical Service if you need help choosing the best column for your volatile analysis.

\*These peaks (41 and 42) share a quantitation ion (43)





Restek "On-the-Road" presents its Comprehensive GC Seminar series. This full-day course is presented in an engaging multimedia format. We teach key chromatographic concepts, tricks of the trade, and little known secrets that are of benefit to the novice and the seasoned veteran. We are chromatographers talking about chromatography, presenting the facts on how to help improve your chromatography analyses. This is a great opportunity to learn tips for saving time and money. The seminar will cover the following:

- Principles and Theory
- Injection Techniques
- Column Selection
- Detection Systems
- Column Installation, Maintenance & Troubleshooting

#### How will you benefit from this training?

This informative, technical seminar provides solutions to a number of challenges in the analytical laboratory. It will help you:

- Improve chromatographic efficiencies.
- Identify and adjust variables to optimize your system.
- Increase sample throughput.
- Identify and troubleshoot problems with your analysis and instrument.



## Welcome Jack Crissman!

Jack is Restek's new Technical Training and Education Manager. He has extensive chromatography knowledge and will work hard to share it with chemists worldwide. Contact Jack at [jcrissman@restekcorp.com](mailto:jcrissman@restekcorp.com) for all of your training and education needs.

# Comprehensive GC Seminar

*Coming to a location near you*

by Jack Crissman, Technical Training and Education Manager

## International Seminars

Restek's Comprehensive Capillary Chromatography Seminars will be presented in the following cities. Please contact these Restek offices for details.

Date	Location	Contact
September 25	Ulm, Germany	<b>Restek GmbH</b> phone: 06172-2797-0
September 26	Berlin, Germany	
September 27	Dusseldorf, Germany	
September 28	Ludwigshafen, Germany	
October 2	Vienna, Austria	<b>CP-Analytica GmbH</b> phone: 61-3-9762-2034
October 4	Maribor, Slovenia	<b>Mikro &amp; Polo d.o.o.</b> phone: 386-62-6373-300
October 6	Budapest, Hungary	<b>Lab-Comp Kft.</b> phone: 36-1-280-6770
October 23	Aarhus, Denmark	<b>Analytical Instruments A/S</b> phone: 44-35-02-02
October 24	Copenhagen, Denmark	
October 26	Cork, Ireland	<b>Restek Ireland</b> phone: 44-28-90-814576
October 30	Dublin, Ireland	
October 31	Belfast, Ireland	



# Rt-XLSulfur™ GC Columns

## Analyze low-level sulfur compounds in C1-C6 hydrocarbon mixes

by Barry Burger, Petrochemical Industrial Innovations Chemist

The analysis of sulfur compounds in C1-C6 hydrocarbon streams by gas chromatography (GC) is an important application in the petrochemical field. The presence of sulfur compounds in petroleum products can affect the longevity and performance of catalysts used in hydrocarbon processing. As requirements for sulfur detection become more stringent, the importance of good chromatographic separation of hydrocarbons from sulfur compounds and the inertness of the analytical columns increases.

Detectors used for sulfur determination generally are specific (e.g., sulfur chemiluminescence detection) and help eliminate positive response from chromatographic interferences. Unfortunately, when high levels of hydrocarbons elute through the detector simultaneously with sulfur compounds, the signal for sulfur is quenched and area counts are low. For a successful analysis, the analytical column must resolve the hydrocarbons listed in Figure 1 from hydrogen sulfide, carbonyl sulfide, methyl mercaptan, ethyl mercaptan and dimethylsulfide. A packed, micropacked, or PLOT column can be used to achieve this requirement.

Hydrocarbons are non-reactive but sulfur compounds, especially hydrogen sulfide and methyl mercaptan, are easily adsorbed by undeactivated surfaces. Therefore, there are two areas of concern when performing this analysis with a packed or micropacked column: one is the inertness and selectivity of the solid support, the other is the inertness of the tubing wall. Metal tubing commonly is used to construct packed and micropacked columns. Metal tubing provides ruggedness, but the surface is very adsorptive for sulfur compounds. Teflon® tubing provides excellent inertness for sulfur analysis but is permeable to contaminants in the surrounding air. Also, Teflon® tubing will expand and contract during temperature changes.

Restek designed the Rt-XLSulfur™ column to address these concerns. The packing material for Rt-XLSulfur™ columns is extensively deactivated for the analysis of low ppbv levels of hydrogen sulfide and methyl mercaptan. It is then treated to achieve the proper sensitive separation of the hydrocarbons from sulfur compounds (Figure 1).

The interior wall and the end-plugs of the Rt-XLSulfur™ column are treated with Sulfinert™ coating, a passivation technique designed to deactivate

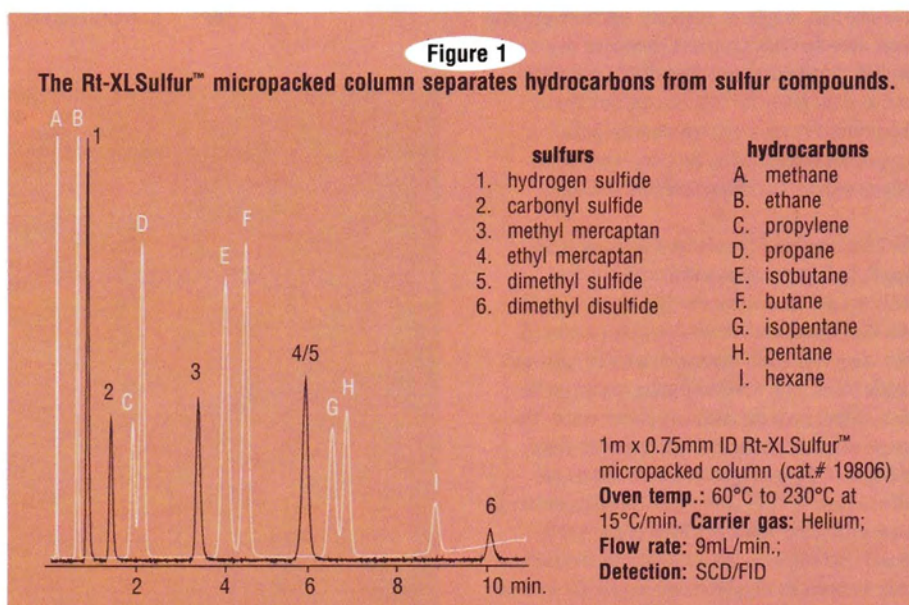
metal surfaces. The Sulfinert™ coating is very inert to hydrogen sulfide and methyl mercaptan. (For more information on Sulfinert™ coating, request lit. cat.# 59203 or visit [www.restekcorp.com/sulfinert.htm](http://www.restekcorp.com/sulfinert.htm).)

The extra care taken with the surfaces in this column result in a more accurate analysis of sulfur compounds in hydrocarbon processing. If you perform this type of analysis, try the Rt-XLSulfur™ column.

*This is the second generation of packing material for the analysis of sulfur compounds. The first packing material, in the Rt-Sulfur™ column, ensured inertness for low ppmv levels of sulfur compounds. Now, with the Rt-XLSulfur™ column, it is possible to achieve low ppbv detection of sulfur compounds.*

## for more info

For detailed information including a chromatogram illustrating a **50ppbv sulfur analysis**, request the Applications Note lit. cat.# 59165.



## Rt-XLSulfur™ Packed & Micropacked Columns\*

OD (in.)	ID (mm)	1-Meter	2-Meter
0.95mm	0.75	19806	19807
3/16	3.2	80482	80483
1/8	2.0	80484	80485
1/16	1.0	19804	19805

**\*Installation kit must be purchased with column** (no kit needed for 3/16" columns)

Kits for 0.95mm OD, 0.75mm ID columns	
For valve applications	cat.# 21062
For split applications	cat.# 21063
For all HP GCs	cat.# 21064
Kits for 1/16" OD, 1mm ID columns	
For valve applications	cat.# 21065
For direct injections	cat.# 21066
Kits for 1/8" OD, 2mm ID columns	
For valve applications	cat.# 21067

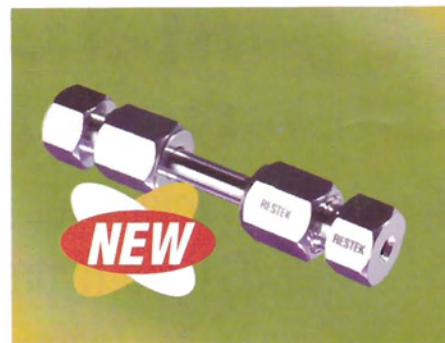


Highly polar compounds such as short-chain organic acids, nucleotides, catecholamines, and water-soluble vitamins are difficult to retain using conventional reversed phase columns, even with little or no organic solvent in the mobile phase. The Ultra Aqueous C18 column enhances the retention of polar analytes, while maintaining a high level of base deactivation and a selectivity that is similar to a conventional C18 column. Six organic acids that are difficult to retain using many conventional C18 columns can be well retained on the Ultra Aqueous C18 column (Figure 1)

The unique qualities of the Ultra Aqueous C18 column are apparent when comparing this column to the Ultra C18 column—a highly retentive, well-deactivated, general-purpose C18 column—in a separation of polar compounds. The Ultra Aqueous C18 column has very similar retention and selectivity to the Ultra C18 column when analyzing neutral, hydrophobic compounds. While phenol, a neutral polar compound, is retained similarly by both columns (Figure 2), the basic compound pyridine is retained approximately 2.5 times longer on the Ultra Aqueous C18 column. Also note that, in spite of its greater retention, pyridine elutes from the Ultra Aqueous C18 column as a relatively symmetrical peak (pyridine is commonly used as a test probe for column base deactivation). This proves that enhanced retention for polar compounds is achieved on the Ultra Aqueous C18 column without sacrificing base deactivation.

The Ultra Aqueous C18 column is designed using Type B, high-purity silica, and a novel bonding chemistry that produces a true C18 phase (USP L1) with alkyl chains that remain completely extended, even when continually exposed to a highly aqueous mobile phase. This is because polar groups on the silica surface keep the stationary phase wetted. The unique secondary polar character prevents chain folding (i.e., the hydrophobic C18 chains do not self-associate or “fold down” onto the silica surface to avoid associating with a very hydrophilic mobile phase), and enhances the retention and selectivity of polar compounds without compromising the level of base deactivation. Ultimately, this means that the Ultra Aqueous C18 column offers stable and reproducible retention, even with 100% aqueous mobile phases.

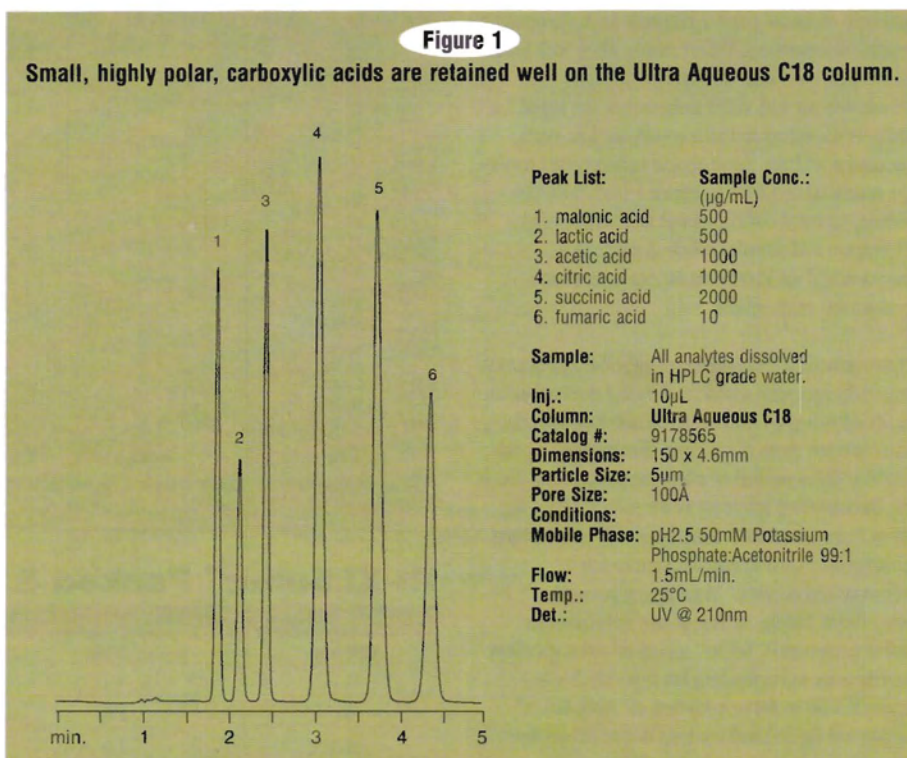
## Ultra Aqueous C18 HPLC Column



### Reversed-phase analysis of polar compounds

by Terry Reid, HPLC R&D Chemist

- ✓ Permits use of highly aqueous mobile phases
- ✓ Enhanced retention and selectivity for polar analytes
- ✓ Excellent base deactivation



### Ultra Aqueous C18 HPLC Columns, 5µm

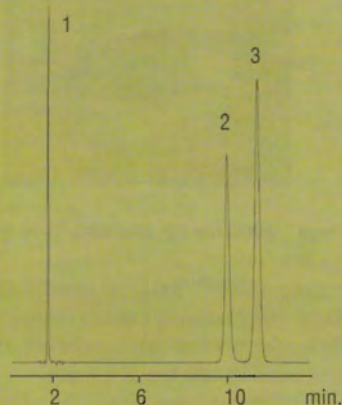
Particle Size: 5µm length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm	9178531	9178532	9178533	9178535
50mm	9178551	9178552	9178553	9178555
100mm	9178511	9178512	9178513	9178515
150mm	9178561	9178562	9178563	9178565
200mm	9178521	9178522	9178523	9178525
250mm	9178571	9178572	9178573	9178575



Figure 2

The Ultra Aqueous C18 column provides enhanced retention of pyridine while maintaining a high level of base deactivation.

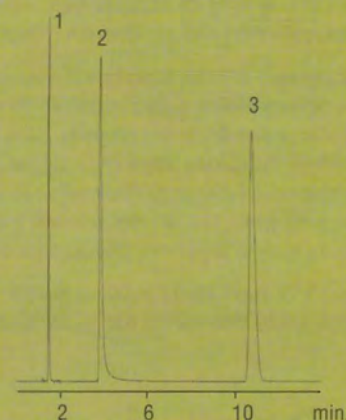
**Ultra Aqueous C18** (cat.# 9178565)  
Dimensions: 150x4.6mm; Particle Size: 5µm;  
Pore Size: 100Å



**Peak List:** 1. uracil  
2. pyridine  
3. phenol

**Conditions:**  
Mobile Phase: 80:20 pH 7.0,  
20mM potassium  
phosphate  
acetonitrile  
Flow: 1.0mL/min.  
Temp: 25°C  
Det.: UV@254nm

**Base-deactivated C18**  
Dimensions: 150x4.6mm; Particle Size: 5µm;  
Pore Size: 100Å



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Restek's new, 68-page **HPLC Columns and Accessories Catalog** features Restek's complete line of HPLC columns and accessories, along with helpful tech tips and over 75 application chromatograms. Chemists will find the column selection and US Pharmacopoeia sections useful. Request yours today! (lit. cat# 59241)

To access HPLC literature and applications **on-line**, go to [www.restekcorp.com/library.htm](http://www.restekcorp.com/library.htm).

## HPLC Application Notes

Improved HPLC Analysis of Analgesics (#59511)  
The Ultra IBD Column Allows HPLC Separation of Polar and Non-Polar Analytes from the Same Sample (#59512)  
HPLC Stationary Phase Selection for the Analysis of Steroids (#59510)  
Allure™ PFP Propyl HPLC Column Provides Improved LC/MS Analyses of Basic Compounds (#59118A)

## HPLC Fast Facts

HPLC Mobile Phase Accessories (#59728)  
Trident™ Integral HPLC Guard Column System (#59896)  
HPLC and LC/MS Column Kits (#59302)  
Allure™ Acidix HPLC Columns (#59303)  
Trident™ Direct Guard Column System (#59314)  
Ultra IBD HPLC Columns (#59614A)



Restek's **HPLC Tech Tips Wall Chart** (lit. cat.# 59894) features step-by-step instructions on HPLC setup and storage. The chart also includes quick reference tables on miscibility and solubility, pressure conversion factors, buffers and other valuable tips to enhance your chromatography.



# EZ-Vent™ 2000

## Saving you time and money when you switch columns in your MS\*

We designed a common sense, affordable solution to the hassle of changing columns in your mass spectrometer (MS). By using the Restek EZ-Vent™ 2000 interface, you can avoid the typical, lengthy vent and pump-down cycles every time you change a column.

The EZ-Vent™ 2000 MS interface will revolutionize the way you connect columns to your MS. When the outlet of a capillary column is inserted into an MS vacuum, the negative pressure propagates several meters inside the column thus decreasing efficiency. Using the EZ-Vent™ 2000 interface minimizes this effect, thereby increasing the effective length of the column. The EZ-Vent™ 2000 interface does not require any additional plumbing and works on a critical orifice principle, thereby eliminating the need to plumb your MS interfaces with purge gases. Plus, it's easy to install! Just connect the EZ-Vent™ 2000 transfer line and connectors and you are ready to begin using your MS without worry of pumping it down during the next column change.

Restek's EZ-Vent™ 2000 interface is available for Hewlett-Packard GCs with a 5971/5972 or 5973 MS and Varian Saturn 2000 systems with 3400, 3600, or 3800 GCs.

### Feature

Decreased column changing time.

Cost effective.

Less Expensive.

Very low dead volume fittings and small ID tubing.

All metal components are Silcosteel®-coated and transfer lines are deactivated for maximum inertness.

### Benefit

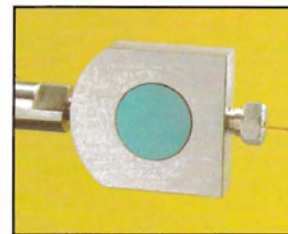
Saves time.

Costs associated with cool-downs and venting are now investments in analytical runs.

Save money compared to other models with equal performance.

Ensure no band broadening or change in analytical performance.

Less adsorption or loss of reactive compounds = more accurate analyses.



### EZ-Vent™ 2000 for HP GCs with 5971/5972 or 5973 MS:

Includes EZ-Vent™ 2000, 1/16" SS nut, 0.4mm ID ferrules for connecting capillary column, 0.4mm ID ferrules for connecting transfer line, 100µm deactivated transfer line (3 ft.), and EZ-Vent™ column plug; cat.# 21013, (kit)

### EZ-Vent™ 2000 for Varian Saturn 2000 systems with 3400, 3600, or 3800 GCs:

Includes EZ-Vent™ 2000, 1/16" SS nut, 0.4mm ID ferrules for connecting capillary column, 0.4mm ID ferrules for connecting transfer line, 100µm deactivated transfer line (3 ft.), and EZ-Vent™ column plug; cat.# 21014, (kit)

### Replacement EZ-Vent™ 2000 ferrules for connecting capillary column to EZ-Vent™:

0.4mm ID: cat.# 21015, (2-pk.)

0.5mm ID: cat.# 21016, (2-pk.)

### Replacement EZ-Vent™ 2000 ferrules for connecting transfer line to EZ-Vent™:

0.4mm ID: cat.# 21043, (2-pk.)

### Replacement EZ-Vent™ 2000 union:

cat.# 21017, (ea.)

### Replacement EZ-Vent™ 2000 deactivated transfer line :

100µm: cat.# 21018, (3 ft.)

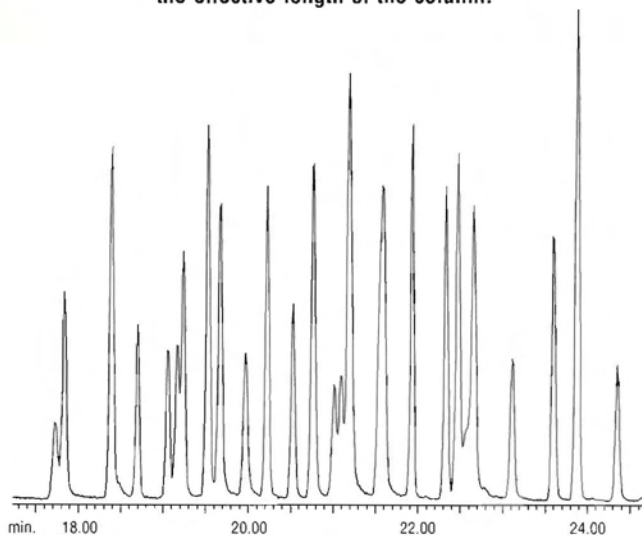
### Open-end wrench tool (1/4"- & 5/16"-inch):

cat.# 20110, (2-pk.)

### Replacement EZ-Vent™ column nut:

cat.# 21072, (2-pk.)

**Figure 1** The EZ-Vent™ interface causes no peak broadening and increases the effective length of the column.



30m, 0.25mm ID, 0.5µm Rtx®-5MS (cat.# 12638)

4mm single gooseneck liner with fused silica wool (cat.# 22405)

Inj.: 50ng/µL semivolatiles standard; Oven temp.: 35°C (hold 1 min.) to 300°C @ 10°C/min. (hold 20 min.); Splitless hold time: 1 min.; Inj. temp.: 300°C; Det. temp.: 310°C; Det.: HP 5971A MS, full scan; Carrier gas: helium @ 15psi

## for more info

Request the EZ-Vent™ 2000 Fast Facts  
(lit. cat.# 59307).

Recently, your feedback on the EZ-Vent™ 2000 has been invaluable in solving column switching problems in MS instruments. Restek is committed to being the source of solutions for your analytical problems. This philosophy is at the core of all services and products that Restek offers. During the development cycle of a product, our Innovation Teams focus on addressing customer needs and making the product easy to use. We solicit input from analysts and use extensive beta testing to prove product design and technology.

\* Available for HP GCs with a 5971/5972 or 5973 MS and Varian Saturn 2000 systems with 3400, 3600, or 3800 GCs.



# Peak Performers

## Precision™ Inlet Liners (Formerly called Focal liners)

- ✓ Wool is placed at the injection point to maximize vaporization and help wipe the needle during injection.
- ✓ Designed for easy changing of the wool—no more guessing where the wool should be placed.
- ✓ Wool stays in position during pressure pulses in the inlet and during injection.
- ✓ Available with all Restek deactivations and packing materials.
- ✓ Direct replacement for SGE's Focus™ liners.

Instrument	each	5-pk.
HP 5890/6890 4mm Split Precision™ Liner	21022	21023
Varian 1078/1079 Split Precision™ Liner	21024	21025
Shimadzu 17A Split Precision™ Liner	21020	21021
Varian 1075/1077 Split Precision™ Liner	21030	21031
Fisons, Trace, 8000 Series 5mm Split Precision™ Liner	21028	21029
PE Auto SYS Split Precision™ Liner	21026	21027

## Encapsulated Ferrules



- ✓ Aluminum encapsulated.
- ✓ Will not deform and stick in fittings.
- ✓ Allows ferrule to be reused.
- ✓ Less torque needed to seal ferrule.
- ✓ Unique blend of graphite minimizes fragmentation and outgassing.
- ✓ For 1/16" compression fittings.

Ferrule ID	Fits column ID	cat.#	10-pk.
0.4mm	0.25mm	21036	
0.5mm	0.32mm	21037	
0.8mm	0.53mm	21038	

## MXT® Connectors

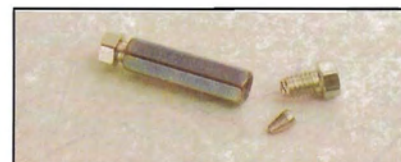
- Stainless steel construction means no more breakage.
- Silcosteel®-treated for inertness, causes no peak tailing.
- Low dead volume minimizes peak tailing.
- Connects metal capillary tubing to fused silica capillary tubing.
- 1/32" union uses metal ferrules with metal tubing, Valcon polyimide ferrules for connecting fused silica tubing.

**MXT® Connector:** for 0.28mm ID columns: cat.# 20397 (ea.)  
for 0.53mm ID columns: cat.# 20394 (ea.)  
**MXT® "Y" Connector:** for 0.28mm ID columns: cat.# 20396 (ea.)  
for 0.53mm ID columns: cat.# 20395 (ea.)

### Connector Replacement Nut and Ferrules

1/32" Replacement Nut: cat.# 20389 (5-pk.)  
1/32", 0.4mm Vespel®/graphite ferrule,  
for use with fused silica tubing in an MXT®  
connector: cat.# 21039 (5-pk.)  
1/32", 0.5mm Vespel®/graphite ferrule,  
for use with fused silica tubing in an MXT®  
connector: cat.# 20259 (5-pk.)

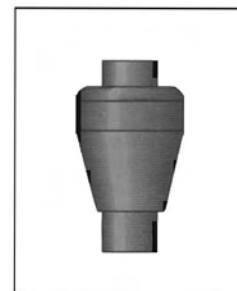
1/32" Stainless Steel Replacement Ferrules for MXT® Connectors			
Ferrule ID	Fits Column	cat.#	10-pk.
0.59mm	0.28mm ID	20398	
0.79mm	0.53mm ID	20399	



## Valco® One-Piece Fused Silica Adaptors

- For use in fittings where the ferrule will not be removed.
- Valcon polyimide for use up to 350°C.
- Compatible with fused silica capillary columns in MXT® connectors.

1/32" Adaptor, 5-pk.			
Tubing OD (mm)	Tubing ID (mm)	Valco® #	Valcon Polyimide
0.25–0.4	0.25	FS.4-5	20137, (5-pk.)
0.4–0.5	0.32	FS.5-5	20140, (5-pk.)
0.5–0.8	0.53	FS.5V-5	20141, (5-pk.)





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the

Innovators of High Resolution Chromatography Products

# RESTEK Advantage

## New Rtx®-VMS Revolutionary New Column Designed for GC/MS Analysis

by Chris English, Environmental Applications Chemist



In the past, stationary phases for gas chromatography (GC) were developed without consideration for their final application. This resulted in long analysis times, high bleed, and coelutions. With the use of computer modeling, Restek has been able to create a new stationary phase that achieves the fewest number of coelutions and the fastest analysis time for the separation of volatile organic compounds, such as those listed in US Environmental Protection Agency (EPA) Method 8260.

### Optimizing performance for GC/MS systems yields higher productivity.

Restek has developed the new Rtx®-VMS column with lower bleed, better selectivity, and an overall faster analysis time than the "624/1301" phase. The Rtx®-VMS stationary phase is a highly stable polymer designed to provide outstanding analysis of volatile compounds using the more sensitive ion traps and HP 5973 mass spectrometers (MS).

Oven cycle time, not analysis time, is the most important factor in increasing productivity. The Rtx®-VMS column was designed for a 40°C starting GC oven temperature, allowing a shorter total oven cycle time (Table I), without sacrificing resolution of the six gases listed in Method 8260.

The Rtx®-VMS column has been optimized in multiple internal diameters; 0.18, 0.25, 0.32, 0.45, and 0.53mm ID, giving the analyst choices on how they prefer to run volatile compounds. The 0.18 and 0.25mm ID columns allow splitting at the injection port, eliminating the added expense and maintenance of a jet-separator. The 0.45mm ID column is recommended because it has a faster analysis time and better efficiency over the 0.53mm ID equivalent, and still has sufficient flow to sweep the sample off the trap and onto the column. The 0.45mm ID Rtx®-VMS optimized phase and column dimensions produce a 17-minute analysis time (Figure 1, pg. 2).

Table I

HP 5890 oven cool-down time decreases drastically when starting at 40°C.

Temperature (C°)	Cool Down Time (min.)
220 to 40	6.0
240 to 40	7.5
220 to 35	9.5
240 to 35	10.5

### Rtx®-VMS columns overcome the challenge of coelutions.

The Rtx®-VMS column was designed to resolve all compounds using primary quantitation ions. Coeluting compounds cannot share quantitation ions or have minor ions that interfere with the primary ion of the coeluting analyte. US EPA methods updates such as Method 524.2, rev. IV, have added coeluting compounds with minor ions of one compound interfering with the quantitation of another compound. An example of this problem occurs when using the "624/1301" 75m x 0.53mm ID column with methyl acrylate and propionitrile. The quantitation ion for methyl acrylate is 55. Propionitrile has a minor ion of 55, which can interfere with determining actual concentrations of methyl acrylate in "real world" samples. Another

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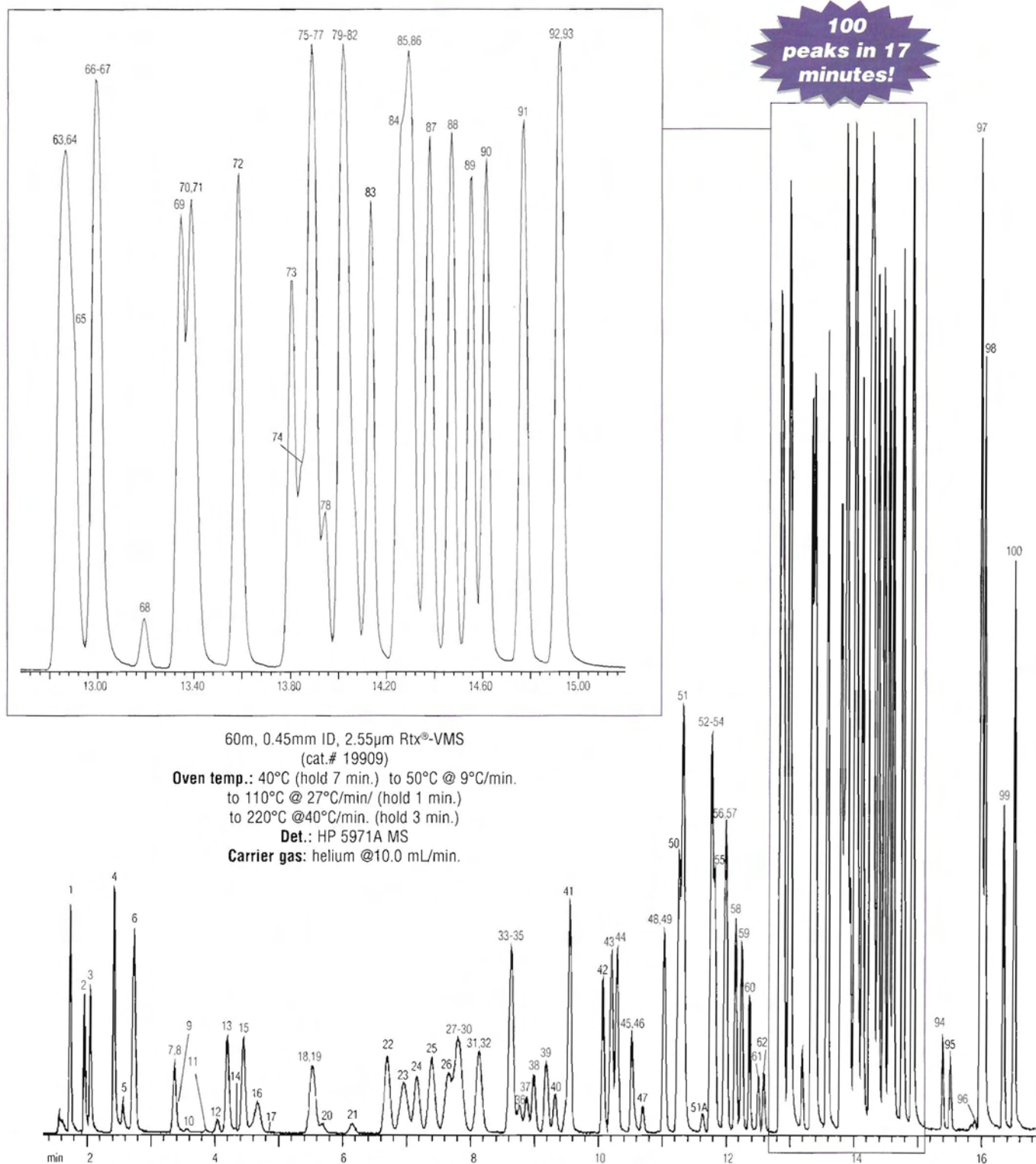


# New Rtx<sup>®</sup>-VMS

## Faster and Better Volatile Organics GC/MS Analysis

Figure 1

A 0.45mm ID Rtx<sup>®</sup>-VMS column analyzes US EPA Method 8260 compounds in less than 17 minutes.



Acknowledgment: Special thanks for technical contribution to Darrel Robbins, STL Laboratories, Burlington, VT.



1. dichlorofluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethane
8. carbon disulfide
9. Freon® 113
10. iodomethane
11. acrolein
12. allyl chloride
13. methylene chloride
14. acetone
15. *trans*-1,2-dichloroethene
16. methyl-*tert*-butyl-ether
17. *tert*-butyl alcohol
18. chloroprene
19. 1,1-dichloroethane
20. acrylonitrile
21. vinyl acetate
22. *cis*-1,2-dichloroethene
23. 2,2-dichloropropane
24. bromochloromethane
25. chloroform
26. carbon tetrachloride
27. tetrahydrofuran
28. methyl acrylate
29. ethyl acetate
30. 1,1,1-trichloroethane
31. 2-butanone
32. 1,1-dichloropropene
33. propionitrile
34. benzene
35. methacrylonitrile
36. 1,2-dichloroethane-d4
37. 1,2-dichloroethane
38. isobutyl alcohol
39. fluorobenzene
40. isopropyl acetate
41. trichloroethene
42. dibromomethane
43. 1,2-dichloropropane
44. bromodichloromethane
45. methyl methacrylate
46. 1,4-dioxane
47. *n*-propyl acetate
48. 2-chloroethyl-vinyl-ether
49. *cis*-1,3-dichloropropene
50. toluene-d8
51. toluene
- 51A. 1,1-dichloro-2-propanone
52. 4-methyl-2-pentanone
53. tetrachloroethene
54. *trans*-1,3-dichloropropene
55. 2-bromo-1-chloropropane
56. 1,1,2-trichloroethane
57. ethyl methacrylate
58. dibromochloromethane
59. 1,3-dichloropropane
60. 1,2-dibromoethane
61. *n*-butyl acetate
62. 2-hexanone
63. chlorobenzene
64. ethylbenzene
65. 1,1,1,2-tetrachloroethane
66. *m*-xylene
67. *p*-xylene
68. 1-chloro-2-fluorobenzene
69. *o*-xylene
70. styrene
71. bromoform
72. isopropylbenzene
73. 4-bromo-1-fluorobenzene
74. *cis*-1,4-dichloro-2-butene
75. bromobenzene
76. 1,4-dichlorobutane
77. *n*-propylbenzene
78. 1,1,2,2-tetrachloroethane
79. 2-chlorotoluene
80. 1,2,3-trichloropropane
81. 1,3,5-trimethylbenzene
82. *trans*-1,4-dichloro-2-butene
83. 4-chlorotoluene
84. *tert*-butylbenzene
85. pentachloroethane
86. 1,2,4-trimethylbenzene
87. *sec*-butylbenzene
88. *p*-isopropyltoluene
89. 1,3-dichlorobenzene
90. 1,4-dichlorobenzene
91. *n*-butylbenzene
92. 1,2-dichlorobenzene-d4
93. 1,2-dichlorobenzene
94. 4-bromo-1-chlorobenzene
95. 1,2-dibromo-3-chloropropane
96. nitrobenzene
97. hexachlorobutadiene
98. 1,2,4-trichlorobenzene
99. naphthalene
100. 1,2,3-trichlorobenzene

difficult pair to separate on the "624/1301" column is 1,1-dichloro-2-propanone and 4-methyl-2-pentanone, which share ion 43. These compounds can be resolved on the "624/1301" 60m x 0.32mm ID column in more than 30 minutes. The Rtx®-VMS column was designed specifically to overcome these challenges and those of the branched aromatics, which share parent ions, in less time than any other column available.

#### Interfacing the column to the MS ion source.

Conventional MS systems operate optimally when the flow of carrier gas into the ionizing chamber is 1mL/min. Flow rates higher than this will increase noise at a greater rate than the increase in sensitivity, resulting in a net loss of the signal-to-noise ratio. Higher flow rates also strain turbo-molecular pumps, adding to maintenance and downtime. The Rtx®-VMS column in 0.18mm and 0.25mm IDs are run optimally at flow rates of 1mL/min, thereby, allowing a direct interface to the MS ion source. To interface the 0.32, 0.45, and 0.53mm ID columns to the MS and keep the flow at 1mL/min, an open split interface (OSI) or jet separator is used.

The OSI generally splits off the carrier gas 10:1, resulting in a 10-fold decrease in sample quantity entering the detector. Due to more sensitive instrumentation like the ion trap and HP5973 quadrupole MS, splitting of the sample at the MS interface still allows for method detection limits (MDLs) to be achieved. Less sensitive instrumentation can overcome the loss of sample and achieve the detection limit required by US EPA Method 8260 and 524.2, rev. IV, by increasing the sample size

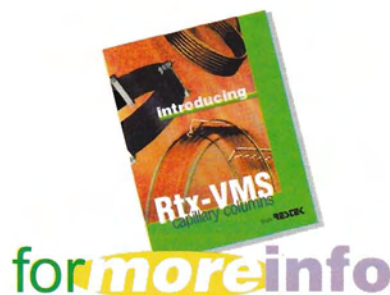
from 5mL to 25mL, thus introducing approximately five times more sample to the column. However, it is more difficult to achieve MDLs for the ketones and other poor-purging compounds using 25mL volume of sample.

The jet separator uses a vacuum pump to pull the carrier gas away from the analytes, allowing a majority of sample to pass into the MS source. Except for the most volatile compounds, 90% of the analytes from the column reach the MS detector. This method is more expensive than the OSI and requires additional maintenance, but the analysis can be accomplished by purging only 5mL of sample to achieve detection limits on less sensitive instrumentation.

Choosing the internal column diameter of the Rtx®-VMS column to interface with the MS ion source is a matter of preference. The Rtx®-VMS column is available in 0.53, 0.45, 0.32, 0.25, and 0.18mm ID column configurations, allowing you to decide which dimension is best for your analysis.

#### Conclusion

The Rtx®-VMS column is the first in a series of volatile columns that Restek has designed to target specific method requirements. Using computer modeled stationary phase development, it is possible to produce a column with unsurpassed separation of US EPA Method 8260B and 524.2, rev. IV, analytes. This column is capable of accurate and fast GC separations, even at a 40°C starting temperature, and exhibits exceptionally low bleed at 240°C allowing optimum performance with ion trap and HP5973 MS systems.



Request the Rtx®-VMS Benefits Brochure  
(lit. cat.# 59209).

#### Rtx®-VMS (Stable to 280°C)

ID	df (µm)	Temp. Limits	30-Meter	60-Meter	105-Meter
0.25mm	1.40	-40 to 240/260°C	19915	19916	—
0.32mm	1.80	-40 to 240/260°C	19919	19920	—
0.45mm	2.55	-40 to 240/260°C	19908	19909	—
0.53mm	3.00	-40 to 240/260°C	19958	19988	19974
ID	df (µm)	Temp. Limits	20-Meter	40-Meter	
0.18mm	1.00	-40 to 240/260°C	49914	49915	

If you're analyzing volatile compounds,  
attend our upcoming

**Environmental GC Seminar.**

See page 14 for more information.



# Siltek® Deactivation

## Minimize Breakdown of Chlorinated Pesticides

by Deb Salabsky, Applications Chemist

Gas chromatographic (GC) analysis of chlorinated pesticides presents unique challenges to environmental laboratories because the compounds are analyzed at trace levels and are susceptible to decomposition. Analytical methods have stringent breakdown and reproducibility criteria (such as the US Environmental Protection Agency (EPA) Methods 8081 and 608).

Breakdown occurs when a compound decomposes into related compounds, generally from a thermal or chemical reaction that has taken place. Two pesticides prone to breakdown are endrin, which breaks down into endrin aldehyde and endrin ketone, and DDT, which breaks down into DDE and DDD. The source of breakdown is most commonly caused by active sites in the GC system. Routine maintenance of the injection port, prevention of sample flashback, and thorough deactivation of the inlet liner and GC columns are essential to minimize compound breakdown.

A study on the effect of inlet liner deactivation on endrin and DDT breakdown shows that breakdown can be decreased drastically by using Siltek™ products (Figure 1). When a direct injection of a pesticide standard is injected into an untreated glass Uniliner®, endrin breakdown is calculated at 62% and DDT breakdown is less than 1%. In a Siltek™ Uniliner® inlet liner, endrin and DDT breakdown measured less than 1%.

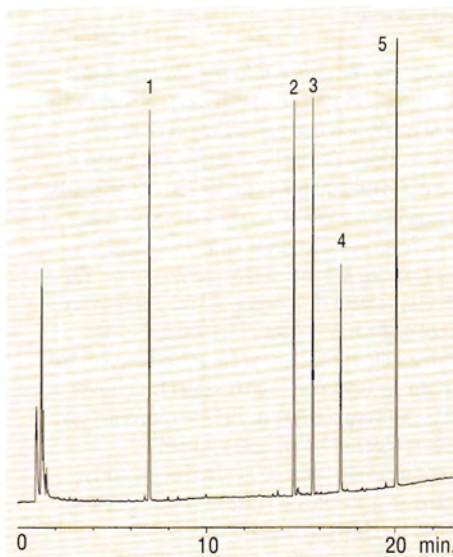
We also studied the effects of hot metal surfaces on endrin and DDT breakdown. For more information, request Applications Note #59111.

Our research demonstrates that direct injection into a Siltek™ Uniliner® inlet liner provides the best protection against problematic breakdown in the injection port when analyzing chlorinated pesticides. Endrin is more prone to breakdown on glass surfaces than metal, and DDT is more prone to breakdown on metal surfaces. Restek's Siltek™ deactivation can yield a minimal endrin breakdown of 1%!

For a complete, highly inert pathway for chlorinated pesticides, Restek also offers Rtx®-CLPesticides and Rtx®-CLPesticides2 columns and Siltek™ guard columns.

### Now available!

New, improved Siltek™ pesticide column kits! Kit includes a Rtx®-CLPesticides column and a Rtx®-CLPesticides2 column; a 5m Siltek™ guard column; and a Siltek™ universal, angled "Y" Press-Tight® connector. (Note: columns are not pre-connected in these kits).



**Figure 1**

**A direct flash injection using a Siltek® inlet liner results in less than 1% breakdown for endrin and DDT.**

1. 2,4,5,6-tetrachloro-*m*-xylene
2. endrin
3. 4,4'-DDT
4. methoxychlor
5. decachlorobiphenyl

30m, 0.53mm ID, 0.42µm Rtx®-CLPesticides2 (cat.# 11340).

Inj.: 1µL of 50pg/µL standard.

Oven temp.: 40°C (hold 1 min.) to 300°C @ 9°C/min. (hold 10 min.)

Inj. temp.: 250°C

Det.: ECD, 300°C

Carrier gas: helium.

### Siltek™ Inlet Liners

Refer to the *Restek 2000 Chromatography Products Guide* or call customer service for liner cat.#'s, then add one of the following suffixes to receive Siltek™ liners.

qty.	Siltek™		Siltek™ inlet liner with Siltek™ wool		Siltek™ inlet liner with CarboFrit™	
each	-214.1,	addl. cost	-213.1,	addl. cost	-216.1,	addl. cost
5-pk.	-214.5,	addl. cost	-213.5,	addl. cost	-216.5,	addl. cost
25-pk.	-214.25,	addl. cost	-213.25,	addl. cost	-216.25,	addl. cost

### Siltek™ Guard Columns

nominal ID	nominal OD	5-meter	10-meter
0.25mm	0.37 ±0.04mm	10026	10036
0.32mm	0.45 ±0.04mm	10027	10037
0.53mm	0.69 ±0.04mm	10028	10038

### Siltek™ Press-Tight® Connectors

type	qty.	cat.#
straight	25-pk.	20449
angled "Y"	3-pk.	20469

### Rtx®-CLPesticides Kits

ID (mm)	cat.#
0.25	11199
0.32	11198
0.53	11197

### Siltek™ Borosilicate Wool

qty.	cat.#
10 g	21100



# USP <467> Updated Calibration Mixtures Match European Pharmacopoeia Limits

by Christopher Cox, Senior R&D Chemist

The United States Pharmacopoeia (USP) <467> Method has been revised in the 24th edition of USP, effective January 1, 2000<sup>1</sup>. The biggest change was to the limit test concentrations for all five residual solvents. The revised test concentrations for the five USP <467> residual solvents are now the same as the concentrations used by the European Pharmacopoeia<sup>2</sup> and as adopted by the International Conference on Harmonization<sup>3</sup>.

Restek has developed two new products to meet this change. These products (cat.# 36006, dissolved in methanol, and cat.# 36007, dissolved in dimethylsulfoxide) are now available from stock for immediate shipment. The Rtx®-G43 GC column is the best choice for USP <467> (Figure 1).

1. USP 24 - NF 19, Organic Volatile Impurities <467>, p. 1877-1878.
2. European Pharmacopoeia, Supplement 1999, pp. 14- 15, 208.
3. International Conference on Harmonization, Note for Guidance on impurities: Residual Solvents, July 17, 1997.

References not available from Restek.

**Figure 1**

**Rtx®-G43 column provides the resolution and detection limits needed for USP <467> revised limit test concentrations.**

1. methylene chloride	600µg/mL
2. chloroform	60
3. benzene	2
4. trichloroethylene	80
5. 1,4-dioxane	380

30m, 0.53mm ID, 3.0µm Rtx®-G43 with 5m phenylmethyl Integra-Guard™ guard column (cat.# 16085-126)

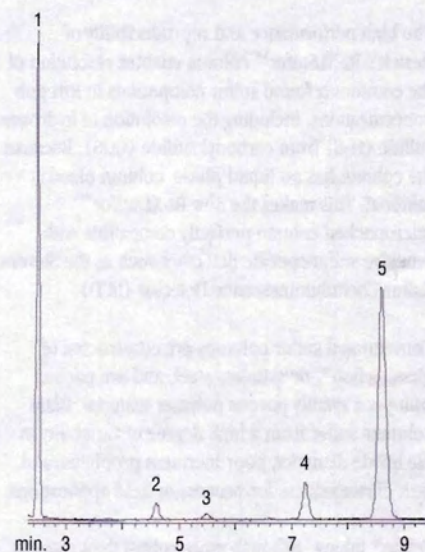
1µL direct injection of USP <467> Calibration Mix #5 (cat.# 36007)

Oven temp.: 40°C (hold 20 min.) to 240°C @ 35°C/min. (hold 10 min.)

Inj.: 180°C

Carrier gas: helium, 40cm/sec.

Det.: FID, 260°C, 1 x 10<sup>-11</sup>AFS



## USP <467> Calibration Mix #4

Prepared in methanol, 1mL per ampul.

benzene	2µg/mL
chloroform	60
1,4-dioxane	380
methylene chloride	600
trichloroethene	80

Each	10-pk.
36006	36106

## USP <467> Calibration Mix #5

Prepared in dimethylsulfoxide, 1mL per ampul.

benzene	2µg/mL
chloroform	60
1,4-dioxane	380
methylene chloride	600
trichloroethene	80

Each	10-pk.
36007	36107

## Rtx®-G43 Column

30m, 0.53mm ID, 3.0µm with 5m, 0.53mm ID Integra-Guard™ column

Temp. limit: -60 to 240°C

cat.# 16085-126



**for more info**

Request the *Siltek™ Deactivation Brochure*  
(lit. cat.# 59803).

*“You guys did a great job!”*

That's what John Syslo, an analytical chemist at Lockheed-Martin, says about Siltek™ deactivation. Pleased with the results of an analysis performed with a Siltek™ deactivated liner, John explains, "I have evaluated the Siltek™ liners against standard silanized deactivation and found significantly lower breakdown for endrin and DDT. The Siltek™ liners are superior to any other deactivation I've seen in relation to inertness."



# Rt-XLSulfur™

## Designed for Low-Concentration Sulfur Analysis

by Barry Burger, Petrochemical Applications Chemist

Restek's research and development team has designed the ultimate micropacked column specifically for the separation and quantitation of COS, H<sub>2</sub>S, SO<sub>2</sub>, CH<sub>3</sub>SH, (CH<sub>3</sub>)<sub>2</sub>S and (CH<sub>3</sub>)<sub>2</sub>S<sub>2</sub> at low ppb levels. These sulfur compounds typically are found in pulp mill by-products, natural gas, and petroleum products. Their reactive properties make them difficult to handle, transport, and analyze, especially at concentrations below 1 ppm.

The high performance and reproducibility of Restek's Rt-XLSulfur™ column enables resolution of the commonly found sulfur compounds in low ppb concentrations, including the resolution of hydrogen sulfide (H<sub>2</sub>S) from carbonyl sulfide (COS). Because the column has no liquid phase, column bleed is minimal. This makes the new Rt-XLSulfur™ micropacked column perfectly compatible with sensitive sulfur-specific detectors such as the Sievers Sulfur Chemiluminescence Detector (SCD).

Conventional sulfur columns are constructed of glass, Teflon®, or stainless steel, and are packed with poor quality porous polymer material. Glass columns suffer from a high degree of variability in the inside diameter, poor inertness problems, and lack of ruggedness for process or field applications.

Teflon® tubing, although more robust than glass, is plagued by three distinct problems: (1) Shrinkage upon oven cool-down results in carrier gas leakage and subsequent back-diffusion of O<sub>2</sub> and H<sub>2</sub>O into the packing material. If not corrected, retention times can vary as much as 15%. (2) O<sub>2</sub> and H<sub>2</sub>O diffusion through the tubing wall significantly decreases column longevity and causes reproducibility problems. (3) Maximum temperature of only 210°C limits the rapid analysis of high molecular weight sulfur compounds.

Stainless steel columns are not recommended because they can absorb low-level sulfur compounds such as H<sub>2</sub>S and methyl mercaptan.

To achieve the high degree of inertness required for the analysis of these reactive, low-level sulfur compounds every aspect of the sample pathway must be addressed (i.e., the porous polymer packing material, column tubing, column end closures, inlet sleeve, and sample loop). The porous polymer has a unique surface modification, which results in excellent peak symmetry and thermal stability to 300°C. There is virtually no bleed after initial installation, purge, and a brief conditioning period. The column tubing, column end closures, inlet sleeve, and sample loop are deactivated with

- ✓ Sulfinert™ deactivation allows low ppb analysis of sulfur compounds.
- ✓ Batch-tested for reproducibility.
- ✓ Low bleed and high thermal stability to 300°C.
- ✓ Rugged column results in longer life and easy field use.

Restek's new revolutionary Sulfinert™ deactivation process, the most inert surface available for trace sulfur compounds. Deactivation of the inlet system for sulfur compounds can provide a high sensitivity and reliability for your low-level sulfur analyses.

The Rt-XLSulfur™ column can resolve several common sulfur compounds at 50ppb levels in nitrogen using the SCD (Figure 1). At this demanding level, column bleed, peak symmetry, and resolution of the sulfurs is excellent.

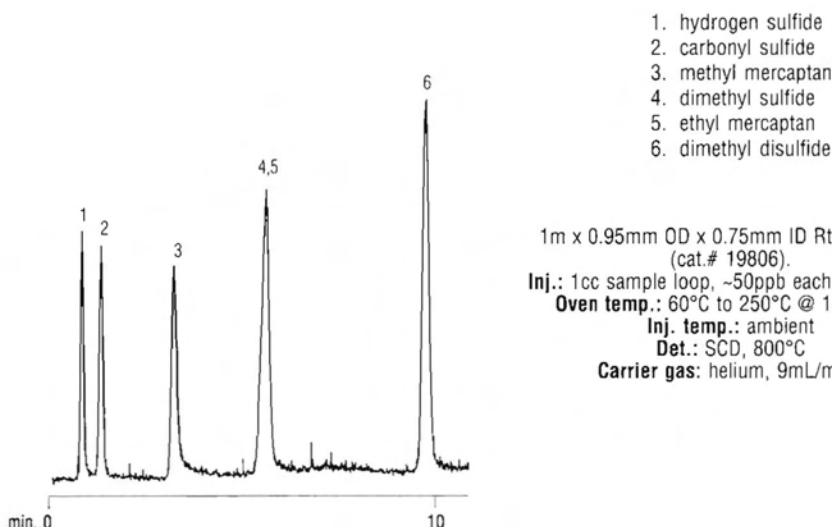
The Rt-XLSulfur™ micropacked column is the one capable of analyzing ppb levels of sulfurs while still offering all the characteristics required in a GC column: inertness, low bleed, high sample capacity, and robustness.

**formoreinfo**

For more information on Sulfinert™ coatings, request lit. cat.# 59203.

**Figure 1**

The Rt-XLSulfur™ analyzes 50ppb levels of sulfur compounds, providing low bleed and good peak symmetry.



1m x 0.95mm OD x 0.75mm ID Rt-XLSulfur™  
(cat.# 19806).  
Inj.: 1cc sample loop, ~50ppb each component  
Oven temp.: 60°C to 250°C @ 15°C/min.  
Inj. temp.: ambient  
Det.: SCD, 800°C  
Carrier gas: helium, 9mL/min..

Rt-XLSulfur™ Packed & Micropacked Columns			
OD (in.)	ID (mm)	1-meter	2-meter
3/16	3.2	80482	80483
1/8	2.0	80484	80485
1/16	1.0	19804	19805
0.95mm	0.75	19806	19807



# Rtx®-VRX

## Specialized for GC Analysis of Volatile Compounds

by Christopher English, Environmental Applications Chemist

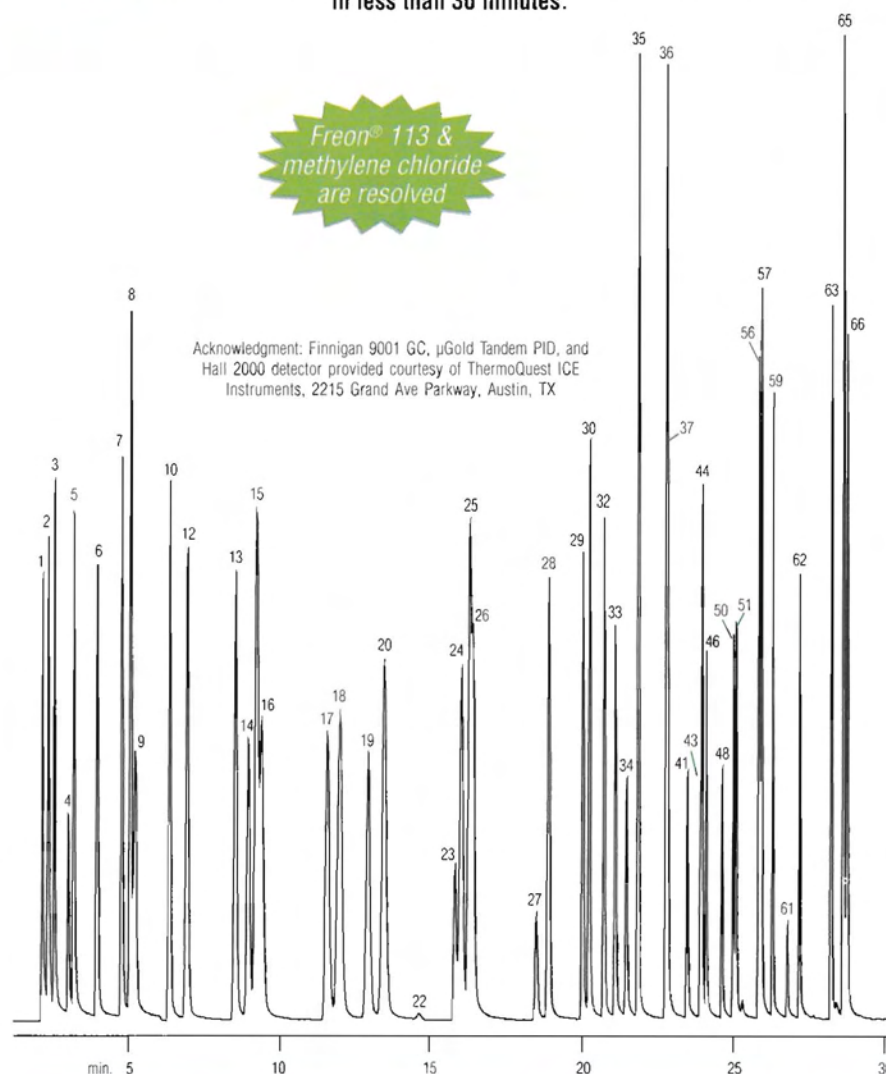
Traditionally, stationary phases for gas chromatography (GC) were developed without consideration for their final application. This resulted in long analysis times, high bleed, and coelutions. Restek has designed the new Rtx®-VRX column with low bleed, excellent selectivity, and an overall faster analysis time than the conventional 502.2, VOCOL™, and other GC phases. The Rtx®-VRX stationary phase and column dimensions have been optimized to provide excellent resolution and fast analysis times for volatile compound analyses such as US Environmental Protection Agency (EPA) Methods 8021 and 502.2. Figure 1 illustrates the resolution of closely eluting analytes and the 30-minute analysis time, permitting high sample throughput and increased laboratory efficiency.

- ✓ Excellent selectivity for volatile compound analysis.
- ✓ Equivalent performance to J&W DB™-VRX column.
- ✓ Excellent column choice for US EPA Methods 8021 and 502.2.

**Figure 1** The Rtx®-VRX provides excellent separation of 66 volatile compounds in less than 30 minutes.

20ppb in 5mL of RO Water.  
75m, 0.45mm ID, 2.55µm Rtx®-VRX (cat.# 19309)  
Concentrator: Tekmar LSC-3000 Purge and Trap  
Trap: Vocab 3000; Purge: 11 min. @ 40 mL/min.  
Dry Purge: 1 min. @ 40mL/min. (MCS by-passed with Silcosteel tubing (cat.# 21035))  
Desorb Preheat: 245°C; Desorb: 250°C for 2 min.  
Bake: 260°C for 8 min.  
Interface: direct  
Transfer Line: 0.32mm ID Siltek™ tubing  
GC: Finnigan 9001  
GC Program:  
35°C (hold 12 min.) to 60°C @ 5°C/min. (hold 1 min.) to 220°C @ 17°C/min. (hold 3 min.)  
Carrier: helium 9mL/min.  
Detectors: µGold Tandem PID/HALL  
PID: makeup 7mL/min., purge 7mL/min, set @ 0.35mV base temp 200°C.  
Hall2000: RxnGas 25mL/min., RxnTemp.: 940°C, propanol flow 470 µL/min.

- |                                       |                                 |
|---------------------------------------|---------------------------------|
| 1. dichlorodifluoromethane            | 34. 1,2-dibromoethane           |
| 2. chloromethane                      | 35. tetrachloroethene           |
| 3. vinyl chloride                     | 36. 1,1,1,2-tetrachloroethane   |
| 4. bromomethane                       | 37. chlorobenzene               |
| 5. chloroethane                       | 38. ethyl benzene               |
| 6. trichlorofluoromethane             | 39. <i>m</i> -xylene            |
| 7. 1,1-dichloroethene                 | 40. <i>p</i> -xylene            |
| 8. methylene chloride                 | 41. bromoform                   |
| 9. Freon®113                          | 42. styrene                     |
| 10. <i>trans</i> -1,2-dichloroethene  | 43. 1,4-dichlorobutane          |
| 11. methyl- <i>tert</i> -butyl-ether  | 44. 1,1,2,2-tetrachloroethane   |
| 12. 1,1-dichloroethane                | 45. <i>o</i> -xylene            |
| 13. <i>cis</i> -1,2-dichloroethene    | 46. 1,2,3-trichloropropane      |
| 14. bromochloromethane                | 47. isopropyl benzene           |
| 15. chloroform                        | 48. bromobenzene                |
| 16. 2,2-dichloropropane               | 49. <i>n</i> -propyl benzene    |
| 17. 1,2-dichloroethane                | 50. 2-chlorotoluene             |
| 18. 1,1,1-trichloroethane             | 51. 4-chlorotoluene             |
| 19. 1,1-dichloropropene               | 52. 1,3,5-trimethylbenzene      |
| 20. carbon tetrachloride              | 53. <i>tert</i> -butylbenzene   |
| 21. benzene                           | 54. 1,2,4-trimethylbenzene      |
| 22. fluorobenzene                     | 55. <i>sec</i> -butylbenzene    |
| 23. dibromomethane                    | 56. 1,3-dichlorobenzene         |
| 24. 1,2-dichloropropane               | 57. 1,4-dichlorobenzene         |
| 25. trichloroethene                   | 58. <i>p</i> -isopropyl toluene |
| 26. bromodichloromethane              | 59. 1,2-dichlorobenzene         |
| 27. 2-chloroethylvinylether           | 60. <i>n</i> -butylbenzene      |
| 28. <i>cis</i> -1,3-dichloropropene   | 61. 1,2-bromo-3-chloropropane   |
| 29. <i>trans</i> -1,3-dichloropropene | 62. 4-bromo-1-chlorobenzene     |
| 30. 1,1,2-trichloroethane             | 63. 1,2,4-trichlorobenzene      |
| 31. toluene                           | 64. naphthalene                 |
| 32. 1,3-dichloropropane               | 65. hexachlorobutadiene         |
| 33. dibromochloromethane              | 66. 1,2,3-trichlorobenzene      |



Acknowledgment: Finnigan 9001 GC, µGold Tandem PID, and Hall 2000 detector provided courtesy of ThermoQuest ICE Instruments, 2215 Grand Ave Parkway, Austin, TX

ID	df (µm)	Temp. Limits	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	-40 to 240/260°C	19315	19316	—	—
0.32mm	1.80	-40 to 240/260°C	19319	19320	—	—
0.45mm	2.55	-40 to 240/260°C	19308	—	19309	—
0.53mm	3.00	-40 to 240/260°C	19385	19388	19374	19389
ID	df (µm)	Temp. Limits	20-Meter		40-Meter	
0.18mm	1.00	-40 to 240/260°C	49314		49315	



# Rtx®-5SilMS

## The New Standard in Semivolatile GC/MS Columns

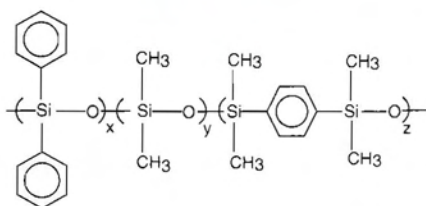
by Frank Dorman, Ph.D., Senior R&D Chemist

- ✓ Superior inertness provides more accurate quantitation at lower concentrations.
- ✓ Optimized dimensions improve resolution and provide more linear calibration curves.
- ✓ Lower bleed than phenyl/methyl phases.

Gas chromatography/mass spectrometry (GC/MS) analysis of semivolatile organic compound extracts from solid waste, soils, air, and water samples is extremely challenging. The target list contains acidic, basic, and neutral compounds ranging in volatility from fluorophenol to high-boiling polycyclic aromatic hydrocarbons (PAHs), all within a single analysis. In addition, the concentrations of both target and non-target contaminants can range from ng/μL to μg/μL levels. Because semivolatile methods monitor such a broad range of compound classes and detection limits, significant demands are placed on the inertness, efficiency, bleed, and ruggedness of the analytical column.

When choosing a GC/MS capillary column for semivolatile analyses, each column parameter must be optimized to provide the required resolution, fast analysis times, and low GC/MS bleed. The column also must have adequate sample capacity to accurately quantitate target analytes from ng to μg levels. Restek has designed the Rtx®-5Sil MS column to address these. The 30m, 0.28mmID, 0.50μm Rtx®-5Sil MS column features optimized column dimensions and stationary phase composition

**Figure 1**  
The Rtx®-5Sil MS phase structure provides ultra-low bleed.



(Figure 1), providing inherently low bleed and exceptional inertness for GC/MS semivolatile analysis.

The Rtx®-5Sil MS stationary phase formulation is based on a silarylene polymer that provides optimal separation of the semivolatile pollutants and exhibits lower GC/MS column bleed than traditional phenyl/methyl phases. By using a 0.28mm ID, the sample capacity of the column is increased to improve method detection and linearity (but column flow rate does not exceed the pumping capacity limits of a

bench-top mass spectrometer). The 0.50μm film thickness provides the necessary capacity for target analytes, while minimizing column bleed and analysis times (Figure 2).

If your lab is involved in GC/MS semivolatile analysis, Restek's new Rtx®-5Sil MS column can improve the consistency and reliability of your analytical data. Every column is tested to ensure excellent inertness towards semivolatile pollutants and provide low GC/MS bleed.



**formoreinfo**

Request the Rtx®-5SilMS Capillary Columns flyer  
(lit. cat.# 59204).

### Rtx®-5Sil MS Columns

ID	df (μm)	Temp. Limits	15-Meter	30-Meter
0.25mm	0.10	-60 to 330/350°C	12705	12708
	0.25	-60 to 330/350°C	12720	12723
	0.50	-60 to 330/350°C	12735	12738
	1.00	-60 to 325/350°C	12750	12753
0.28mm	0.25	-60 to 330/350°C	12790	12793
	0.50	-60 to 330/350°C	12791	12794
	1.00	-60 to 325/350°C	12792	12795
0.32mm	0.10	-60 to 330/350°C	12706	12709
	0.25	-60 to 330/350°C	12721	12724
	0.50	-60 to 330/350°C	12736	12739
	1.00	-60 to 325/350°C	12751	12754
0.53mm	0.50	-60 to 320/340°C	12737	12740
	1.00	-60 to 320/340°C	12752	12755
	1.50	-60 to 310/330°C	12767	12770

### Integra-Guard™ Columns

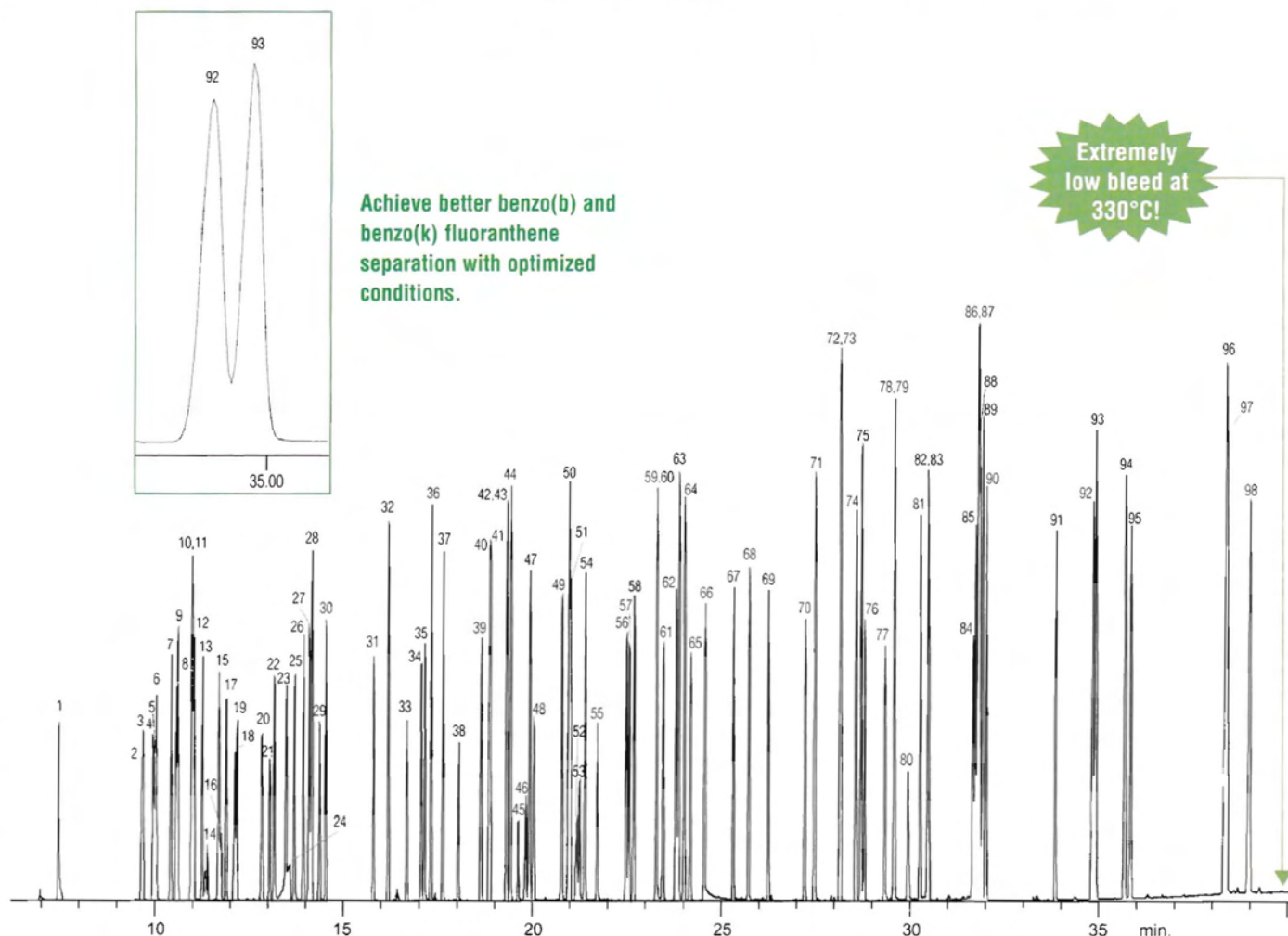
ID	Length	Suffix #	price
0.25mm	5m	-124	
	10m	-127	
0.28mm	5m	-243	
	10m	-244	
0.32mm	5m	-125	
	10m	-128	
0.53mm	5m	-126	
	10m	-129	

Ordering is simple. Just add the appropriate suffix number and price to the analytical column's catalog number and price.



Figure 2

Optimized Rtx®-5Sil MS column provides excellent resolution of EPA Method 8270 semivolatiles and improved resolution of benzo(b) and benzo(k) fluoranthene.



1. 2-fluorophenol
2. phenol-d6
3. phenol
4. bis(2-chloroethyl)ether
5. 2-chlorophenol-d4
6. 2-chlorophenol
7. 1,3-dichlorobenzene
8. 1,4-dichlorobenzene
9. 1,2-dichlorobenzene
10. 1,4-dichlorobenzene-d4 (ISTD)
11. 1,2-dichlorobenzene-d4
12. benzyl alcohol
13. 2-methylphenol (*o*-cresol)
14. 2,2'-oxybis-(1-chloropropane)
15. *N*-nitrosodi-*n*-propylamine
16. 4-methylphenol (*p*-cresol)
17. hexachloroethane
18. nitrobenzene-d5
19. nitrobenzene
20. isophorone
21. 2-nitrophenol
22. 2,4-dimethylphenol
23. bis(2-chloroethoxy)methane
24. 2,4-dichlorophenol
25. benzoic acid
26. 1,2,4-trichlorobenzene
27. naphthalene-d8 (ISTD)
28. naphthalene

29. 4-chloroaniline
30. hexachlorobutadiene
31. 4-chloro-3-methylphenol
32. 2-methylnaphthalene
33. hexachlorocyclopentadiene
34. 2,4,6-trichlorophenol
35. 2,4,5-trichlorophenol
36. 2-fluorobiphenyl
37. 2-chloronaphthalene
38. 2-nitroaniline
39. dimethyl phthalate
40. 2,6-dinitrotoluene
41. acenaphthylene
42. 3-nitroaniline
43. acenaphthene-d10 (ISTD)
44. 2,4-dinitrophenol
45. acenaphthene
46. 4-nitrophenol
47. 2,4-dinitrotoluene
48. dibenzofuran
49. diethyl phthalate
50. 4-nitroaniline
51. fluorene
52. 4-chlorophenyl phenyl ether
53. 4,6-dinitro-2-methylphenol
54. *N*-nitrosodiphenylamine
55. 2,4,6-tribromophenol
56. 4-bromophenyl phenyl ether

57.  $\alpha$ -HCH
58. hexachlorobenzene
59.  $\beta$ -HCH
60.  $\gamma$ -HCH (lindane)
61. pentachlorophenol
62. phenanthrene-d10 (ISTD)
63. phenanthrene
64. anthracene
65.  $\delta$ -HCH
66. carbazole
67. heptachlor
68. di-*n*-butyl phthalate
69. aldrin
70. heptachlor epoxide
71. fluoranthene
72. pyrene
73. endosulfan I
74. 4,4'-DDE
75. *p*-terphenyl-d14
76. dieldrin
77. endrin
78. endosulfan II
79. 4,4'-DDD
80. endrin aldehyde
81. butyl benzyl phthalate
82. 4,4'-DDT
83. endosulfan sulfate
84. endrin ketone

30m, 0.28mm ID, 0.5 $\mu$ m Rtx®-5Sil MS (cat.# 12794)  
4mm single gooseneck liner with fused silica wool  
(cat.# 22405)

Oven temp.: 35°C (hold 1 min.) to 330°C @  
10°C/min. (hold 15 min.)

Splitless hold time: 1min.

Inj. temp.: 250°C

Det. temp.: 310°C

Det.: HP 5971A MS, full scan

Carrier gas: helium @1.0 mL/min.

85. 3,3'-dichlorobenzidine
86. methoxychlor
87. benzo(a)anthracene
88. chrysene-d12 (ISTD)
89. chrysene
90. bis(2-ethylhexyl)phthalate
91. di-*n*-octyl phthalate
92. benzo(b)fluoranthene
93. benzo(k)fluoranthene
94. benzo(a)pyrene
95. perylene-d12 (ISTD)
96. dibenzo(ah)anthracene
97. ideno(1,2,3-cd)pyrene
98. benzo(ghi)perylene



# Allure™ PFP Propyl

## Improved LC/MS Analyses of Basic Compounds

by Vernon Bartlett, HPLC R&D Chemist

- ✓ Increased LC/MS signal.
- ✓ Better analyte retention.
- ✓ Improved peak shape.

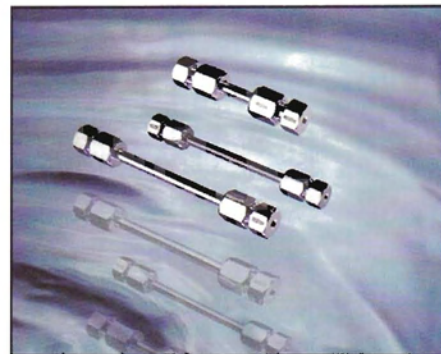
High performance liquid chromatography (HPLC) methods must be optimized to provide the greatest selectivity and sensitivity, and the best peak shape. Unfortunately, many analysts consider switching the stationary phase—the heart of the HPLC system—only as a last resort. Too often, analysts coax the stationary phase to perform a non-native separation by using modifiers and ion pairing agents (the C18 phase is the one most often misused). Use of modifiers and ion pairing agents lead to reduced sensitivity and equilibration problems. Selection of the proper stationary phase for your separation can improve LC/mass spectrometry (MS) sensitivity, analyte retention, and peak shape without the use of modifiers or ion pairing agents. For example, Restek's Allure™ pentafluorophenyl propyl (PFP Propyl) HPLC column easily performs separation of many basic analytes.

Basic analytes are difficult to retain on C18 phases if the analytes have a  $pK_a$  greater than 8. They can be retained on C8 or C18 columns by using modifiers, but at the expense of sensitivity.<sup>1</sup> Sensitivity can be further reduced on LC/MS ESI interfaces when the buffer concentrations exceed 50mM. The Allure™ PFP Propyl column eliminates the need for

modifiers, and analytes such as cocaine (COC) and ecgonine methyl ester (EME) can be separated and retained using 90% acetonitrile in under 4.5 minutes (Figure 1). As the concentration of the organic solvent in the mobile phase increases, the desolvation process becomes more effective and the LC/MS ESI signal increases.<sup>2</sup> Because of this interaction, using the Allure PFP Propyl column with a high organic concentration increases the response of COC twelve times over that from a C18 column.<sup>3</sup>

Proper retention also is needed to separate basic analytes from naturally occurring substances in the blood, urine, or other bodily tissues. If the analytes elute too closely to the void volume, ionization suppression can occur.<sup>4</sup> To be cost effective, however, the analytes should be separated in less than 6 minutes. The high selectivity of the Allure™ PFP Propyl column more than adequately separates EME from COC.<sup>5</sup>

From the research conducted, the Allure™ PFP Propyl stationary phase not only provides the greatest retention for basic analytes such as beta blockers and tricyclic antidepressants, but also the best peak shape. "...The results indicate that both the fluorine groups and the propyl chain are important on the phenyl ring to obtain the best peak shape and retention of the basic solutes when ammonium formate:acetonitrile (10:90) is used as the mobile phase."<sup>6</sup> As peak asymmetry approaches unity, sensitivity is increased.

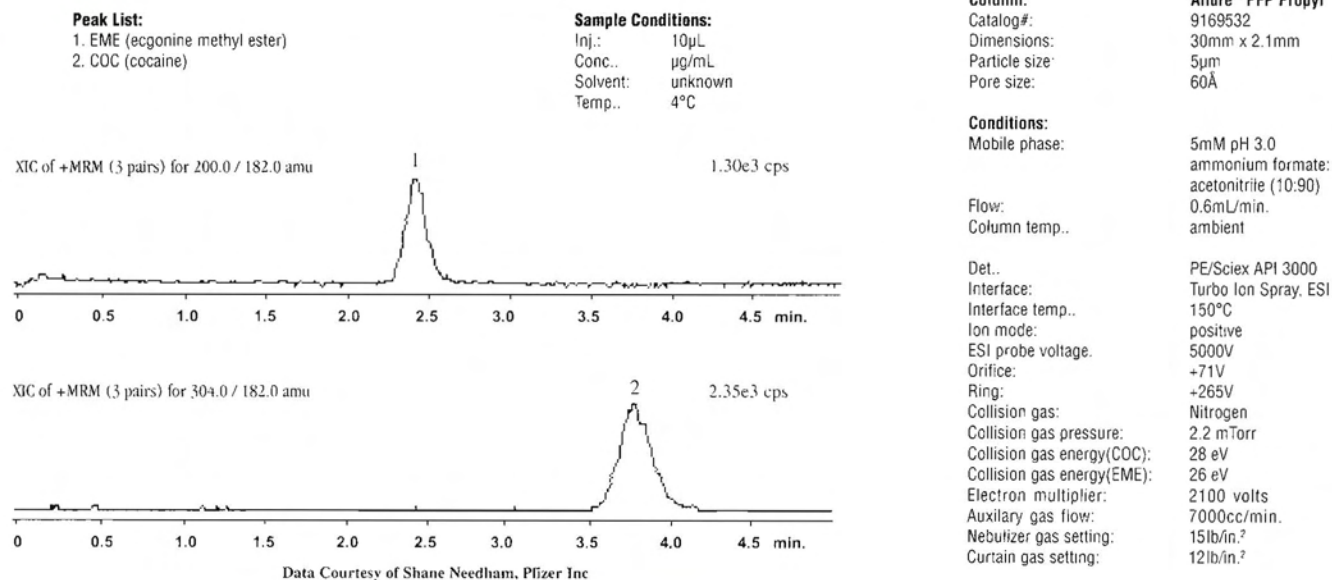


Restek's Allure™ PFP Propyl column is the best choice for analysis of basic analytes. COC, ERP, quinine, morphine, beta blockers, and tricyclic antidepressants have been successfully analyzed in urine, blood, or other tissue samples. Analytes with a  $pK_a$  greater than 8 are retained without modifiers and with superior peak shape. The high level of organic solvent used in the mobile phase and the improved peak shape increase the analytical sensitivity.

1. J. Svensson, *J. Anal. Toxicol.* 10 (1986) 122-124.
2. P. Sjoberg and K. Markides, *J. Chromatogr. A.* 855 (1999) 317-327.
3. S. R. Needham, *op. cit.*
4. B. Matuszewski, M. Constanzer, and C. Chavez-Eng, *Anal. Chem.* 70 (1998) 882-889.
5. S. R. Needham, *op. cit.*
6. S. R. Needham, P.R. Brown, and K. Duff, *Rapid Commun. Mass Spectrom.* 13, 2231-2236 (1999).

References not available from Restek.

**Figure 1** The Allure™ PFP Propyl baseline separates Cocaine Ester (COC) and its hydroxyl precursor, Ecgonine Methyl Ester (EME), with excellent peak shape in less than five minutes.





**Allure™ PFP Propyl, 5µm Columns**

Particle Size:	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
5µm	cat.#	cat.#	cat.#	cat.#
30mm length	9169531	9169532	9169533	9169535
50mm length	9169551	9169552	9169553	9169555
100mm length	9169511	9169512	9169513	9169515
150mm length	9169561	9169562	9169563	9169565
200mm length	9169521	9169522	9169523	9169525
250mm length	9169571	9169572	9169573	9169575

**Allure™ PFP Propyl, 5µm Columns with Trident™ Inlet**

Particle Size:	2.1mm ID	3.2mm ID	4.6mm ID
5µm	cat.#	cat.#	cat.#
30mm length	9169532-700	9169533-700	9169535-700
50mm length	9169552-700	9169553-700	9169555-700
100mm length	9169512-700	9169513-700	9169515-700
150mm length	9169562-700	9169563-700	9169565-700
200mm length	9169522-700	9169523-700	9169525-700
250mm length	9169572-700	9169573-700	9169575-700

**formoreinfo**

Request the Applications Note *Allure™ PFP Propyl HPLC Column Provides Improved LC/MS Analyses of Basic Compounds* (lit. cat.# 59118).

**Free HPLC wall chart and LC/MS Catalog**

Request a free wall chart (lit. cat.# 59894) and catalog (lit. cat.# 59607, international lit. cat.# 59606).

# Reference Materials

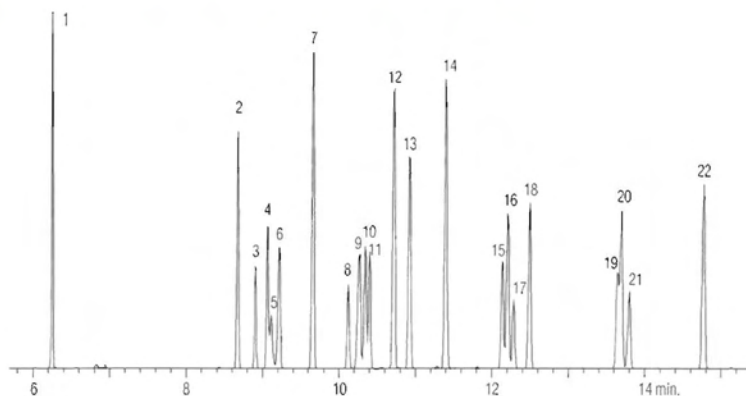
## Minnesota Dept. of Agriculture Adds Dimethenamid to Incident Investigations

by Eric Steindl, Analytical Reference Materials Product Line Manager

Effective January 2000, Minnesota Department of Agriculture has added a new compound to their List 1 pesticide (neutrals) incident investigation requirements.<sup>1</sup> This compound, dimethenamid (CAS # 87674-68-8), has been manufactured by several companies under various trade names. Active product registrations exist for American Cyanamid, BASF, and E.I. Dupont De Nemours. Inactive product registrations exist for Sandoz Agro Inc.

Restek has arranged to purchase dimethenamid from one of the primary manufacturers with the intent to provide calibration mixtures required in this analysis. As with all Restek quantitative environmental mixtures, a complete data pack is available to meet stringent audit requirements.

1. "Analytical Lists for Pesticide Incident Investigations", Minnesota Department of Agriculture, Guidance Document 26 (3/99). St. Paul, MN. For a copy of this and other Minnesota Department of Agriculture documents, visit their web site at: [www.mda.state.mn.us](http://www.mda.state.mn.us)



30m, 0.28mmID, 0.5µm Rtx®-5SiIMS (cat. # 12794)

Oven temp.: 35°C (hold .5 min.) to 160°C @ 50°C/min. to 190°C @ 10°C/min. to 300°C @ 5°C/min.

1µL splitless 0.5 min., 10ppm calibration standard in methylene chloride/acetone, 10ng on column concentration

GC: HP 6890 GC; Inj. temp: 250°C; Inj: 1µL splitless 0.5 min., 10ppm calibration standard in methylene chloride/acetone, 10ng on column concentration; Det: HP 5973 MS, scan 35 - 550 amu

**Minnesota Ag List 1 Pesticides Mix A**

200ppm each in Acetone

1mL per ampul

Mix contains compounds in peak list marked \*

Each	5-pk.	10-pk.
32406	32406-510	—
w/ data pack		
32406-500	32406-520	32506

**Minnesota Ag List 1 Pesticides Mix B**

200ppm each in Acetone

1mL per ampul

Mix contains compounds in peak list marked †

Each	5-pk.	10-pk.
32407	32407-510	—
w/ data pack		
32407-500	32407-520	32507

- |                                |   |
|--------------------------------|---|
| 1. EPTC†<br>(eptam, eradicane) | 12. terbufos†                             |
| 2. propachlor*                 | 13. fonofos†                              |
| 3. ethalfluralin*              | 14. triallate†                            |
| 4. trifluralin*                | 15. dimethenamid*                         |
| 5. desisopropylatrazine*       | 16. acetochlor*                           |
| 6. desethylatrazine*           | 17. metribuzin*                           |
| 7. phorate†                    | 18. alachlor*                             |
| 8. prometon*                   | 19. metolachlor*                          |
| 9. simazine*                   | 20. chlorpyrifos†<br>(chlorpyrifos ethyl) |
| 10. atrazine*                  | 21. cyanazine*                            |
| 11. propazine*                 | 22. pendimethalin*                        |

**Minnesota Ag List 1 Pesticides Kit**

Contains Minnesota Ag List Pesticides Mix A & B: 32408, (ea.) 32408-500, (ea. w/data pack)



# Reference Materials

## US EPA Method 8095 GC Explosives Analysis

by Christopher Cox, Senior R&D Chemist



The standard environmental test method for nitroaromatic, nitramine, and nitroester analyses is US Environmental Protection Agency (EPA) method 8330<sup>1</sup>. This method uses high performance liquid chromatography

(HPLC) separation and detection by ultra-violet absorption. Unfortunately, this method typically results in high solvent usage, multiple coelutions, and long run times. Analysts have been interested in developing a gas chromatography (GC) method that overcomes the disadvantages of HPLC method.

US EPA Method 8095<sup>2</sup> has been proposed as a GC/electron capture detection (ECD) alternative to the HPLC explosives analysis in Method 8330. This proposed GC/ECD method was developed at the US Army Cold Regions Research and Engineering Laboratory<sup>3</sup>. It includes all of the Method 8330 target compounds plus 3,5-dinitroaniline, nitroglycerine, and pentaerythritol tetranitrate (PETN).

The calibration mixtures listed at right contain the additional compounds at the concentration ratios appropriate for ECD. Restek also will be introducing a set of capillary columns—the Rtx®-TNT and Rtx®-TNT2 columns—designed specifically for this method using two new proprietary stationary phases. These 6-meter columns will provide better resolution than the columns recommended in Method 8095. They will use identical temperature programs, allowing simultaneous, dual-column confirmation analysis. Look for future mailings with the details of these columns.

1. U. S. Environmental Protection Agency. *Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. SW-846 Update III*. Office of Solid Waste, Washington, DC, 1997.

2. U. S. Environmental Protection Agency. *Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. SW-846. Proposed Draft Update IVB*. Office of Solid Waste, Washington, DC, 1999.

3. M. E. Walsh, T. Ranney, "Determination of Nitroaromatic, Nitramine, and Nitrate Ester Explosives in Water Using Solid-Phase Extraction and Gas Chromatography-Electron Capture Detection: Comparison with High-Performance Liquid Chromatography," *Journal of Chromatographic Science*, vol. 36, p. 406-416, August 1998.

References not available from Restek.

### 8095 Calibration Mix A

	ppm
2-amino-4,6-dinitrotoluene	1000
4-amino-2,6-dinitrotoluene	1000
1,3-dinitrobenzene	1000
2,4-dinitrotoluene	1000
2,6-dinitrotoluene	1000
HMX	1000
RDX	1000
Tetryl	1000
1,3,5-trinitrobenzene	1000
2,4,6-trinitrotoluene	1000

1000µg/mL ea. in acetonitrile, 1mL/ampul

Each	5-pk.	10-pk.
31607	31607-510	-----
w/ data pack		
31607-500	31607-520	31707

### 8095 Calibration Mix B

	ppm
3,5-dinitroaniline	1000
nitrobenzene	5000
nitroglycerine	5000
2-nitrotoluene	5000
3-nitrotoluene	5000
4-nitrotoluene	5000
PETN	5000

Prepared in acetonitrile, 1mL/ampul

Each	5-pk.	10-pk.
31608	31608-510	-----
w/ data pack		
31608-500	31608-520	31708

### Matrix Spike Solutions

#### 8095 Matrix Spike Mix A

	ppm
2-amino-4,6-dinitrotoluene	200
4-amino-2,6-dinitrotoluene	200
1,3-dinitrobenzene	200
2,4-dinitrotoluene	200
2,6-dinitrotoluene	200
HMX	2000
RDX	200
Tetryl	200
1,3,5-trinitrobenzene	200
2,4,6-trinitrotoluene	200

Prepared in acetonitrile, 1mL/ampul

Each	5-pk.	10-pk.
31609	31609-510	-----
w/ data pack		
31609-500	31609-520	31709

### 8095 Matrix Spike Mix B

	ppm
3,5-dinitroaniline	200
nitrobenzene	1000
nitroglycerine	1000
2-nitrotoluene	1000
3-nitrotoluene	1000
4-nitrotoluene	1000
PETN	1000

Prepared in acetonitrile, 1mL/ampul

Each	5-pk.	10-pk.
31610	31610-510	-----
w/ data pack		
31610-500	31610-520	31710

### 8095 Surrogates

#### 2,5-dinitrotoluene

1000µg/mL in methanol, 1mL/ampul

Each	5-pk.	10-pk.
31611	31611-510	-----
w/ data pack		
31611-500	31611-520	31711

#### 3,4-dinitrotoluene

1000µg/mL in methanol, 1mL/ampul

Each	5-pk.	10-pk.
31452	31452-510	-----
w/ data pack		
31452-500	31452-520	31552

#### 2-methyl-4-nitroaniline

1000µg/mL in methanol, 1mL/ampul

Each	5-pk.	10-pk.
31612	31612-510	-----
w/ data pack		
31612-500	31612-520	31712

Restek's Analytical Reference Materials team:  
(left to right) Joe Moodler, Rick Parmely,  
Mary Ellen Wood, John Lidgett, Chris Cox, Diane  
Shaffer, Eric Steindl, Andrea Gill, Jason Martin,  
Scott Strohm, (sitting at table) Denise Witherite  
and Joe Tallon.





# Rtx®-CLPesticides

## Analysis of Aroclor® Mixtures Using Specialized Columns

by Lydia Nolan, Environmental Applications Chemist

Polychlorinated biphenyls (PCBs) are a group of industrial organochlorine chemicals that have become a major environmental concern. Since the 1950's, over one-million metric tons of PCBs have been produced. They were used commercially because they are chemically inert liquids and are difficult to burn; they have low vapor pressures, are inexpensive to make, and are excellent electrical insulators. As a result, they were used extensively as coolant fluids in transformers and capacitors; and later as plasticizers, de-inking solvents, heat transfer fluids in machinery, and water-proofing agents, among other uses.

Because of their persistence and their solubility in fatty tissue, PCBs in food chains undergo biomagnification. Strong heating of PCBs in the presence of oxygen can lead to the formation of polychloro dibenzofurans (PCDF), which are structurally and toxicologically similar to dioxins. Toxicity also depends on where the chlorine substitution resides on the biphenyl molecule; the congeners without chlorine substitution on the ortho positions are the most toxic. These coplanar PCBs are "dioxin-like," and the most toxic is one-tenth the toxicity of 2,3,7,8-tetrachloro dibenzo dioxin, the most toxic compound known. The coplanar PCBs also have been listed as endocrine disrupters.<sup>1</sup>

It is important, therefore, when designing a PCB analysis method to determine if the separation will be by specific congener (for toxicity) or by Aroclor® mixture (commercial mixture). The commercial synthesis of PCBs result in chlorination of the biphenyl molecule, and this reaction produces a mixture of many of the 209 congeners of the PCB family.

Due to the unreactive nature of the PCBs, instrument conditions and column choice is less critical than when analyzing chlorinated pesticides. The important points are to choose an inlet technique that can handle reasonable levels of contamination and injection volume. When choosing columns, it is important to select stationary phases that have low bleed and high thermal stability; allowing the columns to be baked out at the end of the run to prevent carryover. Because many instruments used for the analysis of PCBs also may be used for pesticide and herbicide analyses, the column pair of choice is the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns. This column pair provides excellent separation of pesticide and herbicide compounds, low bleed, high thermal stability, and are designed to compliment each other for primary column analysis and secondary column confirmation.

Figure 1 shows a dual-column analysis of Aroclor® 1242 on the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns. This configuration is used to minimize injection port maintenance issues, and uses a Siltek™ guard column and 'Y' Press-Tight® connector to connect the two analytical columns to the single injection port. To see chromatograms of all nine of the most common Aroclor® mixtures,

peak identifications, and the elution order for all 209 congeners on these columns, request Applications Note #59120.

1. Environmental Chemistry, Colin Baird, W.H. Freeman and Co., 1998, pp. 337-353.

References not available from Restek.

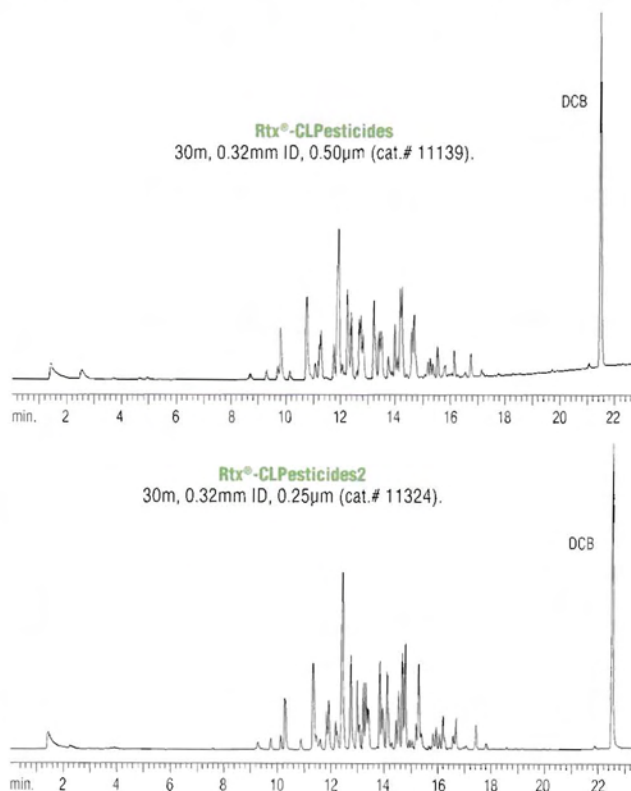
for more info

Request the Applications Note *Analysis of PCB Congeners Using Rtx®-CLPesticides Columns* (lit. cat.# 59120).

**Figure 1** Rtx®-CLPesticides columns provide excellent separation of Aroclor® 1242.

#### Dual-Column Analysis Conditions:

Inj.: 0.5µL direct injection  
Aroclor® 1242 mixture,  
0.16ng concentration  
Oven temp.: 120°C (hold 1 min.)  
to 300°C @ 9°C/min.  
(hold 10 min.)  
Inj. temp.: 210°C; Det.: ECD with  
anode purge, 310°C  
Carrier gas: helium



#### Rtx®-CLPesticides Kits

ID (mm)	cat.#
0.25	11199
0.32	11198
0.53	11197

#### Each kit includes:

- 1-Rtx®-CLPesticides column
- 1-Rtx®-CLPesticides2 column
- 1-Siltek™ Angled "Y" Press-Tight® connector
- 1-Siltek™ guard column



# Peak Performers

## Focal Inlet Liners

- ✓ Wool is placed at the injection point to maximize vaporization and help wipe the needle during injection.
- ✓ Designed for easy changing of the wool.
- ✓ No more guessing where the wool should be placed.
- ✓ Wool stays in position during pressure pulses in the inlet and during injection.
- ✓ 100% polymeric deactivation.





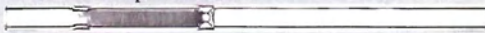



## Low Pressure Drop Liner

for HP 6890  
Split/Splitless Injection Port

- ✓ OD slightly smaller than standard splitless liners for increased flow during injection.
- ✓ Dual taper design for reproducible positioning of wool.
- ✓ Wool positioned to allow wiping of the syringe on injection.
- ✓ Glass bead at end of liner to allow easier installation.



cat # 21032, (ea.)  
cat# 21033, (5-pk.)

Instrument	each	5-pk.
HP 4mm Split Focal Liner 	21022	21023
Varian 1078/1079 Split Focal Liner 	21024	21025
Shimadzu 17A Split Focal Liner 	21020	21021
Varian 1075/1077 Split Focal Liner 	21030	21031
Fisons 5mm Split Focal Liner 	21028	21029
PE Auto SYS Split Focal Liner 	21026	21027

# ON-THE-ROAD

## Environmental GC Seminar

Restek is "On-the-Road" again! A new tour is being added to our popular comprehensive GC tour—"Chromatography Training for the Environmental Industry." This seminar is scheduled to begin in the spring of 2000 and is cost-effective.

### Who Should Attend?

The environmental seminar will teach basic and advanced concepts on environmental analyses. Whether you are a novice, an expert, or a manager, this seminar will prove useful. The content is technical in nature and provides solutions for a variety of problems related to environmental analyses. Those who attend should be familiar with the basics of gas chromatography (GC). This one-day course covers semi-volatile and volatile organics, pesticides and PCB's, and air analysis. Materials, refreshments, and lunch are provided.

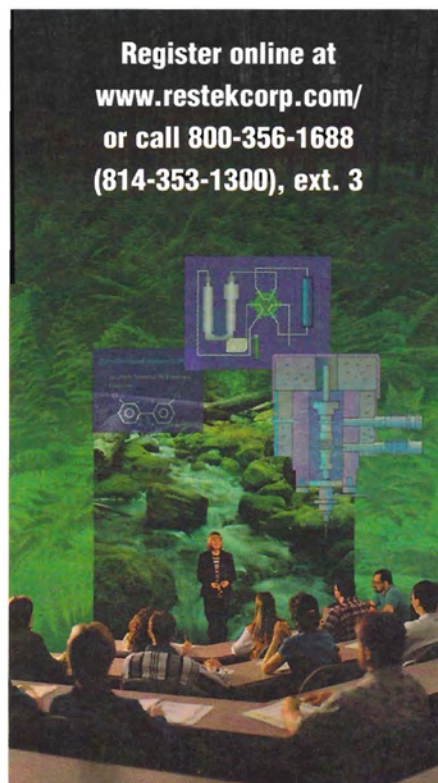
**for more info**

Request the *Environmental GC Seminar* brochure for an agenda and a registration form.  
(lit. cat.# 59210).

Location	Date	Cat.#
San Jose, CA	March 30	65400
Seattle, WA	March 31	65401
Tampa, FL	April 3	65402
Jacksonville, FL	April 4	65403
Atlanta, GA	April 5	65404
Durham, NC	April 10	65405
Baltimore, MD	April 12	65406
Philadelphia, PA	April 13	65407
Somerset, NJ	April 14	65408

### Can't attend? Here are some recommended reference materials:

- ✓ *Guide to Environmental Analytical Methods, 4th Edition*, cat.# 20465
- ✓ *Solid Phase Extraction, Principles and Practice* (E.M. Thurman & M.S. Mills) cat.# 20494
- ✓ *GC/MS: A Practical User's Guide* (M. McMaster & C. McMaster), cat.# 20496
- ✓ *A Technical Guide for Static Headspace Using GC*, cat.#59895
- ✓ *Helpful Hints for Analyzing Volatile Organics*, cat.# 59887

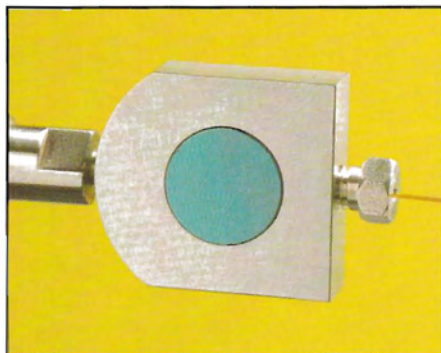


Register online at  
[www.restekcorp.com/](http://www.restekcorp.com/)  
or call 800-356-1688  
(814-353-1300), ext. 3



# EZ-Vent™ 2000

## *Saving You Time and Money When Changing GC/MS Columns*



We designed a common sense, affordable solution to the hassle of changing columns in your mass spectrometer (MS). By using the Restek EZ-Vent™ 2000 interface, you can avoid the typical, lengthy vent and pump-down cycles every time you want to change a column.

The EZ-Vent™ 2000 MS interface will revolutionize the way you connect columns to your MS. When the outlet of a capillary column is inserted into an MS vacuum, the negative pressure propagates several meters inside the column thus decreasing efficiency. Using the EZ-Vent™ 2000 interface minimizes this effect, thereby increasing the effective length of the column. The EZ-Vent™ 2000 interface does not require any additional plumbing and works on a critical orifice principle, thereby eliminating the need to plumb your MS interfaces with purge gases. Plus, it's easy to install! Just connect the EZ-Vent™ 2000 transfer line and connectors and you are ready to begin using your MS without worry of pumping it down during the next column change.

Restek's EZ-Vent™ 2000 interface is available for Hewlett-Packard 5971, 5972, and 5973 MS and Varian Saturn MS systems.

### Feature

- ✓ Save time
- ✓ Cost effective
- ✓ Save money
- ✓ Very low dead volume fittings and small ID tubing.
- ✓ All metal components are Silcosteel®-coated and transfer lines are deactivated for maximum inertness.

### Benefit

- Decreased column changing time.
- Costs associated with cool-downs and pump-downs are now investments in analytical runs.
- Many times less expensive than other models with equal performance.
- Ensure no band broadening or change in analytical performance.
- Less adsorption or loss of reactive compounds = more accurate analyses.

### EZ-Vent™ 2000 for HP GCs:

Includes EZ-Vent™ 2000, 1/16" SS nut, two 0.4mm ferrules, 100µm deactivated transfer line, and EZ-Vent™ column plug; cat.# 21013, (kit)

### EZ-Vent™ 2000 for Varian GCs:

Includes, EZ-Vent™ 2000, 1/16" SS nut, two 0.4mm ferrules, 100µm deactivated transfer line, and EZ-Vent™ column plug; cat.# 21014, (kit)

### Replacement EZ-Vent™ 2000 ferrules:

0.4mm ID: cat.# 21015, (2-pk.)

0.5mm ID: cat.# 21016, (2-pk.)

### Replacement EZ-Vent™ 2000 union:

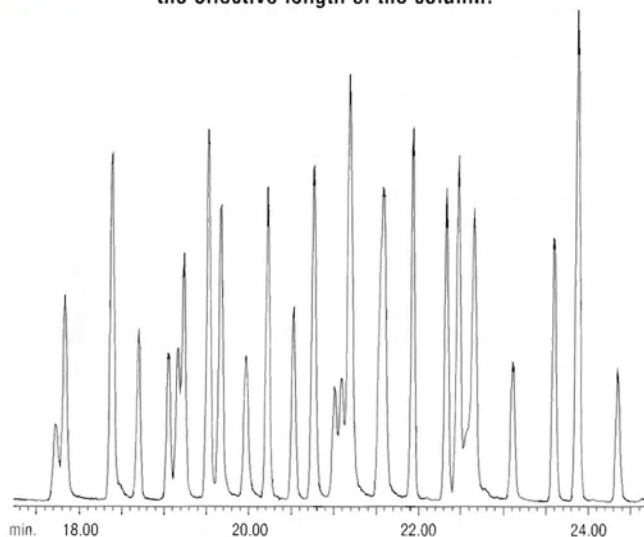
cat.# 21017, (ea.)

### Replacement EZ-Vent™ 2000 deactivated transfer line :

100µm: cat.# 21018, (3 ft.)

Open-end wrench tool (1/4" - & 5/16"-inch): cat.# 20110, (2-pk.)

**Figure 1** The EZ-Vent™ interface causes no peak broadening and increases the effective length of the column.



30m, 0.25mm ID, 0.5µm Rtx®-5MS (cat.# 12638)  
4mm single gooseneck liner with fused silica wool (cat.# 22405)  
Inj.: 50ng/µL semivolatle standard

Oven temp.: 35°C (hold 1 min.) to 300°C @ 10°C/min. (hold 20 min.); Splitless hold time: 1min.; Inj. temp.: 300°C;  
Det. temp.: 310°C; Det.: HP 5971A MS, full scan; Carrier gas: helium @ 15psi

**for more info**

Request the EZ-Vent™ 2000 Fast Fact  
(lit. cat.# 59307).

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**chromatogram  
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the

# RESTEK Advantage

Innovators of High Resolution Chromatography Products

## Rtx<sup>®</sup>-VGC Columns

### Revolutionary New Column Designed for GC Volatiles Analysis

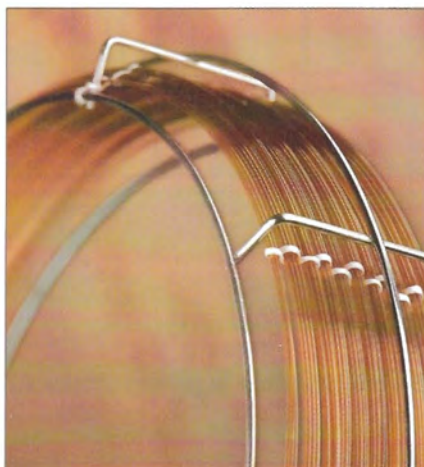
by Christopher English, Applications Chemist, and Christine Vargo, GC Columns Product Manager

- ✓ Better resolution than VRX phases.
- ✓ Designed specifically for volatiles analysis using PID/ELCD.
- ✓ Provides fast analysis times without coelutions for volatile organic compounds (VOCs).
- ✓ Increases lab productivity and sample throughput compared to traditional 502.2 and 624 columns.

Volatile organic compound (VOC) analysis employs purge and trap instrumentation to concentrate volatile contaminants in water, soil, and wastewater. While purge and trap significantly increases sensitivity relative to other sample introduction techniques, early eluting volatile compounds typically exhibit broad peak shape. This decreases the separation between closely eluting compounds, which places high demands on the analytical system. Another issue with VOC analyses is the long analysis time caused by purge and trap cycle time, GC oven cycle time, and column length.

Restek designed the Rtx<sup>®</sup>-VGC column to provide the best GC separation of VOCs such as those listed in US Environmental Protection Agency (EPA) Methods 502.2 and 8021. Also, based on customer input, we added some commonly analyzed, but unregulated compounds to our target list, such as methyl-tert-butyl ether (MTBE), tert-butyl alcohol, and Freon<sup>®</sup> 113. Figures 1a and 1b show the analysis of this expanded VOC list on the new Rtx<sup>®</sup>-VGC column. The Rtx<sup>®</sup>-VGC provides excellent separation of the purgeable gases and trihalomethanes (see Figure 2 for examples). Notice the excellent resolution of these VOCs—there are no coelutions using PID/ELCD!

Why is resolution so important? Resolution requirements are compound- and method-dependent. The more toxic and common a pollutant is in the environment, the more important accurate



deeper the valley between two peaks, the more accurate the quantitative results. The Rtx<sup>®</sup>-VGC column provides 30% resolution of 1,1,2,2-tetrachloroethane/ 2-chlorotoluene (Figure 1b, inset), and ≥60% resolution of all other volatile compounds found in US EPA Methods 502.2 and 8021, thereby increasing the quantitative reliability of your data!

If your lab is performing VOC analyses by PID/ELCD (e.g., US EPA Methods 502.2, 601, 602, 8010, 8020, or 8021), Restek's new Rtx<sup>®</sup>-VGC capillary column will provide the best resolution of any column

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1 (of 16) 2000 Summer

**HROMalytic** +61(0)3 9762 2034  
**ECHnology** Pty Ltd

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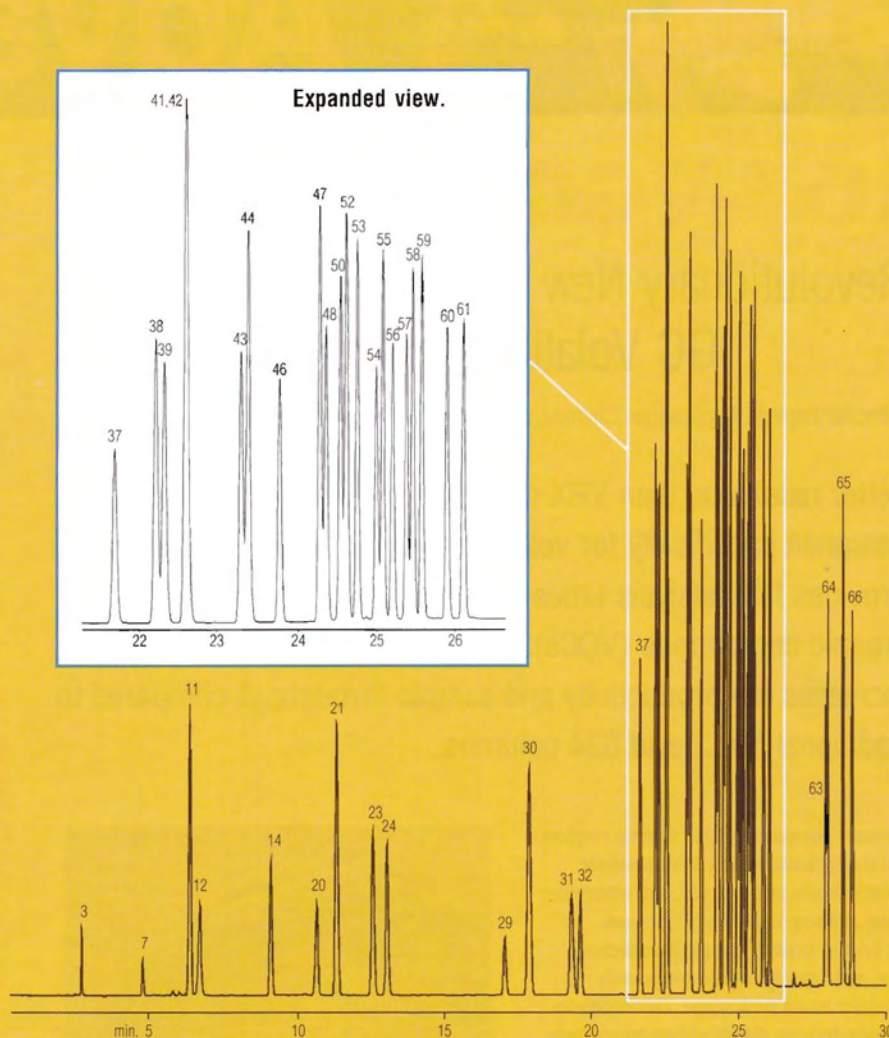
**Figure 1a**

The Rtx®-VGC column separates US EPA Method 8021 compounds — with better resolution and in less time than any other column available.

**PID**

75m, 0.45mm ID, 2.55µm Rtx®-VGC (cat.#19409).  
20ppb in 5mL of RO water. **Concentrator:** Tekmar  
LSC-3000 Purge and Trap; **Trap:** Vocab® 3000;  
**Purge:** 11 min. @ 40mL/min.; **Dry purge:** 1 min.  
@ 40mL/min. [MCS by-passed with Silcosteel®  
tubing (cat.# 21035)]; **Desorb preheat:** 245°C;  
**Desorb:** 250°C for 2 min.; **Bake:** 260°C for 8  
min.; **Interface:** direct; **Transfer line:** 0.32mm ID  
Siltek™ tubing; **GC:** Finnigan 9001; **Oven temp.:**  
35°C (hold 4 min.) to 75°C @ 3°C/min. (hold 2  
min.) to 175°C @ 21°C/min. to 205°C @ 35°C/  
min. (hold 5 min.); **Carrier gas:** helium 11mL/  
min., constant pressure; **Detectors:** µGold Tandem  
PID/HALL; **PID:** makeup 7mL/min., purge 7mL/  
min., set @ 0.35mV, base temp. 200°C; **Hall 2000:**  
RxnGas 25mL/min., RxnTemp 940°C, propanol  
flow 470µL/min.

**Acknowledgment:** Finnigan 9001 GC, µGold  
Tandem Photoionization detector and Hall 2000  
detector provided courtesy of ThermoQuest/CE  
Instruments, 2215 Grand Avenue Parkway, Austin,  
TX 78728.

**Figure 2**

The Rtx®-VGC column resolves chloroform in drinking water better than VRX columns.

Disinfectant by-products of drinking water (i.e., trihalomethanes [THMs]) are the most commonly found contaminants in the public water supply. Chloroform—one of the THMs—is poorly resolved when using traditional VRX phases. The new Rtx®-VGC column provides better resolution of chloroform as well as all other compounds in US EPA Methods 502.2 and 8081. See Figure 1b for separation of all four THMs.

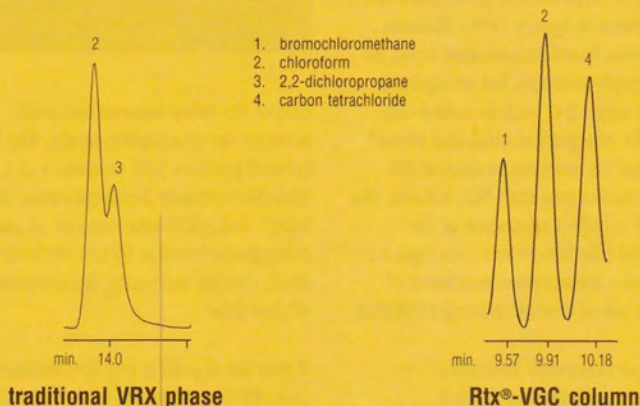
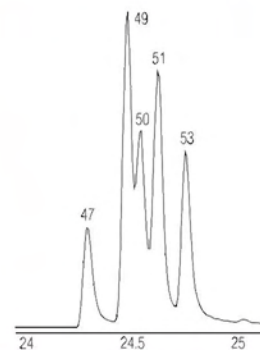




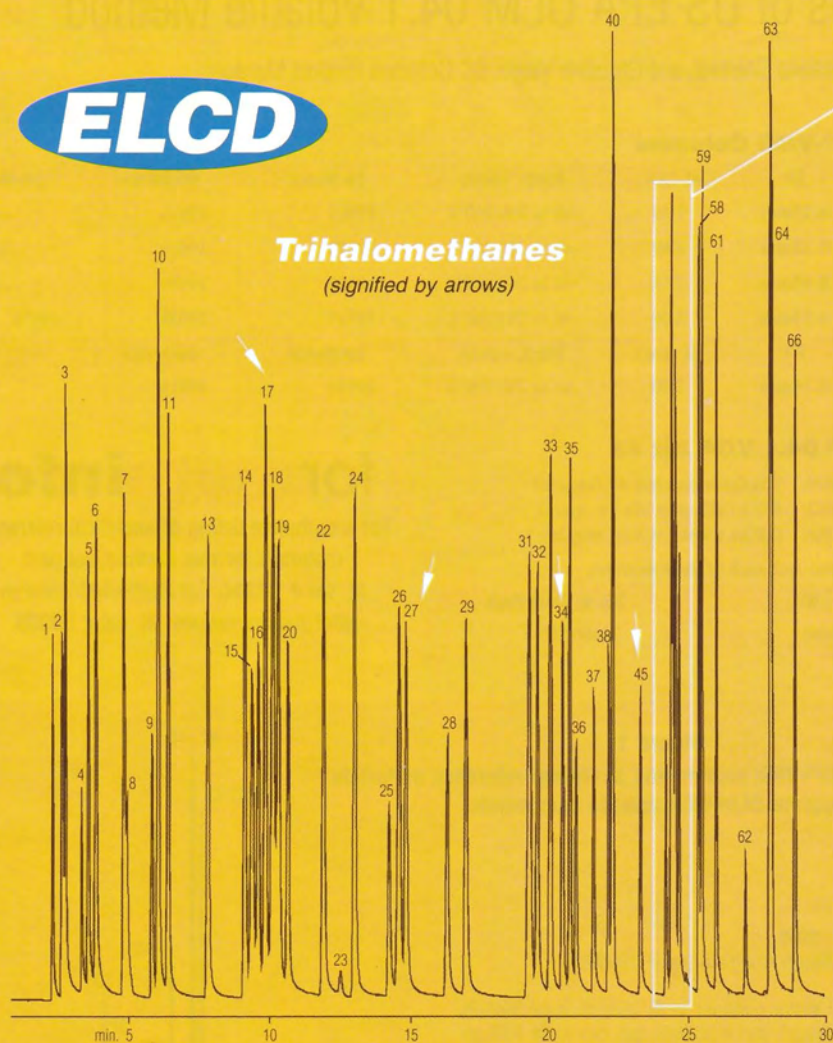
Figure 1b

The Rtx®-VGC column provides greater than 30% resolution of these difficult-to-separate compounds.



**ELCD**

### Trihalomethanes (signified by arrows)



- |                                       |                                 |
|---------------------------------------|---------------------------------|
| 1. dichlorofluoromethane              | 34. dibromochloromethane        |
| 2. chloromethane                      | 35. 1,3-dichloropropane         |
| 3. vinyl chloride                     | 36. 1,2-dibromoethane           |
| 4. bromomethane                       | 37. 1-chloro-3-fluorobenzene    |
| 5. chloroethane                       | 38. chlorobenzene               |
| 6. trichlorofluoromethane             | 39. ethylbenzene                |
| 7. 1,1-dichloroethene                 | 40. 1,1,1,2-tetrachloroethane   |
| 8. Freon® 113                         | 41. <i>m</i> -xylene            |
| 9. allyl chloride                     | 42. <i>p</i> -xylene            |
| 10. methylene chloride                | 43. <i>o</i> -xylene            |
| 11. <i>trans</i> -1,2-dichloroethene  | 44. styrene                     |
| 12. methyl- <i>tert</i> -butyl-ether  | 45. bromoform                   |
| 13. 1,1-dichloroethane                | 46. isopropylbenzene            |
| 14. <i>cis</i> -1,2-dichloroethene    | 47. bromobenzene                |
| 15. 2,2-dichloropropane               | 48. <i>n</i> -propylbenzene     |
| 16. bromochloromethane                | 49. 1,1,2,2-tetrachloroethane   |
| 17. chloroform                        | 50. 2-chlorotoluene             |
| 18. carbon tetrachloride              | 51. 1,2,3-trichloropropane      |
| 19. 1,1,1-trichloroethane             | 52. 1,3,5-trimethylbenzene      |
| 20. 1,1-dichloropropene               | 53. 4-chlorotoluene             |
| 21. benzene                           | 54. <i>tert</i> -butylbenzene   |
| 22. 1,2-dichloroethane                | 55. 1,2,4-trimethylbenzene      |
| 23. fluorobenzene                     | 56. <i>sec</i> -butylbenzene    |
| 24. trichloroethene                   | 57. <i>p</i> -isopropyltoluene  |
| 25. dibromomethane                    | 58. 1,3-dichlorobenzene         |
| 26. 1,2-dichloropropane               | 59. 1,4-dichlorobenzene         |
| 27. bromodichloromethane              | 60. <i>n</i> -butylbenzene      |
| 28. 1-bromo-2-chloroethane            | 61. 1,2-dichlorobenzene         |
| 29. <i>cis</i> -1,3-dichloropropene   | 62. 1,2-dibromo-3-chloropropane |
| 30. toluene                           | 63. hexachlorobutadiene         |
| 31. tetrachloroethene                 | 64. 1,2,4-trichlorobenzene      |
| 32. <i>trans</i> -1,3-dichloropropene | 65. naphthalene                 |
| 33. 1,1,2-trichloroethane             | 66. 1,2,3-trichlorobenzene      |

## Rtx®-VGC Columns

ID	df (µm)	Temp. Limits	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	-40 to 240/260°C	19415	19416	—	—
0.32mm	1.80	-40 to 240/260°C	19419	19420	—	—
0.45mm	2.55	-40 to 240/260°C	19408	—	19409	—
0.53mm	3.00	-40 to 240/260°C	19485	19488	19474	19489
ID	df (µm)	Temp. Limits	20-Meter	40-Meter	75-Meter	105-Meter
0.18mm	1.00	-40 to 240/260°C	49414	49415	—	—



## Analytical Reference Materials

Restek manufactures a variety of volatile calibration mixtures for environmental applications. Individual laboratory target compound lists may vary, so we offer a range of stock and custom mixtures to meet specific requirements. Please contact our technical service team at 800-356-1688 or 814-353-1300, ext. 4, or call your local Restek representative to discuss your needs.

**for more info**

For more information, request the  
**"Rtx®-VGC Columns Brochure"**  
 (lit. cat.# 59217).

Call 800-356-1688 or 814-353-1300, ext. 5,  
 or contact your local Restek representative.



# Rtx®-VMS Columns

## Fast, Accurate Analysis of US EPA OLM 04.1 Volatile Method

by Christopher English, Applications Chemist, and Christine Vargo, GC Columns Product Manager

- ✓ Higher productivity.
- ✓ Overcomes co-elutions.
- ✓ Tuned selectivity.

The US Environmental Protection Agency (EPA) has recently awarded contracts for organic low medium (OLM) concentration samples within the Superfund program under the 04.2 revision Statement of Work. Restek has designed the perfect gas chromatography (GC) capillary column and analytical reference materials to meet these requirements. This new column features tuned selectivity and faster separations than traditional volatile analysis columns. Combine the Rtx®-VMS column with the new CLP OLM 04.1 (04.2) volatiles reference materials, and you can accomplish this analysis with better results than ever before (Figure 1).

### Rtx®-VMS Columns

ID	df (µm)	Temp. Limits	30-Meter	60-Meter	75-Meter
0.25mm	1.40	-40 to 240/260°C	19915	19916	—
0.32mm	1.80	-40 to 240/260°C	19919	19920	—
0.45mm	2.55	-40 to 240/260°C	19908	19909	—
0.53mm	3.00	-40 to 240/260°C	19985	19988	19974
ID	df (µm)	Temp. Limits	20-Meter	40-Meter	
0.18mm	1.00	-40 to 240/260°C	49914	49915	

### CLP 04.1 VOA Kit #3

- 30006: VOA Calibration Mix #1 (ketones)
- 30042: 502.2 Calibration Mix #1 (gases)
- 30456: CLP 04.1 VOA CAL2000 MegaMix™

Contains 1mL each of these mixtures.

Kit	Kit w/ Data Pack
30460	30460-500

for **moreinfo**

For a complete listing of analytical reference materials for this method, request lit. cat.# 59304. For Rtx®-VMS column information, request lit. cat.# 59209.

Figure 1

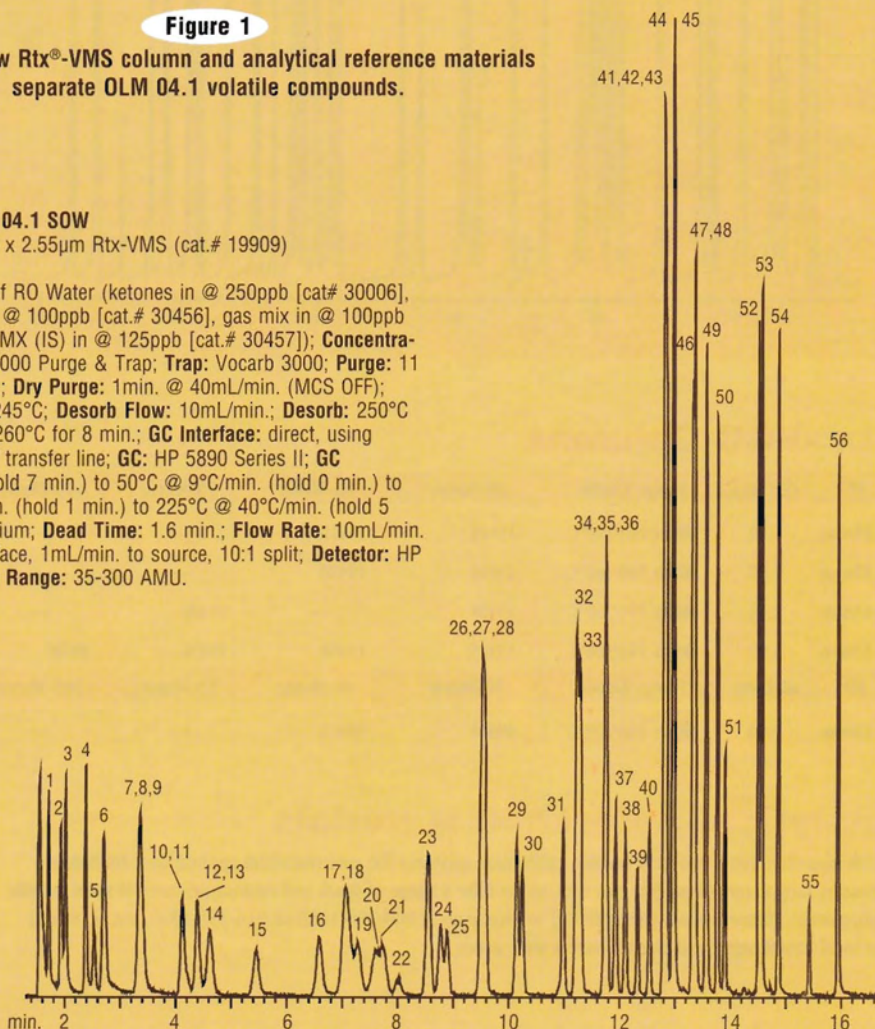
Restek's new Rtx®-VMS column and analytical reference materials separate OLM 04.1 volatile compounds.

### EPA Method OLM 04.1 SOW

60m x 0.45mm ID x 2.55µm Rtx-VMS (cat.# 19909)

100ppb in 25mL of RO Water (ketones @ 250ppb [cat# 30006], OLM MegaMix™ in @ 100ppb [cat.# 30456], gas mix in @ 100ppb [cat.# 30042], SS MX (IS) in @ 125ppb [cat.# 30457]); **Concentrator:** Tekmar LSC-3000 Purge & Trap; **Trap:** Vocarb 3000; **Purge:** 11 min. @ 40mL/min.; **Dry Purge:** 1min. @ 40mL/min. (MCS OFF); **Desorb Preheat:** 245°C; **Desorb Flow:** 10mL/min.; **Desorb:** 250°C for 2 min.; **Bake:** 260°C for 8 min.; **GC Interface:** direct, using 0.32mm ID Siltek™ transfer line; **GC:** HP 5890 Series II; **GC Program:** 40°C (hold 7 min.) to 50°C @ 9°C/min. (hold 0 min.) to 110°C @ 27°C/min. (hold 1 min.) to 225°C @ 40°C/min. (hold 5 min.); **Carrier:** Helium; **Dead Time:** 1.6 min.; **Flow Rate:** 10mL/min. to open-split interface, 1mL/min. to source, 10:1 split; **Detector:** HP 5971A MSD; **Scan Range:** 35-300 AMU.

1. dichlorofluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethane
8. carbon disulfide
9. 1,1,2-trichloro-1,2,2-trifluoroethane
10. methylene chloride
11. acetone
12. trans-1,2-dichloroethane
13. methyl acetate
14. methyl-tert-butyl-ether (MTBE)
15. 1,1-dichloroethane
16. cis-1,2-dichloroethane
17. bromochloromethane (IS)
18. cyclohexane
19. chloroform
20. carbon tetrachloride
21. 1,1,1-trichloroethane
22. 2-butanone
23. benzene
24. 1,2-dichloroethane-d4 (SMC)
25. 1,2-dichloroethane
26. trichloroethylene
27. methyl cyclohexane
28. 1,4-difluorobenzene (IS)
29. 1,2-dichloropropane
30. bromodichloromethane
31. cis-1,3-dichloropropene
32. toluene-d8 (SMC)
33. toluene
34. 4-methyl-2-pentanone
35. tetrachloroethane
36. trans-1,3-dichloropropene
37. 1,1,2-trichloroethane
38. dibromochloromethane
39. 1,2-dibromoethane
40. 2-hexanone
41. chlorobenzene-d5 (IS)
42. chlorobenzene
43. ethyl-benzene
44. m-xylene
45. p-xylene
46. o-xylene
47. styrene
48. bromoform
49. isopropyl benzene
50. 4-bromofluorobenzene (SMC)
51. 1,1,2,2-tetrachloroethane
52. 1,3-dichlorobenzene
53. 1,4-dichlorobenzene
54. 1,2-dichlorobenzene
55. 1,2-dibromo-3-chloropropane
56. 1,2,4-trichlorobenzene





# GC Methods Validated for Saw Palmetto

## Using Rtx®-5 and Stabilwax® Columns

by Sherry Navaroli, Food, Flavor, Fragrance Innovations Team Manager

Consumer demand for natural products and dietary supplements has grown exponentially, with increasing amounts of botanical materials being used in the manufacture of a large variety of products. The newly formed Institute for Nutraceutical Advancement (INA) has developed the Methods Validation Program (MVP) under the direction of a broad range of representatives from within the natural products industry. Companies from both the United States and Europe are represented. In addition, ten major natural products organizations and the Food and Drug Administration (FDA), have accepted seats on the INA MVP Advisory Committee as a way of ensuring that the process is inclusive.\*

Two methods involve gas chromatography (GC) for the analysis of fatty acids and sterols in saw palmetto, a fruit which is thought to have physiological benefits.

### Determination of Fatty Acids in Serenoa Repens (Saw Palmetto or Sabel) by GC

The INA assay can be used to determine fatty acid distribution in saw palmetto fruit, oil extract, and blended powders. Determination is performed using GC, after transesterification of the triglycerides into the fatty acid methyl esters. For more specific information on the method itself and all procedures involved, please refer to <http://www.nutraceuticalinstitute.com/methods/fattyacids.html>

The fatty acids from saw palmetto are separated in Figure 1, which was obtained using a Restek Stabilwax® column and a Shimadzu GC-14A, with split injection and a flame ionization detector (FID). The Stabilwax® column offers the necessary efficiency and selectivity to provide accurate identification of the fatty acids (as methyl esters).

### Determination of Sterols in Serenoa Repens (Saw Palmetto or Sabel) by GC

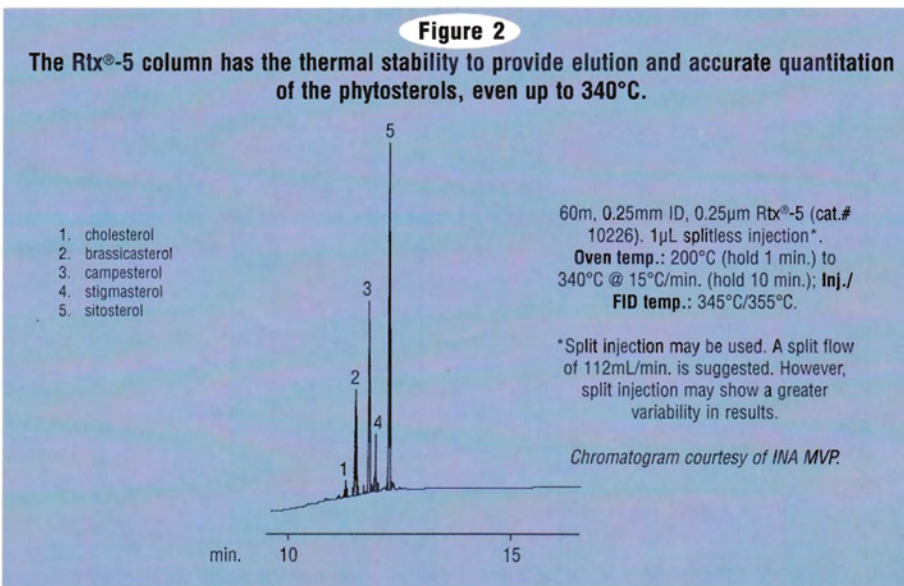
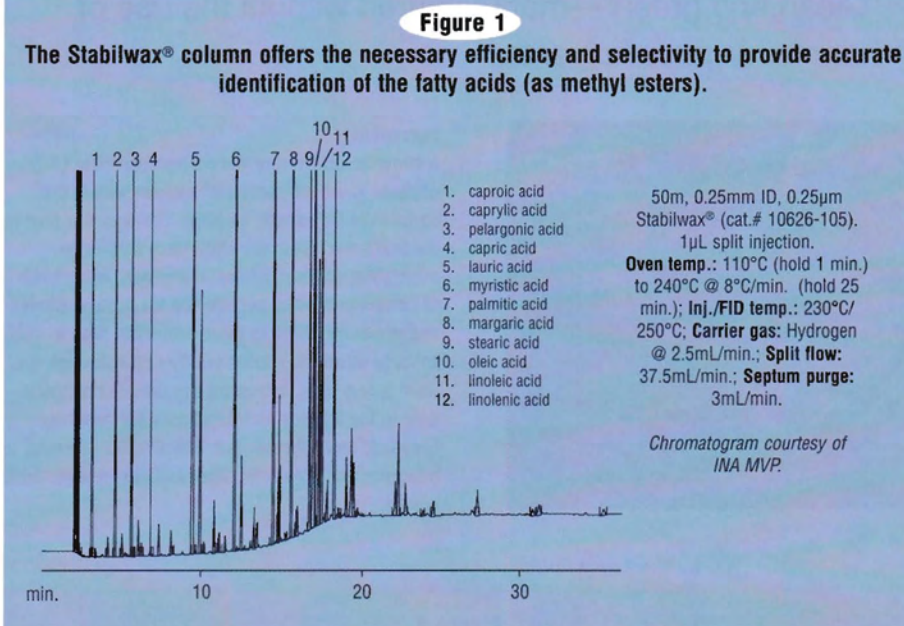
This assay can be used to determine stigmasterol, campesterol, brassicasterol, and sitosterol in saw palmetto fruit, oil extract, and blended powders. Determination is performed using GC after hydrolysis, saponification, and derivatization. For more specific information on the method itself and all procedures involved, please refer to <http://www.nutraceuticalinstitute.com/methods/sterols.html>

The sterols from saw palmetto are shown in Figure 2, which was obtained by using a Restek Rtx®-5 column and a Hewlett Packard 5890 Series II GC equipped with an FID and an autosampler. The Restek Rtx®-5 column contains a 5% diphenyl/95% dimethyl polysiloxane phase, and has the thermal stability to provide elution and accurate quantitation of the phytosterols, even up to 340°C.

Special thanks to Dr. Mark Lange, Director, and to Kathryn Bass, Marketing Director, of MVP for allowing us to print this material.

for **moreinfo**

Request the Applications Note  
lit. cat.# 59136.



### Stabilwax® Column

ID	df (µm)	Stable to	50m
0.25mm	0.25	250°C	10626-105

### Rtx®-5 Column

ID	df (µm)	Stable to	60m
0.25mm	0.25	360°C	10226

\*All information about the INA can be viewed at their website: <http://www.nutraceuticalinstitute.com/whoweare>



# Siltek™ Deactivation Delivers Inertness and Durability

by Deborah Salabsky, Applications Chemist

- ✓ Maximizes the inertness of sample pathway.
- ✓ Minimizes breakdown.
- ✓ Low bleed, thermally stable.
- ✓ Resistant to chemical attack.
- ✓ "Clean and green"—manufactured without the use of harmful organic solvents.



Siltek™—by Restek



## Inertness

A common concern in gas chromatographic (GC) analyses is the interaction of analytes with active surfaces in the sample pathway. The injection port is the first source of active sites, often leading to adsorption and breakdown of analytes. Trace levels of compounds often are injected via splitless mode, and are more prone to these problems. With a splitless injection, carrier gas flow rate through the liner is very slow, increasing the sample residence time in the injector and the chance for reactivity. Complete and effective liner deactivation is crucial to minimize available active sites and ensure repeatable results.

## Durability

Restek has designed Siltek™ deactivation to deliver both enhanced inertness and durability. Gas chromatography accessories coated with Siltek™ deactivation provide durability for matrices of pH extremes and high-temperature applications.

A splitless injection of the XTI® mix with an on-column concentration of 4-10ng shows an excellent response for all of the probes, including the active compounds dinitrophenol, 1,2-hexanediol, and benzoic acid (Figure 1).

## Resistance to chemical attack.

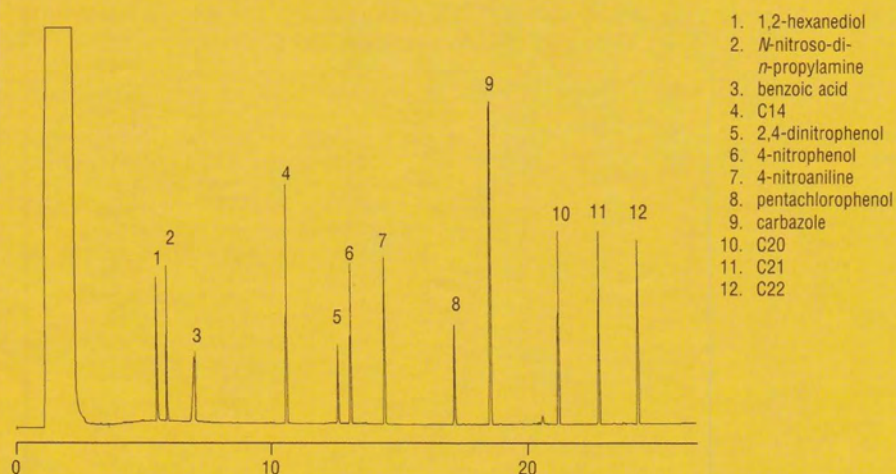
Very low or very high pH samples can degrade the deactivation layer, eventually resulting in an active surface. For this study, a baseline XTI® injection was made via direct injection and relative response factors were calculated. Key probes, such as 2,4-dinitrophenol, pentachlorophenol, and 1,2-hexanediol, retained their responses up to at least 120µL injected (Figure 2). The experiment was repeated with an identical set-up using aqueous NH<sub>4</sub>OH injections, pH 10.1. Under these demanding conditions, the response for the XTI® compounds was consistent for 70 injections.\*

## Conclusion

Siltek™ deactivation offers both inertness and resistance to temperature and pH extremes within a GC system. For more information, request Applications Note lit. cat.# 59113.

**Figure 1**

Siltek™ liner shows excellent inertness for acidic and basic probes.



30m, 0.25mm ID, 0.25µm XTI®-5 (cat.# 12223) with a Siltek™ 4mm splitless single gooseneck sleeve (cat.# 20798-214.1).

On-column concentration of 4-10ng. **Oven temp.:** 40°C (hold 2 min.) to 100°C @ 30°C/min., to 180°C @ 9°C/min., to 330°C @ 30°C/min. (hold 10 min.); **Inj. temp.:** 250°C; **Det.:** 330°C; **Carrier gas:** He.

\*Refer to Applications Note lit. cat.# 59113.

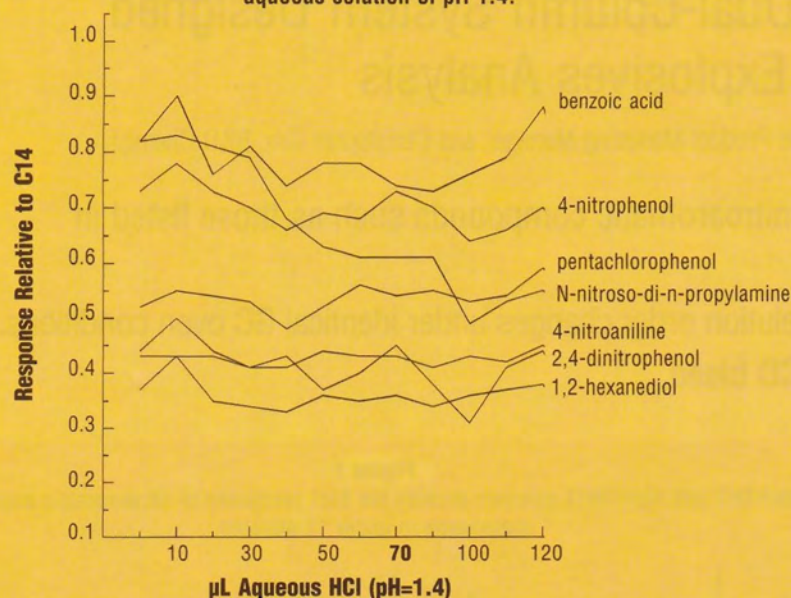
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For more information on Siltek™ deactivation, request information packet lit. cat.# 59803.



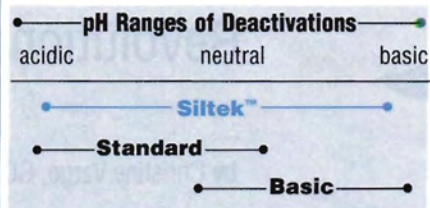
**Figure 2**

Difficult probes retain their response on Siltek™ deactivation up to 120µL of an aqueous solution of pH 1.4.

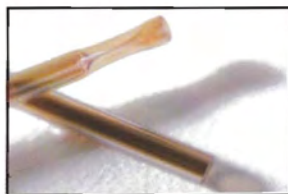


30m, 0.25mm ID, 0.25µm XTl®-5 (cat.# 12223) with a Siltek™-deactivated 4mm open-top Uniliner® w/o wool (cat.# 20843-214.1).  
Oven temp.: 40°C (hold 2 min.) to 190°C @ 6°C/min., to 330°C @ 30°C/min. (hold 10 min.); Inj. temp.: 250°C; Det.: 330°C; Carrier gas: He.

## Correction



In the Winter 2000 Restek Advantage (lit. cat.# 59402) article, "Siltek™ Deactivation" (p. 12), we inadvertently placed numerical pH indicators on a figure depicting the performance variations of various deactivation surfaces with acidic and basic analytes. The purpose of this figure is to present a general classification of analytes that are compatible with either Siltek™ deactivation, Restek standard deactivation, and Restek basic deactivation. The use of actual pH values is inappropriate for this purpose and the figure is reprinted correctly above.



## Siltek™ Inlet Liners

For Siltek™-deactivated inlet liners, add the corresponding suffix number to your liner catalog number. Refer to the Annual Chromatography Products Guide for liner catalog numbers.

qty.	Siltek™	Siltek™ with Siltek™-deactivated wool	Siltek™ with CarboFrit™
each	-214.1	-213.1	-216.1
5-pk.	-214.5	-213.5	-216.5



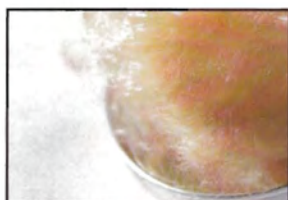
## Siltek™ Press-Tight™ Connectors

Type	ea.	3-pk.	5-pk.	25-pk.	100-pk.
straight	—	—	20480	20449	20481
angled	—	—	20482	20483	20484
"Y"	20485	20486	—	—	—
angled "Y"	20487	20469	—	—	—



## Siltek™ Guard Columns

nominal ID	nominal OD	5-meter	10-meter
0.25mm	0.37 ±0.04mm	10026	10036
0.32mm	0.45 ±0.04mm	10027	10037
0.53mm	0.69 ±0.04mm	10028	10038



## Siltek™ Borosilicate Wool

qty.	cat.#
10g	21100





# Rtx®-TNT/TNT2 Columns

## Revolutionary Dual-Column System Designed for Explosives Analysis

by Christine Vargo, GC Columns Product Marketing Manager, and Christopher Cox, R&D Chemist

- ✓ Provides baseline resolution of nitroaromatic compounds such as those listed in US EPA Method 8095.
- ✓ Confirmation column achieves 8 elution order changes under identical GC oven conditions.
- ✓ High thermal stability for low ECD bleed.

Restek has designed the Rtx®-TNT and Rtx®-TNT2 columns specifically to analyze nitroaromatic compounds by GC/ECD, such as those listed in US Environmental Protection Agency (EPA) Method 8095. The TNT columns provide better resolution and higher thermal stability than any of the columns currently recommended in Method 8095. The Rtx®-TNT primary column and Rtx®-TNT2 confirmation column operate under identical GC oven temperature programs, allowing simultaneous dual-column confirmational analysis of all 16 nitroaromatic compounds.

Column stationary phase, dimensions, and injection technique have been optimized to ensure the best chromatographic performance. The Rtx®-TNT/Rtx®-TNT2 columns are optimized at 6 meters, 0.53mm ID, and 1.5µm film thickness. The 6-meter length minimizes surface area and contact times of thermally labile explosives such as octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX). For explosives analysis, a 100% dimethyl polysiloxane (Rtx®-1) phase typically is used for the primary column and either a 100% trifluoropropylmethyl polysiloxane phase (Rtx®-200) or a 50% cyanopropylmethyl/50% phenylmethyl polysiloxane (Rtx®-225) phase is used for the confirmational column. The Rtx®-TNT and Rtx®-TNT2 columns provide the best resolution of the nitroaromatics in the fastest analysis time compared to recommended columns (Figure 1). A direct injection of the explosives using a Siltek™ Uniliner® is recommended to minimize injection port discrimination, sample adsorption, and low response of HMX.

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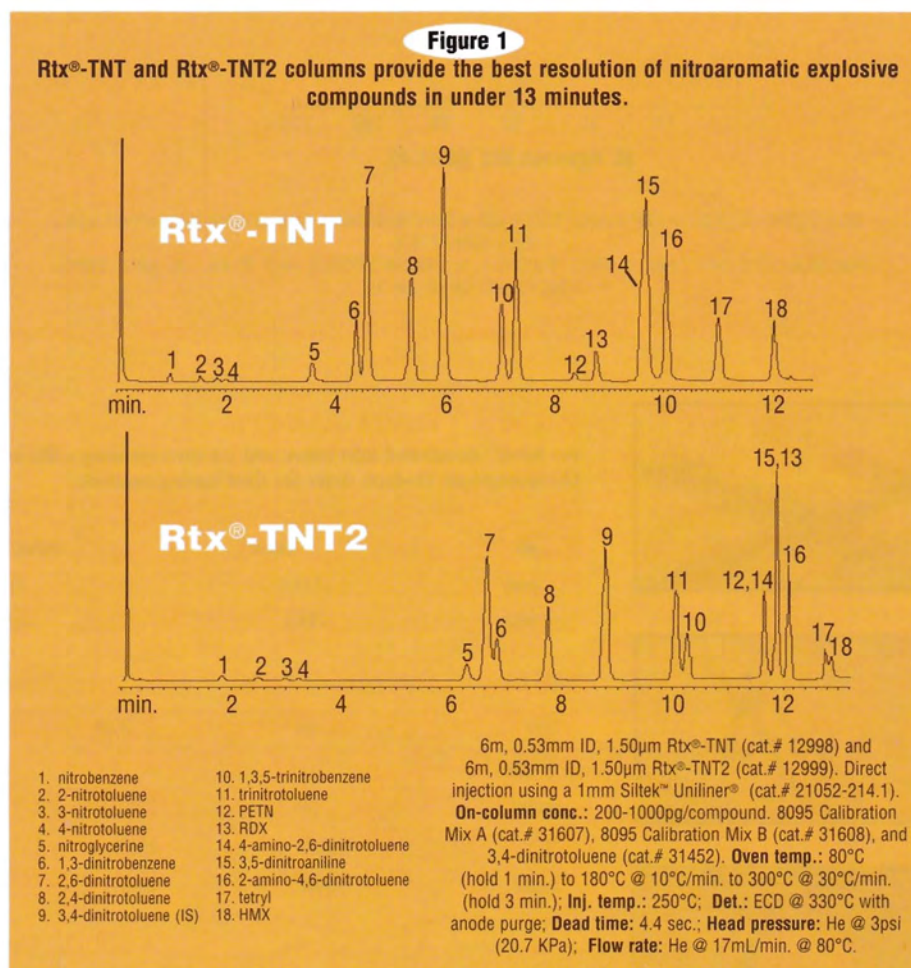
Request the Rtx®-TNT Columns Brochure  
(lit. cat.# 59218).

#### 1mm Siltek™ Uniliner® Inlet Liner

For HP 5890 split/splitless injection ports

cat.# 21052-214.1 (ea.)

cat.# 21053-214.5 (5-pk.)



#### Rtx®-TNT & Rtx®-TNT2 Columns (Columns provided in a 3-pack without cages.)

Column	ID	df (µm)	Temp. Limits	6-Meter (3-pk.)
Rtx®-TNT	0.53mm	1.50	-20 to 300/310°C	12998
Rtx®-TNT2	0.53mm	1.50	-20 to 300/310°C	12999

US EPA 8095 explosive calibration mixtures are available from Restek.  
Request lit. cat.# 59229 for more information.



# Rtx®-5Sil MS

## Best Resolution of Gasoline Range Organic (GRO) Compounds

by Christopher English, Applications Chemist

The Alaska Department of Environmental Conservation (ADEC) developed a new technique for the gas chromatographic (GC) analysis of gasoline range organic (GRO) compounds in soil, water, and waste water—Method AK101AA. This method quantitates aromatic and aliphatic compounds from C6 (hexane) to C10 (decane), and is capable of a higher level of accuracy over existing GRO methods. Restek's Rtx®-5Sil MS column is ideal for the analysis of GRO compounds, and specifically meets the requirements of Method AK101AA.

### Alaska UST Method AK101AA

benzene	toluene
ethylbenzene	1,2,3-trimethylbenzene
1-ethyl-2-methylbenzene	1,2,4-trimethylbenzene
1-ethyl-3-methylbenzene	1,3,5-trimethylbenzene
1-ethyl-4-methylbenzene	<i>o</i> -xylene
isopropylbenzene	<i>m</i> -xylene
<i>n</i> -propylbenzene	<i>p</i> -xylene

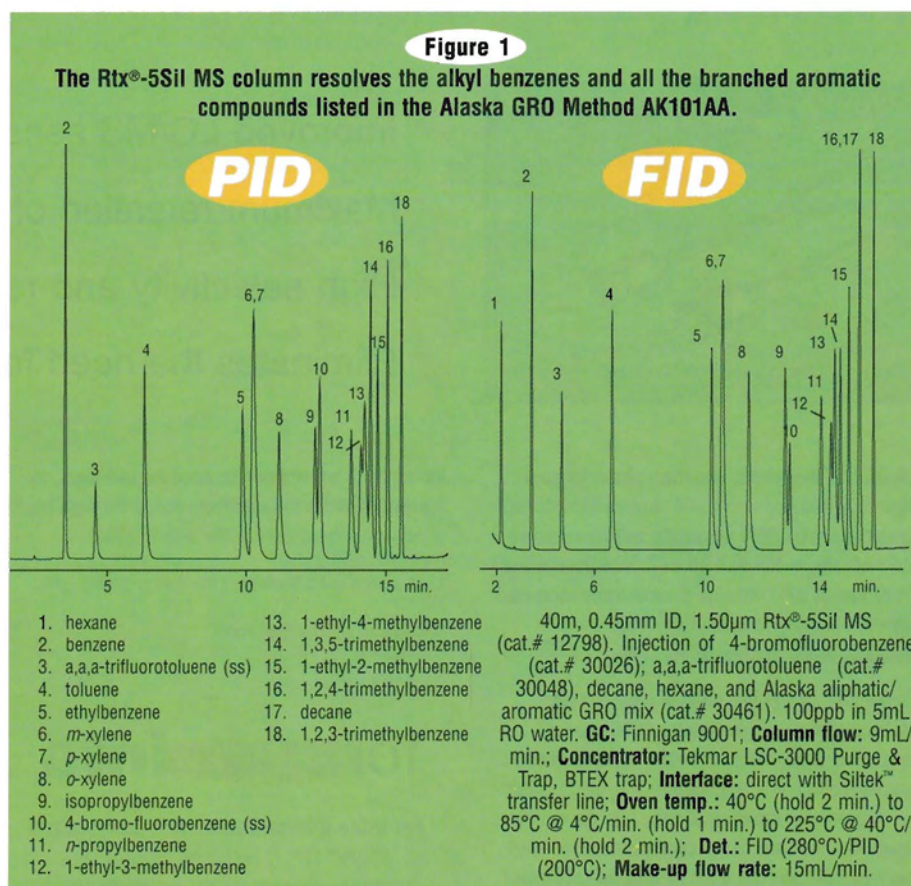
1,000µg/mL ea. in P&T methanol, 1mL/ampul.

Each	5-pk.	10-pk.
<b>without data pack</b>		
30461	30461-510	—
<b>w/ data pack</b>		
30461-500	30461-520	30561

### Rtx®-5Sil MS Columns

(Stable to 300/320°C)

ID	df (µm)	30m	40m	60m
0.45mm	1.50	—	12798	—



## Connecticut ETPH Method

### New Analytical Reference Materials Available

by Christopher Cox, R&D Chemist

Connecticut has recently changed the analytical procedure to test for the presence of petroleum products in the nonane (C9) to hexatriacontane (C36) hydrocarbon range.<sup>1</sup> This new method uses methylene chloride extraction, high resolution gas chromatography (GC) separation, and flame ionization detection (FID). Restek offers the calibration mixtures, surrogates, internal standards, and composite petroleum standards needed for this method. Restek also offers the high resolution capillary columns, direct injection Uniliner® sleeves, and septa needed for this analysis.

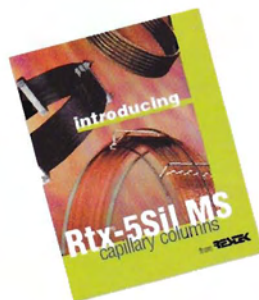
1 Analysis of Extractable Total Petroleum Hydrocarbons (ETPH) Using Methylene Chloride Gas Chromatography/Flame Ionization Detection, Environmental Research Institute, University of Connecticut, March, 1999. Reference not available from Restek. Available from Department of Environmental Protection, 79 Elm Street, Hartford, CT 06106.

### Connecticut ETPH Calibration Mixture

nonane (C9)	tetracosane (C24)
decane (C10)	hexacosane (C26)
dodecane (C12)	octacosane (C28)
tetradecane (C14)	triacontane (C30)
hexadecane (C16)	dotriacontane (C32)
octadecane (C18)	tetratriacontane (C34)
eicosane (C20)	hexatriacontane (C36)
docosane (C22)	

1000µg/mL each in methylene chloride, 1mL per ampul

Each	5-pk.	10-pk.
<b>without data back</b>		
31614	31614-510	—
<b>w/ data back</b>		
31614-500	31614-520	31714





# Allure™ PFP Propyl Columns

## Improved LC/MS Analysis of Basic Compounds

by C. Vernon Bartlett, HPLC Applications Manager



- ✓ Improved LC/MS sensitivity.
- ✓ Maximum retention of basic compounds.
- ✓ High selectivity and reproducibility.
- ✓ Eliminates the need for modifiers.

Selection of the proper stationary phase for your separation can improve liquid chromatography/mass spectrometry (LC/MS) sensitivity, analyte retention, and peak shape. Restek's Allure™ pentafluorophenyl (PFP) Propyl HPLC column demonstrates increased retention of basic analytes without the use of modifiers or ion pairing agents (Figure 1).

The Allure™ PFP Propyl column separates and retains analytes such as cocaine (COC) and its metabolite, ecgonine methyl ester (EME), using 90% acetonitrile in under 4.5 minutes (Figure 2). As the concentration of the organic solvent in the mobile phase increases, the desolvation process becomes more effective and the LC/MS ESI signal increases.<sup>1</sup>

Restek's Allure™ PFP Propyl column is the best choice for analysis of basic analytes. It provides superior retention and peak shape for analytes

having  $pK_a$ 's >8 without the need for modifiers, and it provides increased sensitivity due to the high level of organic solvent used in the mobile phase.

1. P. Sjöberg and K. Markides, *J. Chromatogr. A*, 855 (1999) 317-327.

Reference not available from Restek.

### for *more* info

For more information on this analysis and Allure™ HPLC columns, request the Applications Note lit. cat.# 59118.

### Free HPLC Wall Chart & LC/MS Catalog

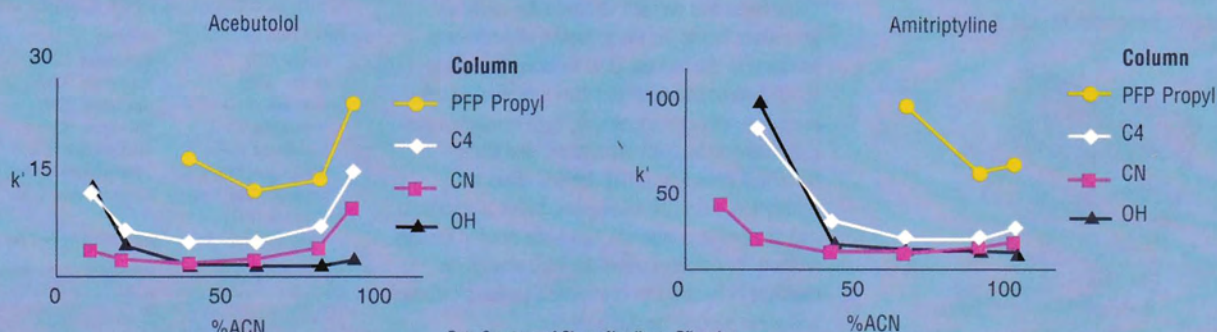


To receive your free wall chart (lit. cat.# 59894) and catalog (lit. cat.# 59607) call 800-356-1688 or 814-353-1300, ext. 5, or visit us online at [www.restekcorp.com](http://www.restekcorp.com).

**Figure 1**

**Allure™ PFP Propyl demonstrates increased retention of basic analytes**

5mM NH<sub>4</sub>OAc, pH 4.5; ACN; 30x2.1mm; 0.4mL/min.



Data Courtesy of Shane Needham, Pfizer Inc

PLC-0001



Figure 2

Allure™ PFP Propyl achieves improved response for cocaine and its metabolites.

5mM NH<sub>4</sub>OAc, pH 4.5:ACN; 30x2.1mm; 0.4mL/min.

## Peak List:

1. EME (ecgonine methyl ester)
2. COC (cocaine)

## Sample Conditions:

Inj.: 10µL  
 Conc.: 1µg/mL  
 Solvent: water  
 Temp.: 4°C

**Optimized  
mobile phase provides  
12-fold increase in COC  
response!**

## Column:

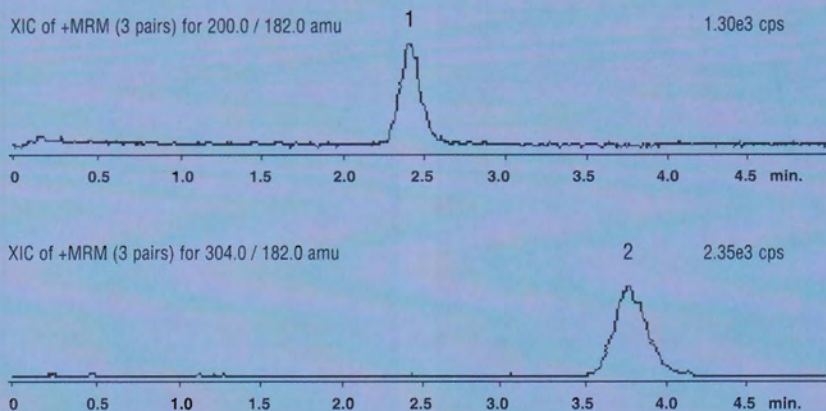
Column: Allure™ PFP Propyl  
 Catalog#: 9169532  
 Dimensions: 30mm x 2.1mm  
 Particle size: 5µm  
 Pore size: 60Å

## Conditions:

Mobile phase: 5mM pH 3.0  
 ammonium formate:  
 acetonitrile (10:90)  
 0.6mL/min.  
 Column temp.: ambient

## Det.:

PE/Sciex API 3000  
 Interface: Turbo Ion Spray, ESI  
 Interface temp.: 150°C  
 Ion mode: positive  
 ESI probe voltage: 5000V  
 Orifice: +/- 71V  
 Ring: +/- 265V  
 Collision gas: Nitrogen  
 Collision gas pressure: 2.2 mTorr  
 Collision gas energy(COC): 28 eV  
 Collision gas energy(EME): 26 eV  
 Electron multiplier: 2100 volts  
 Auxiliary gas flow: 7000cc/min.  
 Nebulizer gas setting: 15lb/in.<sup>2</sup>  
 Curtain gas setting: 12lb/in.<sup>2</sup>



Data Courtesy of Shane Needham, Pfizer Inc

LC\_0126

## Allure™ PFP Propyl, 5µm Columns

Particle Size: 5µm	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	9169531	9169532	9169533	9169535
50mm length	9169551	9169552	9169553	9169555
100mm length	9169511	9169512	9169513	9169515
150mm length	9169561	9169562	9169563	9169565
200mm length	9169521	9169522	9169523	9169525
250mm length	9169571	9169572	9169573	9169575

## Allure™ PFP Propyl, 5µm Columns with Trident™ Inlet

Particle Size: 5µm	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	—	9169532-700	9169533-700
50mm length	—	9169552-700	9169553-700
100mm length	—	9169512-700	9169513-700
150mm length	—	9169562-700	9169563-700
200mm length	—	9169522-700	9169523-700
250mm length	—	9169572-700	9169573-700



## Restek Survival Kit for HPLC

The Restek Survival Kit is an invaluable analytical spare-parts kit that contains the essential tools and supplies to maintain and set-up your solvent delivery system. For start-up and standard use in all HPLC systems.

cat.# 25322



# USP <467> Method Updates

## GC Analysis of Organic Volatile Impurities

by Christopher Cox, R&D Chemist

Since its original appearance in the USP, the testing protocol for gas chromatographic (GC) analysis of Organic Volatile Impurities (OVI) in pharmaceutical products (USP <467>) has undergone many revisions and additions.<sup>1-6</sup> The most recent of which was published in USP 24, effective January 1, 2000.<sup>7</sup> The biggest change was to the limit test concentrations, which now match the European Pharmacopoeia (EP) concentrations and the ICH guidelines for the five USP <467>-regulated solvents (Table 1).<sup>8,9</sup>

**Table 1**

**New limit test concentrations for USP <467>.  
Effective January 1, 2000.**

benzene	2ppm
chloroform	60ppm
1,4-dioxane	380ppm
methylene chloride	600ppm
trichloroethene	80ppm

USP issued an in-process revision announcement that the limit test for benzene is not required unless a specific limit for benzene is included in the individual drug monograph (12). This was needed because of the inability to detect benzene at 2 ppm with method I and method IV. Method IV is currently the only method that detects benzene at 2 ppm. Our understanding, at the time of this printing, is that the FDA has not waived the need to test for benzene and that most laboratories running method I and V are testing for benzene at 100ppm. It is anticipated that USP will make more revisions to the benzene detection limits or testing procedures this year.

**Figure 1** shows an analysis using USP <467> Method I on a G27 analytical column with a phenylmethyl guard column. Please note that the sample preparation used in this analysis deviates from the method-specified 1:50 dilution in distilled water. A 1:10 dilution in distilled water was used to obtain a detectable amount of benzene by direct injection.

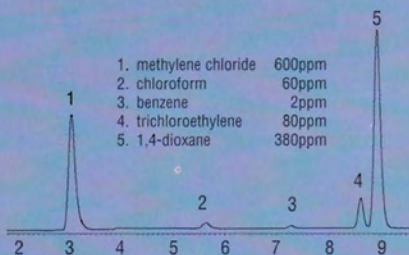
USP also has clarified that a 5m phenylmethyl guard column is not needed for the Method IV, headspace analysis.<sup>10</sup> **Figure 2** shows an analysis using Method IV at the revised concentrations, the method-specified sample preparation procedure, a G43 analytical column, and no guard column.

We have developed three new products to meet the current change:

- USP <467> Calibration Mix #4 (cat.# 36006, dissolved in methanol)
- USP <467> Calibration Mix #5 (cat.# 36007, dissolved in dimethylsulfoxide)
- Rtx®-G43 column without an Integra-Guard™ guard column

**Figure 1**

**A minor modification of the dilution concentration for Method I allows analysis of 2ppm benzene using an Rtx®-G27 column.**

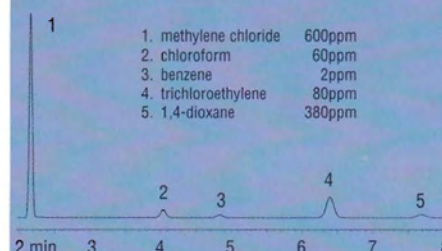


**Sample Preparation:** 1:10 dilution of cat.# 36007 in distilled water (this deviation from the method's 1:50 dilution was needed to obtain a detectable benzene peak).

30m, 0.53mm ID, 5.0µm Rtx®-G27 with 5m phenylmethyl Integra-Guard™ (cat.# 10279-126).  
**Oven temp.:** 35°C (hold 5 min.) to 175°C @ 8°C/min., to 260°C @ 35°C/min. (hold 16 min.); **Inj. port:** Uniliner® direct injection sleeve 70°C; **Inj. size:** 1µL; **Det. temp.:** 260°C; **FID sensitivity:** 1 x 10<sup>-12</sup> AFS; **Carrier gas:** helium, 4.1psi constant pressure, 35cm/sec. set @ 35°C.

**Figure 2**

**The Rtx®-G43 column provides the resolution and detection limits needed for USP 24<sup>th</sup> edition <467> revised limit test concentrations in USP Method IV.**



**Sample Preparation:** 100µL of cat.# 36007 in 5mL distilled water, 1 gram sodium sulfate in a 20mL headspace vial.

30m, 0.53mm ID, 3.0µm Rtx®-G43 (cat.# 16085)  
**Oven temp.:** 40°C (hold 20 min.) to 240°C @ 35°C/min. (hold 20 min.); **Inj. temp:** 140°C, 1mm split sleeve (cat.# 20916); **Det. temp.:** 260°C; **FID sensitivity:** 1.25 x 10<sup>-11</sup> AFS; **Carrier gas:** helium, 3.5psi constant pressure, 35cm/sec. set @ 40°C; **Split ratio:** 2:1; ThermoQuest HS 2000 Headspace Autosampler Vial 80°C, 60 min. shaker on.

**Rtx®-G27 with 5m phenylmethyl Integra-Guard™** (5% phenyl/95% methyl polysiloxane)

ID	df (µm)	Temp. Limits	30-Meter
0.53mm	5.00	-60 to 270/290°C	10279-126

**Rtx®-G43 with 5m phenylmethyl Integra-Guard™**

(6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	Temp. Limits	30-Meter
0.53mm	3.00	-20 to 240°C	16085-126

**Rtx®-G43 w/out Integra-Guard™ USP <467> Method IV**

(6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	Temp. Limits	NEW 30-Meter
0.53mm	3.00	-20 to 240°C	16085

**USP 467 Calibration Mix #4**

benzene	2µg/mL
chloroform	60
1,4-dioxane	380
methylene chloride	600
trichloroethene	80

Prepared in methanol, 1mL/ampul

each	10-pk.
36006	36106

**USP 467 Calibration Mix #5**

benzene	2µg/mL
chloroform	60
1,4-dioxane	380
methylene chloride	600
trichloroethene	80

Prepared in dimethylsulfoxide, 1mL/ampul

each	10-pk.
36007	36107

**formoreinfo**

For the latest details on this analysis and a list of references, request the Applications Note lit. cat.# 59577A.

<sup>1-10</sup> Request lit. cat# 59577A for a list of references.



# New Stationary Phases and Solid Supports for USP Methods

by Barry Burger, Applications Chemist

- ✓ New Silcoport™ S1AB replacement for recently-discontinued Gas-Chrom® Q.
- ✓ New Rt-XE 60 liquid phase replacement for GE®-XE-60.
- ✓ Phases and solid supports fully tested to assure lot-to-lot reproducibility.

Two commonly-used products for the gas chromatographic (GC) analyses specified in US Pharmacopoeia (USP) methods are no longer available from their original manufacturers—Gas-Chrom Q (S1AB), a deactivated acid/base-modified solid support that is specified in over 40 USP methods, and GE® XE-60, a 25% cyanoethyl/75% methylpolysiloxane liquid phase specified as USP G26.

To meet continuing needs for these products, Restek offers a complete line of high-quality, fully-tested liquid phases and solid supports for USP methods (Tables 1 and 2). **SilcoPort™ S1AB** solid support, a deactivated acid/base-modified diatomaceous earth, was designed specifically for USP methods. Restek's polymer chemists also synthesized **Rt-XE 60** liquid phase, a direct replacement for the original GE®-XE-60 material. Figure 1 compares the Kovats data generated by the new Rt-XE 60 phase and the original GE®-XE-60 liquid phase. Each polymer batch is QA tested and must conform to rigid Kovats specifications.

All liquid phases and solid supports available from Restek are fully tested in accordance with our in-house Quality Control program. All solid supports are classified to meet stringent particle size distribution windows for maximum performance and subsequently are deactivated and tested.

**Figure 1**

Quality assurance testing includes retention indices for lot-to-lot reproducibility.

## Kovats Retention Indices

	GE XE-60	Restek Rt-XE 60
X' benzene	857	857
Y' n-butanol	967	966
Z' 2-pentanone	967	966
U' 1-nitropropane	1147	1146
S' pyridine	1064	1063

**Table 1**

Restek offers all GC liquid phases listed in the USP, 24th edition.

USP	Phase description	Restek equivalent
G1	Dimethylpolysiloxane oil	Rt-2100, OV-101, Rtx®-1
G2	Dimethylpolysiloxane gum	OV-1
G3	50% phenyl-50% methylpolysiloxane	Rt-2250, OV-17
G4	Diethylene glycol succinate polyester	Rt-DEGS
G5	3 cyanopropylpolysiloxane	Rt-2340
G6	trifluoropropylmethylpolysiloxane	Rt-2401, OV-210
G7	50% 3 cyanopropyl-50% phenylmethylsilicone	Rt-2300
G8	90% 3 cyanopropyl-10% phenylmethylsilicone	Rt-2330
G9	methylvinylpolysiloxane	UCW 98
G10	Polyamide	Polyamide
G11	bis(2 ethylhexyl) sebacate polyester	bis(2 ethylhexyl) sebacate polyester
G12	phenyldiethanolamine succinate polyester	phenyldiethanolamine succinate polyester
G13	sorbitol	sorbitol
G14	polyethylene glycol (ave. mol. wt. 950-1050)	Carbowax® 1000
G15	polyethylene glycol (ave. mol. wt. 3000-3700)	Carbowax® 4000
G16	Polyethylene glycol compound (ave. mol. wt. 15,000) A high molecular weight compound of polyethylene glycol and a diepoxide linker.	Carbowax® 20M
G17	75% phenyl-25% methylpolysiloxane	OV-25
G18	Polyalkylene glycol	UCON LB 550X
G19	25% phenyl-25% cyanopropyl-50% methylsilicone	OV 225
G20	polyethylene glycol (ave. mol. wt. 380-420)	Carbowax® 400
G21	neopentyl glycol succinate	neopentyl glycol succinate
G22	bis(2 ethylhexyl) phthalate	bis(2 ethylhexyl) phthalate
G23	polyethylene glycol adipate	EGA
G24	diisodecyl phthalate	diisodecyl phthalate
G25	Polyethylene glycol compound TPA. A high molecular weight compound of a polyethylene glycol and a diepoxide that is esterified with terephthalic acid	Carbowax® 20M TPA
G26	25% 2-cyanoethyl-75% methylpolysiloxane	Rt-XE 60
G27	5% phenyl-95% methylpolysiloxane	SE-52
G28	25% phenyl-75% methylpolysiloxane	DC 550
G29	3,3'-thiodipropionitrile	TDPN
G30	tetraethylene glycol dimethyl ether	tetraethylene glycol dimethyl ether
G31	nonylphenoxypoly(ethyleneoxy)ethanol (ave. ethyleneoxy chain length is 30); nonoxynol 30	Igepal® CO 880
G32	20% phenylmethyl-80% dimethylpolysiloxane	OV-7
G33	20% Carborane® 80% methylsilicone	Dexsil® 300
G34	Diethylene glycol succinate polyester stabilized with phosphoric acid	Rt-DEGS PS
G35	A high molecular weight compound of a polyethylene glycol and a diepoxide that is esterified with nitroterephthalic acid	Rt-1000
G36	1% vinyl 5% phenylmethylpolysiloxane	SE 54, Rtx®-5
G37	polyimide	polyimide
G38	phase G1 containing a small amount of tail inhibitor	Rt-2100/0.1% Carbowax 1500
G39	polyethylene glycol (ave mol. wt. 1500)	Carbowax® 1500
G40	ethylene glycol adipate	Rt-EGA

<sup>1</sup>USP 24/NF19 <621> Chromatography pp.1924-1926



# New Stationary Phases and Solid Supports for USP Methods, (cont.)

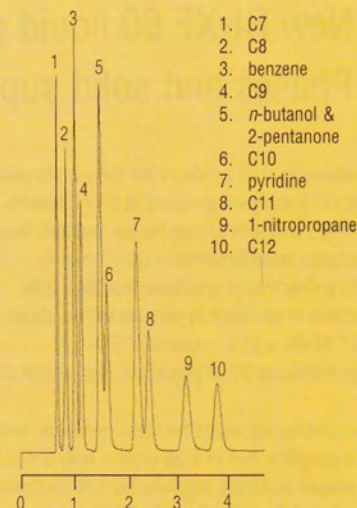
**Table 2**

Restek offers all GC solid supports listed in the USP, 24th edition.

USP	Support description	Restek equivalent
S1A	siliceous earth, see method 1 for details on treatment	Silcoport™
S1AB	siliceous earth, treated as S1A and both acid- and base-washed	Silcoport™ WBW
S1C	Crushed firebrick, calcined or burned with a clay binder above 900°C, acid-washed, may be silanized	Chromosorb® PAW or PAW DMDCS
SINS	untreated siliceous earth	Chromosorb® W- Non Acid Washed
S2	styrene-divinylbenzene copolymer with nominal surface area of less than 50m <sup>2</sup> /g and an ave. pore diameter of 0.3 to 0.4µm	Chromosorb® 101
S3	styrene-divinylbenzene copolymer with nominal surface area of 500 to 600m <sup>2</sup> /g and an ave. pore diameter of 0.0075µm	Hayesep® Q
S4	styrene-divinylbenzene copolymer with aromatic -O and -N groups having a nominal surface area of 400 to 600m <sup>2</sup> /g and an ave. pore diameter of 0.0076µm	Hayesep® R
S5	high molecular weight tetrafluorethylene polymer, 40- to 60-mesh	Chromosorb® T
S6	styrene-divinylbenzene copolymer having a nominal surface area of 250 to 350m <sup>2</sup> /g and an ave. pore diameter of 0.0091µm	Chromosorb® 102
S7	graphitized carbon having a nominal surface area of 12m <sup>2</sup> /g	CarboBlack™ C
S8	copolymer of 4-vinyl-pyridine and styrene divinylbenzene	Hayesep® S
S9	porous polymer based on 2,6-diphenyl-p-phenylene oxide	Tenax® TA
S10	highly cross-linked copolymer of acrylonitrile and divinylbenzene	Hayesep® C
S11	graphitized carbon having a nominal surface area of 100m <sup>2</sup> /g, modified with small amounts of petrolatum and polyethylene glycol compound	CarboBlack™ B 80/120 3% Rt 1500
S12	graphitized carbon having a nominal surface area of 100m <sup>2</sup> /g	CarboBlack™ B

**Figure 2**

Quality assurance determination shows liquid phase selectivity using Kovats retention indices.



20% Rt-XE60 on 100/120 Chromosorb® W NAW  
2-Meter x 2mm ID Silcosteel® column

Col. temp.: 120°C; Flow rate: Helium, 20 mL/min.;  
Inj. temp.: 200°C; Det. temp.: 250°C; Sample  
conc.: neat; Sample size: 0.01µL

## Precision™ Inlet Liners (Formerly called Focal liners)

- ✓ Wool is placed at the injection point to maximize vaporization and help wipe the needle during injection.
- ✓ Designed for easy changing of the wool—no more guessing where the wool should be placed.
- ✓ Wool stays in position during pressure pulses in the inlet and during injection.
- ✓ Available with all Restek deactivations and packing materials.
- ✓ Direct replacement for SGE's Focus™ liners.

Instrument	each	<b>NEW</b>	5-pk.
HP 5890/6890 4mm Split Precision Liner	21022		21023
Varian 1078/1079 Split Precision Liner	21024		21025
Shimadzu 17A Split Precision Liner	21020		21021
Varian 1075/1077 Split Precision Liner	21030		21031
Fisons, Trace, 8000 Series 5mm Split Precision Liner	21028		21029
PE Auto SYS Split Precision Liner	21026		21027

## Capillary Column Caps



- ✓ Eliminates moisture and air from entering the column during storage.
  - ✓ Color-coded for identifying detector and injector end.
  - ✓ Attach to the column in seconds for an airtight seal.
  - ✓ Increases column lifetime.
  - ✓ Not recommended for reuse.
- cat.# 21044, (10-pk.)



# Peak Performers

by Brad Rightnour, Instrument Innovations Team Manager

## New GC Accessories!

### Drilled Uniliner® Sleeve for HP 6890 GCs



- ✓ Allows direct injections when using an EPC-equipped HP 6890.
  - ✓ Minimizes injection port discrimination in splitless mode.
  - ✓ Available with all Restek deactivations.
- cat.# 21054, (ea.)  
cat.# 21055, (5-pk.)

**NEW**

### Varian Split 1177 Inlet Liner



- ✓ Meets or exceeds instrument manufacturer specifications.
  - ✓ Available with all Restek deactivations.
- cat.# 21045, (ea.)  
cat.# 21046, (5-pk.)

**NEW**

### ReadySpike™ Vial & Syringe Holders



**NEW**

#### 21-Hole Holder

- ✓ Convenient storage for 2 syringes and 19 vials.
- ✓ Includes 19 pre-cleaned vials and Mininert® caps.
- ✓ Holds vials securely for easy transportation.
- ✓ Allows easy spiking of samples.

21-Hole Holder only: cat.# 21048, (ea.)

21-Hole Holder Kit: cat.# 21068, (kit)

#### 5-Hole Holder

- ✓ Convenient storage for syringes and vials.
  - ✓ Mounts onto GC for convenient accessibility.
- cat.# 21049, (ea.)

### Capillary Installation Gauge



**NEW**

- ✓ For use with HP-style nuts and ferrules.
  - ✓ Pre-seats ferrule onto column for consistent installations.
  - ✓ Made from high-quality stainless steel.
- cat.# 21034, (ea.)

### Finger-Tight Column Nuts



**NEW**

- ✓ Allows wrench-free column installations.
- ✓ Works with standard and compact (HP-style) ferrules.
- ✓ Made from high-quality stainless steel.

Finger-tight nut for use with compact (HP-style) ferrules: 21040, (2-pk.)

Finger-tight nut for use with standard (1/16") ferrules: 21041, (2-pk.)

Finger-tight nut for use with standard (1/16") compression fittings: 21042, (2-pk.)

### Encapsulated Ferrules

**NEW**

- ✓ Will not deform and stick in fittings.
- ✓ Allows re-usability of the ferrule.
- ✓ Less torque needed to seal ferrule.
- ✓ Unique blend of graphite provides less fragmentation and outgassing.
- ✓ For 1/16" compression fittings.



Ferrule ID (mm)	Fits column ID (mm)	cat.#	10-pk.
0.4	0.25	21036	
0.5	0.32	21037	
0.8	0.53	21038	

### GC Column Hangers



**NEW**

- ✓ Secure, short-term storage.
- ✓ Easily attaches to side of GC.
- ✓ Includes capillary column caps for sealing used columns.

GC Column Hangers: 21047, (2-pk.)

### Moisture Control By-Pass Line for Tekmar 3000 Purge and Trap



**NEW**

- ✓ Increase response for ketones, alcohols, and acetates.
  - ✓ Silcosteel®-treated tubing for increased inertness.
  - ✓ Suitable for US EPA Methods 8260, 524.2, and OLM4.1.
  - ✓ Easily attaches in minutes.
- cat.# 21035, (ea.)

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# RESTEK ADVANTAGE Newsletter

## 2012,2,3,4,5.1 >

**ARCHIVE**

**Overview**

**2006 -2012 x16**

**1992-2006 x 62**

### RESTEK ADVANTAGE

Our expertise, experience, and enthusiasm is your Advantage.

2012

#### New Approaches for Increasing Analytical Sensitivity

- 1,4-dioxane at 5.0 ppt in water via large volume injection in an unmodified splitless GC inlet...pp. 6-7
- Lower detection limits without dilution using extended calibration range for semivolatiles...pp. 8-9
- QuEChERS with LC-MS/MS and GCxGC-TOFMS for comprehensive pesticide residue testing...pp. 12-13

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2013

#### Get Sizzling Separations with the Right Selectivity

- New Rxt<sup>®</sup>-PAH column selectivity resolves all EFSA PAH4 compounds...pp. 8-9
- Strategies for mitigating matrix effects in pesticide analysis of foods...pp. 10-11
- Unique selectivity of Rtx<sup>®</sup>-Dioxin2 column: Isomer specificity for toxic dioxins and

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#### Speed up Your Separations With Restek

- ▶ Run urine pain panels in under 5 minutes on new Raptor<sup>™</sup> Biphenyl LC columns...pp. 6-7

▶ New 100% CLPesticides column set and resolves in less than 7 minutes using hydrogen...pp. 10-11

▶ Run in half with our 9-minute GC semivolatiles...pp. 12-14

▶ Run 14% faster with a combined 1,4-dioxane and nitrosamines column...pp. 16-17

#### Also in this issue >>>>

- ▶ How to speedup GC separations using hydrogen...pp. 15
- ▶ Kick-start method development with Restek's EZGC<sup>™</sup> chromatogram modeler...pp. 18-19
- ▶ ADXT<sup>™</sup>-HT SimPlot columns: individual application-specific testing guarantees method performance...pp. 23

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#### Make Method Development Faster, Easier, and More Reliable with Restek

- ▶ FPP vs. SPP Raptor<sup>™</sup> LC columns—when to use which...pp. 6-7
- ▶ Switching from helium to hydrogen using the EZGC<sup>™</sup> method translator...pp. 8-9
- ▶ How to choose an inlet liner...pp. 10-11
- ▶ Phase selectivity & method development...pp. 22-23

#### ALSO IN THIS ISSUE:

- ▶ Simple sample prep and improved accuracy for PAHs in tea by GC...pp. 14-15
- ▶ High-throughput LC-MS/MS analysis of vitamin D in plasma...pp. 16-17
- ▶ Fast cannabis potency methods for LC and GC...pp. 18-19

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