the RESTEK Advantage

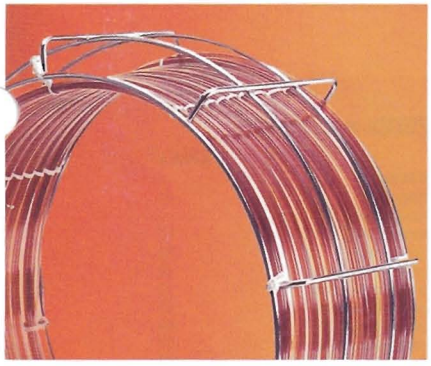
Innovators of High Resolution Chromatography Products

# Optimized Analysis of Brominated Flame Retardants

## Using an Rtx®-500 GC Capillary Column

by Frank Dorman, Ph.D., Innovations Team, Director of Technical Development

- ✔ Elutes decabromodiphenyl ether in 30 minutes.
- ✔ Low bleed for sensitive ECD and MS analyses.
- ✔ Separates other higher molecular weight compounds.



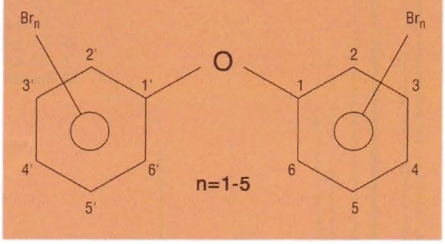
Brominated flame retardants are an emerging environmental concern that present a unique challenge to analysts. One of the most heavily used types of brominated flame retardants are the polybrominated diphenyl ethers (PBDEs). These compounds have the structure shown in Figure 1, with 1 to 10 bromines substituted on the two rings. This makes these compounds fairly heavy (up to approximately 1000amu), thus placing difficult requirements on the gas chromatographic (GC) analytical system. While the analysis may be performed using either electron capture detection (ECD) or mass spectrometric detection (MS), the compounds require a high oven temperature to elute in a reasonable amount of time. This requires an analytical GC column featuring high-temperature fused silica or metal tubing and a high-temperature stationary phase that has both low bleed and the selectivity necessary to separate the PBDE congeners.

Restek chemists, working in conjunction with Karen MacPherson and Eric Reiner at the Ontario Ministry of the Environment, have developed a new fused silica capillary GC column and analytical procedure for separating PBDE congeners in a reasonable time. The new Rtx®-500 column incorporates a carborane-stabilized polydimethylsiloxane polymer in special high-temperature fused silica tubing. The column can be heated to 380°C, and exhibits very low bleed at this extreme temperature. The column combines the stability required for separating higher molecular weight compounds with the sensitivity required for ECD or MS analysis.

PBDE congeners up to decabromodiphenyl ether (PBDE 209) are separated in less than 45 minutes using the new Rtx®-500 column in a GC-high resolution MS analysis (Figure 2). In a GC-ECD analysis of the same PBDE congeners, decabromodiphenyl ether is eluted in 30 minutes. This same system has been used for baseline separation of toxic PCB congeners and is currently under investigation for analyses of the brominated and chlorinated dioxins and furans. If you must analyze PBDEs or other high molecular weight compounds, we highly recommend the Rtx®-500 column for fast separations and reliable quantitation.

**Figure 1**

PBDEs can have high molecular weight, due to bromine substitution around the two rings.




*(contd. on page 2)*

new

in this issue



RESTEK

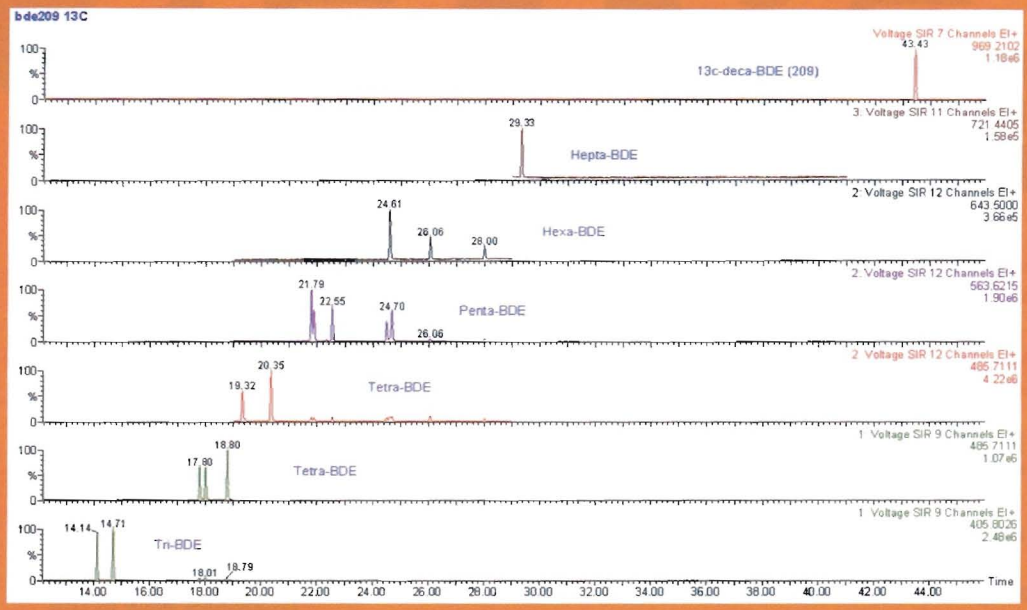
www.restekcorp.com

2002 vol. 3

IONAL

## Figure 2

The Rtx®-500 column completely separates PBDE congeners in less than 45 minutes in a GC/MS analysis.




**NB DecaBDE (last elutor) elutes at ~43 min.**

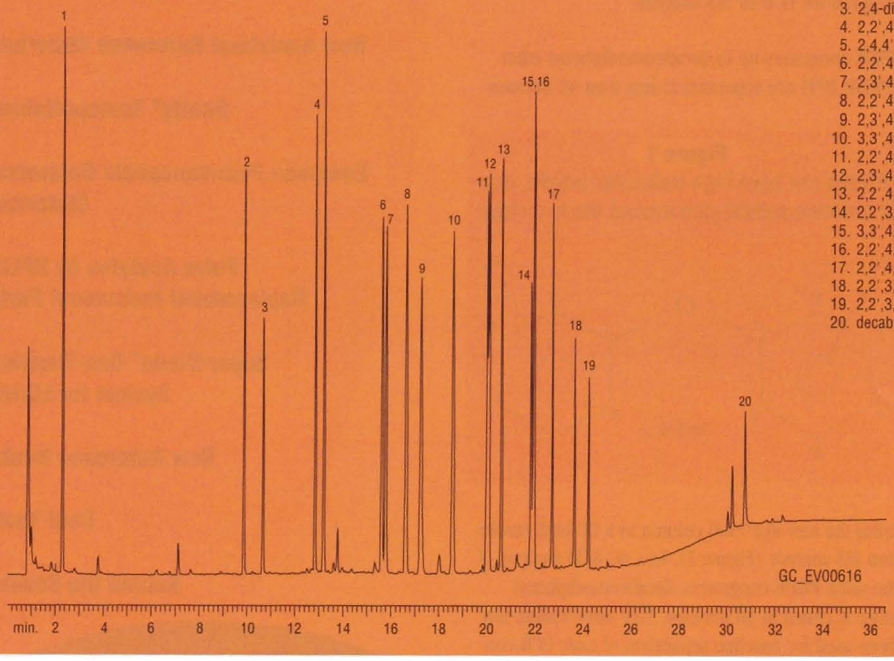
*Chromatogram courtesy of Ontario Ministry of the Environment*

*Reference materials courtesy of Wellington Laboratories, Guelph, Ontario, Canada*
*www.well-labs.com*
*US Distributor:*
*TerraChem Inc. U.S.A.*
*8600 Shawnee Mission Pkwy., Suite 305*
*Shawnee Mission, KS 66202*
*Phone: 913-722-4919*
*Toll-free: 877-809-7039*
*Fax: 913-722-4669*
*Website: http://www.terrachem.com*
*Email: info@terrachem.com*

**Column:** Rtx®-500 30m, 0.25mm, 0.15µm (cat.# 10750); **GC:** Agilent 6890+; **Sample:** Wellington Laboratories BDE Mix C (300pg/µL each in nonane); **Inj.:** 1µL splitless injection, drilled Uniliner® (cat.# 21054); **Oven temp.:** 100°C (hold 0.64 min.) to 110°C @ 10°C/min. (hold 0 min.) to 180°C @ 80°C/min. (hold 23 min.) to 350°C @ 5°C/min.; **Flow rate:** constant @ 1.5mL/min; **Injector temp.:** 300°C; **Instrument configuration:** Micromass Autospec-UltimaNT (High Resolution Mass Spectrometer); Source Temperature: 300°C

## Figure 3

When used with less expensive ECD methodology, an Rtx®-500 column resolves PBDEs in 30 minutes.




| Peak | IUPAC # |
|---|---|
| 1. 4-bromodiphenyl ether | 3 |
| 2. 4,4'-dibromodiphenyl ether | 15 |
| 3. 2,4-dibromodiphenyl ether | 7 |
| 4. 2,2',4-tribromodiphenyl ether | 17 |
| 5. 2,4,4'-tribromodiphenyl ether | 28 |
| 6. 2,2',4,5'-tetrabromodiphenyl ether | 49 |
| 7. 2,3',4',6-tetrabromodiphenyl ether | 71 |
| 8. 2,2',4,4'-tetrabromodiphenyl ether | 47 |
| 9. 2,3',4,4'-tetrabromodiphenyl ether | 66 |
| 10. 3,3',4,4'-tetrabromodiphenyl ether | 77 |
| 11. 2,2',4,4',6-pentabromodiphenyl ether | 100 |
| 12. 2,3',4,4',6-pentabromodiphenyl ether | 119 |
| 13. 2,2',4,4',5-pentabromodiphenyl ether | 99 |
| 14. 2,2',3,4,4'-pentabromodiphenyl ether | 85 |
| 15. 3,3',4,4',5-pentabromodiphenyl ether | 126 |
| 16. 2,2',4,4',5,6'-hexabromodiphenyl ether | 154 |
| 17. 2,2',4,4',5,5'-hexabromodiphenyl ether | 153 |
| 18. 2,2',3,4,4',5'-hexabromodiphenyl ether | 138 |
| 19. 2,2',3,4,4',5',6-heptabromodiphenyl ether | 183 |
| 20. decabromodiphenyl ether | 209 |

| | |
|---|---|
| **Column:** | Rtx®-500 30m, 0.53mm ID, 0.15µm (cat.# 10752) |
| **GC:** | Agilent 5890 |
| **Sample:** | Wellington Laboratories BDE-Mix C (300pg/µL each in nonane) |
| **Inj.:** | 1.0µL direct injection, drilled Uniliner® (cat.# 21054) |
| **Inj. temp.:** | 380°C |
| **Carrier gas:** | hydrogen, constant pressure |
| **Linear velocity:** | 7.69mL/min. (66.7 cm/sec.) @ 100°C |
| **Dead time:** | $CH_2Cl_2$ headspace 0.76 min. @ 100°C |
| **Make-up gas:** | 40mL/min. |
| **Oven temp.:** | 100°C (hold 1 min.) to 260°C @ 15°C (hold 5 min.) to 380°C @ 15°C (hold 15 min.) |

## Ordering Information | Rtx®-500 Columns (Fused Silica)

*(Crossbond® carborane/dimethyl polysiloxane) Stable to 380°C*

| ID | df (µm) | temp. limits | 30-Meter | 60-Meter |
|---|---|---|---|---|
| 0.25mm | 0.15 | -60°C to 380°C | 10750 | 10751 |
| 0.53mm | 0.15 | -60°C to 380°C | 10752 | |

for more info
request Applications Note 59389A.

# Vespel® Ring Inlet Seal

## Seals the First Time, Every Time

by Donna Lidgett, GC Accessories Product Marketing Manager

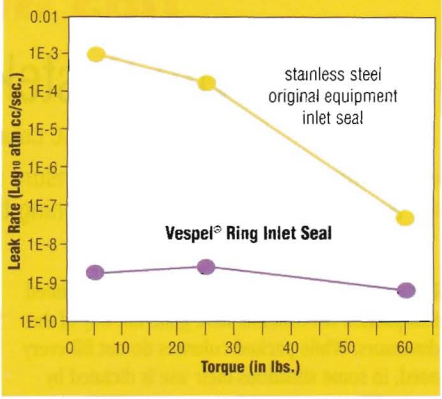
- ✔ Easy-to-use, patent-pending design makes a better seal, easily.
- ✔ Prevents oxygen from damaging your columns.
- ✔ Reduces wear on the injection port body.

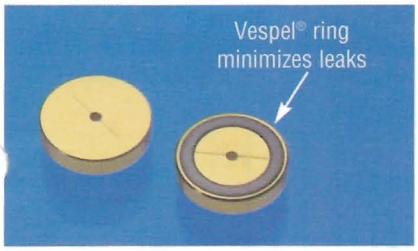
In Agilent split/splitless injection ports, it can be difficult to make and maintain a good seal with a conventional metal inlet disk. The metal-to-metal seal dictates that the analyst apply considerable torque to the reducing nut, and, based on our testing, this does not ensure a leak-tight seal. Over the course of oven temperature cycling, metal seals are prone to leaks, which ultimately can degrade the capillary column, and cause other analytical difficulties.

Our Vespel® Ring Inlet Seal greatly improves injection port performance—it seals even after repeated temperature cycles and without retightening the reducing nut! This seal features a Vespel® ring embedded into its face. This soft Vespel® ring will not harm the critical seal on the injector body, and is outside the sample flow path. Tests using a high sensitivity helium leak detector indicate the Vespel® Ring Inlet Seal seals equally effectively at torques of 5lb. or 60lb. (Figure 1).

Why trust a metal-to-metal seal when you can make leak-tight seals quickly and easily—and more reliably—with the Restek Vespel® Ring Inlet Seal? Use the stainless steel seal for analysis of unreactive compounds. To reduce breakdown and adsorption of active compounds, use the gold-plated or Silcosteel®-treated seals. The gold surface offers better inertness than standard stainless steel; Silcosteel® treatment provides inertness similar to that of fused silica capillary columns.

**Figure 1**
The Vespel® Ring Inlet Seal achieves leak-tight seals even at low torque, reducing the chance of leaks.




**Ordering Information** | Vespel® Ring Inlet Seals for Agilent 5890/6890 and 6850 GCs

| 0.8mm ID Vespel Ring Inlet Seal (washers included) | 2-pk. | 10-pk. |
|---|---|---|
| Gold-Plated | 21562 | 21563 |
| Silcosteel® | 21564 | 21565 |
| Stainless Steel | 21560 | 21561 |
| **1.2mm ID Vespel Ring Inlet Seal (washers included)** | **2-pk.** | **10-pk.** |
| Gold-Plated | 21568 | 21569 |
| Silcosteel® | 21570 | 21571 |
| Stainless Steel | 21566 | 21567 |

# A Compact, Sensitive Leak Detector For Every GC Analyst

## The Restek Leak Detective™ II

by Donna Lidgett, GC Accessories Product Marketing Manager

- ✔ Fast results—responds to trace leaks in less than 2 seconds.
- ✔ Sensitive—detects trace leaks at 1 x $10^{-4}$cc/sec.; as low as 100ppm.
- ✔ Micro-chip design improves sensitivity and response time over previous models.
- ✔ Compact, ergonomic design is easy to hold and operate with one hand.
- ✔ Battery-operated for portability (one 9 volt) instant auto-zeroing.

Gas leaks in your GC system can increase detector noise, cause baseline instability, waste carrier gas, and damage valuable analytical columns. Leak checks should be a regular part of your GC maintenance program. The new Leak Detective™ II electronic leak detector is the affordable solution for detecting gas leaks. It will identify minute gas leaks that might go undetected by liquid leak detectors.*

The Leak Detective™ II electronic leak detector incorporates micro-chip technology and a new design, to give you better sensitivity and faster response time in a more compact unit. An auto-zero feature allows you to instantly zero the leak detector with a push of a button, and the ergonomic design brings all the controls to your fingertips for easy use. The unit responds in less than two seconds to trace leaks of gases with thermal conductivities different than air. Leaks are indicated by an audible alarm, as well as by an LED readout. For easy, sensitive, and reliable leak detection, order a new Leak Detective™ II electronic leak detector today.


**Ordering Information** | Leak Detective II™

| Description | qty. | cat.# |
|---|---|---|
| Leak Detective™ II Leak Detector (9 volt, Battery-Operated) | ea. | 20413 |

**Never use liquid leak detectors on a capillary system because liquids can be drawn into the column.* ***Caution:*** *NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.*

# Packed Column Technology Has New Life

## With Restek Innovations

by Neil Mosesman, GC Columns Product Marketing Manager

- ✔ Optimized tubing and support ensure unsurpassed inertness for trace analyses.
- ✔ Bonded phases shorten conditioning times, greatly reduce bleed, and prolong column lifetimes.

For years column manufacturers have proclaimed that packed GC columns have gone the way of the dinosaurs. While packed columns do not fill every need, in some situations their use is dictated by methodology, and, for some analysts, they are the column of preference. Restek innovations have brought new life to the "expired" packed column in all three aspects of column technology: the support, the phase, and the tubing. Silcoport™ support provides unsurpassed inertness for trace analyses. Our bonded phase packings are a revolution in packed column technology. They significantly shorten conditioning times, greatly reduce column bleed, and prolong column lifetimes. SilcoSmooth™ tubing combines the inertness of glass with the durability of stainless steel.

The great sensitivity of modern detection systems and a progressing need to reduce detection limits place challenging demands on a chromatography column. Silcoport™ diatomaceous earth support is the modern solid support that we developed to meet these demands. Unlike conventional DMDCS deactivation, we use a proprietary fused silica deactivation technology and a special mixture of deactivants to ensure the greatest inertness (Figure 1) without changing the polarity of the stationary phase. Each batch of support is carefully tested to confirm a uniform particle size distribution that ensures columns with maximum efficiency.

By applying our experience in stationary phase synthesis in conjunction with our unique Silcoport™ packing deactivation process we create completely bonded packing materials. To encompass a wide range of applications, we offer Rtx®-1 and Rtx®-5, bonded methyl silicone phases, and Stabilwax®, a bonded Carbowax® phase. Each phase is completely cross-linked on Silcoport™ support. In side-by-side comparisons with conventional nonbonded methyl silicone phase columns, Rtx®-1 and Rtx®-5 columns have lower bleed, improved peak shape, and longer useful lives (Figure 2). Evaluations with an Rtx®-1 column show retention times are repeatable after only 30 minutes of conditioning.

If your analysis involves reactive compounds, you probably have used fragile, inflexible glass columns, but now you can do better. Made from ultra-smooth, seamless 304 stainless steel, and treated with our innovative Silcosteel® deactivation process, SilcoSmooth™ tubing combines the inertness of glass with the strength and flexibility of stainless steel. SilcoSmooth™ tubing can replace glass tubing in virtually any application. For analyses of ppb levels of sulfur-containing compounds, use Sulfinert™ tubing packed with Rt-XLSulfur™ packing. For undemanding applications we can make columns from conventional tubing: stainless steel, Hastelloy®, nickel, copper, or Teflon®.

In combination, Silcoport™ support, our bonded phase packings, and SilcoSmooth™ tubing make packed column GC a viable alternative in many applications in which the technique had been endangered. If you use packed columns, and think that you have to live with limitations, call us. We can provide the column that will give you the separation you need, but with convenience, inertness, and column lifetimes you never expected from a packed column.

**Figure 1**

A trace level analysis of pesticides, including labile endrin and DDT, demonstrates the inertness of Silcoport™ support.

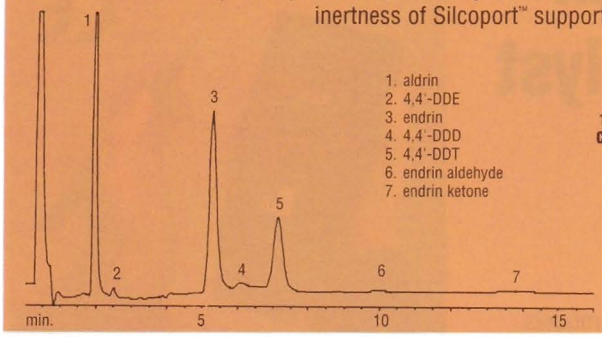
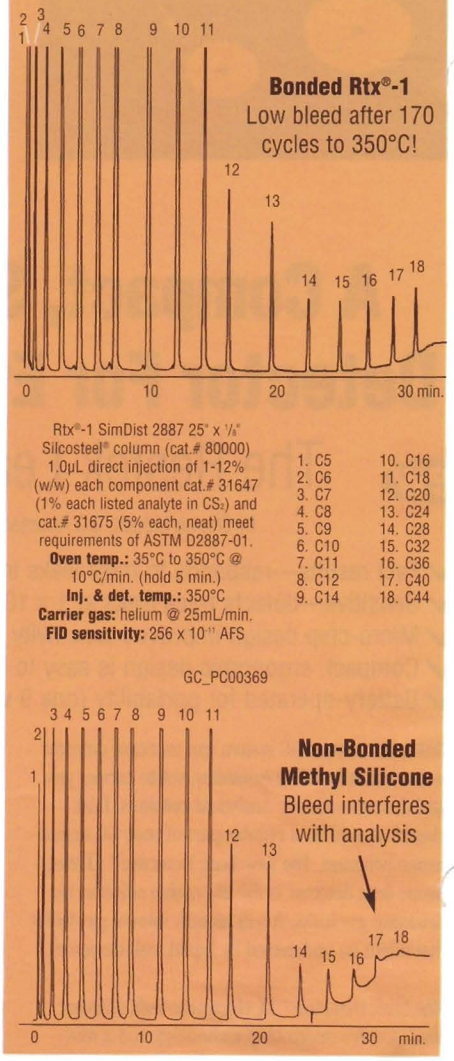



**Figure 2**

Bonded packed columns exhibit longer lifetimes than nonbonded columns.




**Ordering Information** | Bonded Packed Column Stationary Phases:

| | Stainless Steel Tubing | | | | SilcoSmooth™ Tubing | | | |
|---|---|---|---|---|---|---|---|---|
| **Liquid Phases Bonded on 100/120 Silcoport™ P** | **L (ft.)** | **OD (in.)** | **ID (mm)** | **cat.#*** | **L (m)** | **OD (in.)** | **ID (mm)** | **cat.#*** |
| 3% Rtx®-1 | 6 | ⅛ | 2.1 | 80441- | 2 | ⅛ | 2 | 80401- |
| 10% Rtx®-1 | 6 | ⅛ | 2.1 | 80442- | 2 | ⅛ | 2 | 80405- |
| 20% Rtx®-1 | 6 | ⅛ | 2.1 | 80443- | 2 | ⅛ | 2 | 80409- |
| 3% Rtx®-5 | 6 | ⅛ | 2.1 | 80444- | 2 | ⅛ | 2 | 80477- |
| 10% Rtx®-5 | 6 | ⅛ | 2.1 | 80445- | 2 | ⅛ | 2 | 80478- |
| 20% Rtx®-5 | 6 | ⅛ | 2.1 | 80446- | 2 | ⅛ | 2 | 80479- |
| 5% Rtx®-Stabilwax® | 6 | ⅛ | 2.1 | 80447- | 2 | ⅛ | 2 | 80415- |
| 10% Rtx®-Stabilwax® | 6 | ⅛ | 2.1 | 80448- | 2 | ⅛ | 2 | 80416- |
| 20% Rtx®-Stabilwax® | 6 | ⅛ | 2.1 | 80449- | 2 | ⅛ | 2 | 80417- |
| Rtx®-1 SimDist 2887 | 25* | ⅛ | 2.1 | 80450 | 25* | ⅛ | 2 | 80000 |

*Please include configuration suffix number (refer to our catalog, lit. cat.# 59662).

# Rt-Msieve™ 5A & MXT®-5A PLOT Columns

## Superior Analyses of Permanent Gases

by Neil Mosesman, GC Columns Product Marketing Manager

✔ Fast, efficient separations at above ambient temperatures.
✔ 100% bonding process eliminates the need for particle traps.
✔ Stainless steel columns for durability.

Gas-liquid chromatography (GLC), the most common mode of gas chromatography, has limited application in analyses of gases. Subambient temperatures often are required to achieve a separation, and cryogenic cooling systems are costly and inconvenient. Gas-solid chromatography (GSC), in which gaseous analytes are absorbed into the packing particles, rather than into a surface coating, is far more effective for separating gases. Difficult-to-separate small molecules, such as argon and oxygen, butene isomers, and many others, can be separated by GSC at above ambient temperatures.

Just as capillary columns offer important advantages over packed GLC columns, porous layer open tubular columns-PLOT columns-offer significant advantages over packed GSC columns. Their open design gives PLOT columns greater permeability, and their narrow diameter ensures sharper peaks. The open construction also affords a smaller pressure drop per unit length, so longer columns can be used. This means much higher column efficiency and, therefore, superior resolution. In brief, PLOT columns provide faster and more sensitive analyses than packed GSC columns.

Restek PLOT columns are especially effective for separating mixtures of gaseous analytes. Rt-Msieve™ 5A and MXT®-Msieve 5A PLOT columns contain molecular sieve 5A particles that are bonded to the inner surface of the tubing, using a proprietary process that prevents particle dislocation that could damage valves and detection systems. They are designed for fast, efficient separation of argon and oxygen, hydrogen and helium, and other permanent gases, including permanent gases admixed in refinery or natural gas. Special coating and deactivation procedures ensure chromatographic efficiency and the integrity of the porous layer bonding. Finely controlled pore size allows selective adsorption of specific target compounds, ensuring difficult separations can be made without subambient temperatures. Figure 1 shows an Rt-Msieve™ 5A column can separate oxygen from argon to baseline, at above ambient temperature, in approximately 2 min. Figure 2 shows the permanent gases resolved from methane in 4 minutes.* Stainless steel MXT®-Msieve 5A PLOT columns offer the same powerful separating capabilities as fused silica Rt-Msieve™ 5A PLOT columns, plus high resistance to physical damage and ability to be coiled to diameters as small as 3.5" (<9cm), making MXT® columns ideal for portable GCs, process control applications, and other demanding situations.

In addition to Rt-Msieve™ 5A and MXT®-Msieve 5A columns, we manufacture PLOT columns for a wide range of other applications. Rt-Alumina™ PLOT columns ($Al_2O_3$ solid phase) offer fast, reproducible performance for determining hydrocarbon purity or monitoring hydrocarbon streams. Porous polymer Rt-QPLOT™ and MXT®-QPLOT columns (nonpolar), Rt-SPLOT™ and MXT®-SPLOT columns (intermediate polarity), and Rt-UPLOT™ and MXT®-UPLOT columns (highly polar) are particularly useful for situations in which water is likely to be encountered. Applications for these columns include permanent gases at subambient temperatures, carbon dioxide and other inorganic gases, hydrocarbon mixtures, and many nonpolar, intermediate polarity, and polar solvents. For more information and example analyses on Restek PLOT columns, refer to our current chromatography products catalog or our website, or request our new PLOT column flyer (lit. cat. #59456).

If your analyses call for difficult separations of gaseous analytes, and neither conventional packed GC columns nor WCOT capillary columns are providing the separations you want, or your analyses depend on costly or time-consuming conditions, a Restek PLOT column can make your work simpler.

**Restek PLOT columns are superior for 5 reasons:**

1. Most efficient and consistent analyses.
2. No need for particle traps.
3. Reproducible quality at affordable prices.
4. Most effective phase for your separation: alumina, molecular sieve 5A, or porous polymer.
5. Fused silica columns for most applications, metal columns for exceptional durability.

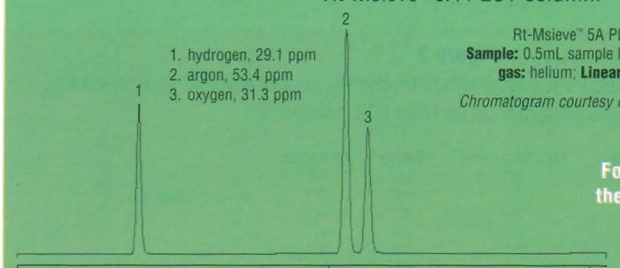
**Ordering Information** | Rt-Msieve™ 5A (Fused Silica PLOT) Temp. limit to 300°C

| ID | df (µm) | 15-Meter | 30-Meter |
|---|---|---|---|
| 0.32mm | 30 | 19720 | 19722 |
| 0.53mm | 50 | 19721 | 19723 |

**Ordering Information** | MXT®-Msieve 5A (Metal PLOT) Temp. limit to 300°C

| ID | df (µm) | 15-Meter | 30-Meter |
|---|---|---|---|
| 0.53mm | 50 | 79721 | 79723 |

**Figure 1**
Oxygen and argon resolved to baseline at above ambient temperature, using an Rt-Msieve™ 5A PLOT column.

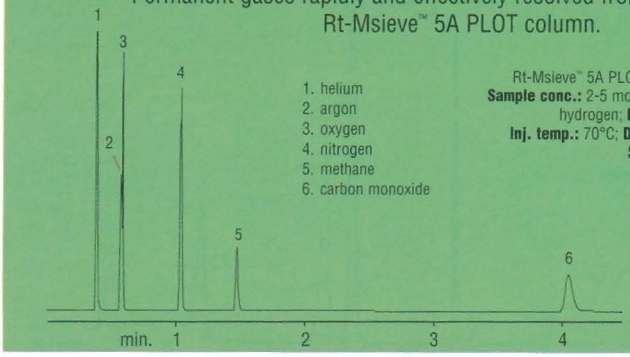



Rt-Msieve™ 5A PLOT 30m, 0.53mm ID (cat. # 19723)
**Sample:** 0.5mL sample loop injection; **Column temp.:** 27°C; **Carrier gas:** helium; **Linear velocity:** 34 cm/sec.; **Det.:** Valco HID

*Chromatogram courtesy of Larry McElmurry, Mobile Analytical Labs.*

**For gas reference standards for these applications, see page 10.**

**Figure 2**
Permanent gases rapidly and effectively resolved from methane by an Rt-Msieve™ 5A PLOT column.




Rt-Msieve™ 5A PLOT 15m, 0.32mm ID (cat. # 19720)
**Sample conc.:** 2-5 mol%; **Column temp.:** 70°C; **Carrier gas:** hydrogen; **Linear velocity:** 64cm/sec.; **Inj. temp.:** 70°C; **Det.:** HP µTCD; **Det. temp.:** 200°C ; **Sensitivity:** high

*Carbon dioxide is difficult to elute from a molecular sieve column, but is isolated easily on an Rt-QPLOT™ porous polymer column at 30°C. Request lit. cat.# 59540 for details.

# HPLC Analysis of Narcotic/ Acetaminophen Admixtures

## What to Do If a Compendium Method Doesn't Work

by Vernon Bartlett, HPLC Innovations Manager

✔ Make changes or modifications stepwise, with defined purpose in mind.
✔ When possible, create and validate a single method for a range of similar analytes.

Sometimes methods described in the United States Pharmacopoeia (USP), the European Pharmacopoeia (EP), the British Pharmacopoeia (BP), or other compendia do not provide the desired robustness in separation or reproducibility, or results barely pass system suitability requirements. Modifications can be made to improve the methodology, and the results compared statistically to the original. To improve analysis efficiency and reduce costs associated with revalidating and testing, it may be desirable to create and validate a single analytical method for a range of similar drug products.

Many narcotics are very similar in structure, often varying by only a single substitution. Morphine, codeine, hydrocodone, and oxycodone are quite similar, for example (Figure 1). Some of these closely related compounds—all but morphine, in fact—might be blended with other analgesics, such as acetaminophen (APAP). USP 25 describes more than 7 different methods to test these raw materials and admixtures; some of the older methods do not use HPLC as a primary test for purity.

**Figure 1**
Chemical structures of narcotics and acetaminophen.

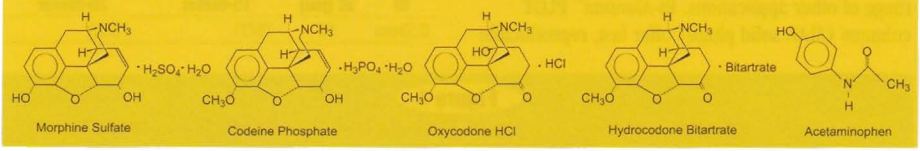


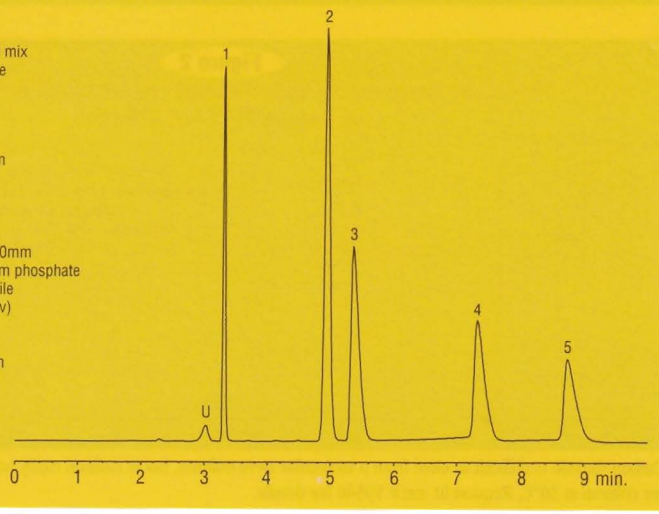

**Figure 2**
A single, modified USP procedure for separating structurally similar narcotic analgesics and acetaminophen on an Ultra C18 column.

| Peak | Conc. (µg/mL) | Ret. Time (min.) | Tailing | Resolution |
|---|---|---|---|---|
| U. unknown | unknown | 3.0 | NA | NA |
| 1. morphine sulfate | 204 | 3.3 | 0.97 | 2.3 |
| 2. acetaminophen | 92 | 5.0 | 1.1 | 14.9 |
| 3. codeine phosphate | 216 | 5.3 | 1.8 | 2.1 |
| 4. oxycodone HCl | 206 | 7.3 | 1.9 | 6.9 |
| 5. hydrocodone bitartrate | 218 | 8.8 | 1.9 | 4.1 |

**Sample:**
**Inj.:** 10µL
**Sample:** raw material mix
**Solvent:** mobile phase

**Column:** **Ultra C18**
**Catalog #:** 9174575
**Dimensions:** 250 x 4.6mm
**Particle size:** 5µm
**Pore size:** 100Å

**Conditions:**
**Mobile Phase:** A: pH 2.8 10mm potassium phosphate
B: acetonitrile
(85A:15B, v/v)
**Flow:** 1.0 mL/min.
**Temp.:** 27°C
**Det.:** UV @ 235nm




One of the chromatographic applications in USP 25 is for the analysis of oxycodone raw material. After reading the mobile phase section, we saw some potential problems with the method, including:

1) The use of methanol in this analysis could lead to high background absorption and loss of linear range, because the analytical wavelength is 206nm, and the UV cutoff for methanol is 235nm. In extreme cases this also can reduce sensitivity—the more energy the background absorbs, the less is available to the analyte.

2) An ion-pairing agent (hexane sulfonic acid) is introduced into the mobile phase without a buffer to maintain pH. This could lead to widened peaks, tailing peaks, and retention time drift.

3) Triethylamine (TEA) modifier is included in the method. When basic compounds are analyzed on older-type HPLC columns, TEA often is added as competing base, to reduce the tailing caused by acidic silanol activity. If the analytical species are neutral, or have been "neutralized" by an ion-pairing agent, TEA should have no beneficial effect. Adding TEA, a base, to a mobile phase containing sulfonic acids will cause acid/base neutralization, producing a salt and water and reducing the effective concentration of the acidic ion-pairing agent. This could lead to the formation of undesirable side products in the mobile phase that also will absorb in the low UV range, creating noisy baselines. Furthermore, TEA is volatile, and its composition might change over time if the mobile phase is sparged.

Thus, some aspects of the method appear redundant and some might actually compromise the separation. In addition, some of the reagents, such as TEA, might not be necessary for modern columns. After performing the USP 25 method as written, we made some tests to determine actual needs to achieve the system suitability requirements as specified.

With peak shape, separation, and proper analytical technique in mind, we attempted to eliminate some of the perceived problems. We realized that by using 284nm as the detection wavelength, rather than 206nm as used in USP 25, we might not see some impurities, but in real life the material should be tested against some known source for potency. (Note that with the additional reagents removed, both Ultra C8 and Pinnacle II™ C8 columns provided good results at the 206nm wavelength.)

Next we removed the ion pairing agent and the TEA. We elected to keep a 20 mM phosphate buffer system to maintain a pH of 2.5. Then we reduced the temperature from 35°C to 27°C, to determine whether the greater mass transfer and analyte solubility in the mobile phase at 35°C had been masking other potential problems.

These changes led to a slight increase in tailing for all compounds on both Ultra C8 and Pinnacle II™ C8 columns, but this was acceptable, especially because the run time for the analysis was reduced by a factor of 3 and resolution was improved by 59% to 79%. The system passed the system suitability requirements in the USP monograph.

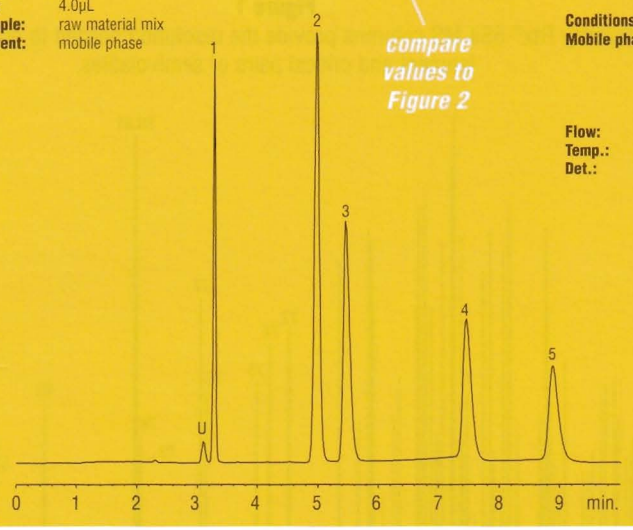
In the next experiment, we re-introduced the ion pair reagent hexane sulfonic acid into the system under the control of the pH 2.5 phosphate buffer system. The run time doubled, relative to the original procedure, demonstrating that TEA did affect the concentration of the ion-pairing agent. Reducing the concentration of ion pairing agent, or using a shorter chain length ion-pairing agent, might have been a better alternative to adding TEA. The system still passed the system suitability requirements listed by the USP, but the chromatogram was much noisier—and equilibration problems seen in the USP 25 analysis returned.

**Figure 3**

A small amount of methanol finely tunes the separation of narcotic analgesics and acetaminophen on an Ultra C18 column, as indicated by less peak tailing.

| Peak List: | Conc. (µg/mL) | Ret. Time (min.) | Tailing | Resolution |
|---|---|---|---|---|
| U. unknown | unknown | 3.1 | NA | NA |
| 1. morphine sulfate | 204 | 3.3 | 1.0 | 1.8 |
| 2. acetaminophen | 92 | 5.0 | 1.1 | 14.1 |
| 3. codeine phosphate | 216 | 5.5 | 1.4 | 2.6 |
| 4. oxycodone HCl | 206 | 7.5 | 1.4 | 8.5 |
| 5. hydrocodone bitartrate | 218 | 8.9 | 1.4 | 5.0 |

**Sample:**
**Inj.:** 4.0µL
**Sample:** raw material mix
**Solvent:** mobile phase




**Column:** Ultra C18
**Catalog #:** 9174575
**Dimensions:** 250 x 4.6mm
**Particle size:** 5µm
**Pore size:** 100Å

**Conditions:**
**Mobile phase:** A: pH 2.8 10mm potassium phosphate
B: acetonitrile: methanol (90:10 v/v) (85A:15B, v/v)
**Flow:** 1.0mL/min.
**Temp.:** 27°C
**Det.:** UV @ 235nm

## Ordering Information | Ultra C18 5µm Columns

| Length | 1.0mm ID cat.# | 2.1mm ID cat.# | 3.2mm ID cat.# | 4.0mm ID cat.# | 4.6mm ID cat.# |
|---|---|---|---|---|---|
| 30mm | 9174531 | 9174532 | 9174533 | — | 9174535 |
| 50mm | 9174551 | 9174552 | 9174553 | — | 9174555 |
| 100mm | 9174511 | 9174512 | 9174513 | 9174514 | 9174515 |
| 150mm | 9174561 | 9174562 | 9174563 | 9174564 | 9174565 |
| 200mm | 9174521 | 9174522 | 9174523 | — | 9174525 |
| 250mm | 9174571 | 9174572 | 9174573 | — | 9174575 |

After reviewing the monographs for admixtures containing structurally related narcotics and acetaminophen, we created a single separation for morphine sulfate, acetaminophen, codeine phosphate, oxycodone HCl, and hydrocodone bitartrate. The goal was to create an adequate separation while keeping the method as simple as possible. We chose an Ultra C18 column and set detection to 235nm. All components, including a small unknown peak, were separated to baseline (Figure 2).

Next, we increased the amount of buffer to 90% (a 5% increase). This simple increase doubled the analysis time. Resolution doubled between most components, with the greatest change between acetaminophen and codeine. The unknown peak disappeared and probably co-eluted with morphine.

We adjusted the mobile phase ratio to 85:15, buffer:organic solvent, using a 90:10 mixture of acetonitrile and methanol as the organic solvent. Resolution improved, relative to the original mobile phase composition, analysis again was under 10 minutes, and the unknown peak returned (Figure 3). For this analysis, these conditions provided the most desirable results.

In summary, the goal of any method should be to achieve the most stable and robust separation. Sometimes methods are more complicated than they need to be, and this can make analysis unnecessarily difficult. Even troubleshooting such methods adds to production costs. When preparing to follow a method always attempt to determine the reason a reagent would be included in a mobile phase. Any change or modification should have an established scientific purpose. By creating more universal methods for analyses of structurally related compounds, it should be possible to reduce costs for supplies, increase laboratory analysis efficiency, and reduce personnel training time.

For chromatograms illustrating the changes in separation that occur with each change in the mobile phase, please request Applications Note #59453. If you encounter problems when analyzing your samples according to an established method, our experienced Technical Service chemists will be glad to help. Contact them at 800-356-1688, ext. 4 or 814-353-1300, ext. 4, or contact your Restek representative.

# More Reliable Results From Semivolatiles Analysis

## Using Restek Columns and Standards

by Christopher English, Environmental Innovations Chemist

- ✔ Rtx®-5Sil MS columns resolve critical pairs and minimize bleed.
- ✔ Integral guard column available.
- ✔ 8270MegaMix™ minimizes mixtures needed, has maximum stability.
- ✔ Monitor all relevant semivolatiles at one detector sensitivity—8270 MegaMix™ includes 3 and 4 methylphenol at 0.5x concentrations of other components.

Complex mixtures of semivolatile organic compounds are extracted from water, soil, or solid waste samples, concentrated, and analyzed by gas chromatography. The current compound list for US EPA Method 8270D, for example, includes basic, neutral, and acidic compounds with boiling points from 150°C to 500°C. Other semivolatiles methods are similarly complex. Because these analyses encompass a broad range of compound classes and require low detection limits, and because sample extracts can include non-target contaminants, significant demand is placed on the efficiency, inertness, thermal stability, and sample capacity of the analytical column. These parameters must be optimized to provide good resolution, fast analysis times, and high sample throughput. The column must have adequate sample capacity to handle the high concentrations of contaminants sometimes found in these extracts, while exhibiting the high inertness needed for accurate quantification of target analytes down to low ng/µL levels.

Restek has designed Rtx®-5Sil MS capillary columns to address the demands of semivolatile by GC/MS. Silarylene polymer technology stiffens the siloxane chain, preventing its thermal breakdown (column bleed). The content of this aryl functionality has been adjusted to give excellent efficiency and lower bleed, compared to conventional 5% diphenyl/95%dimethyl phases; Rtx®-5Sil MS columns exhibit excellent inertness and low bleed, even at 330°C. The optimized stationary phase, proprietary deactivations, and inherently low bleed of the Rtx®-5Sil MS phase, combined with the integral guard column, overcome the problems presented by the compounds and conditions inherent to semivolatiles analysis. High column efficiency ensures the resolution needed to quantify critical pairs and structural isomers, as shown by the separation of benzo(b)- and benzo(k)fluoranthene (peaks 83/84) in Figure 1.

Rtx®-5Sil MS columns are available with an integral, deactivated 5- or 10-meter Integra-Guard™ guard column that prevents non-volatile residues from collecting in the analytical column, where they could interfere with the analytes. Made from a continuous length of tubing, innovative Integra-Guard™ columns offer the column-protecting advantages of a guard column without the potential for leaks at the interface.*

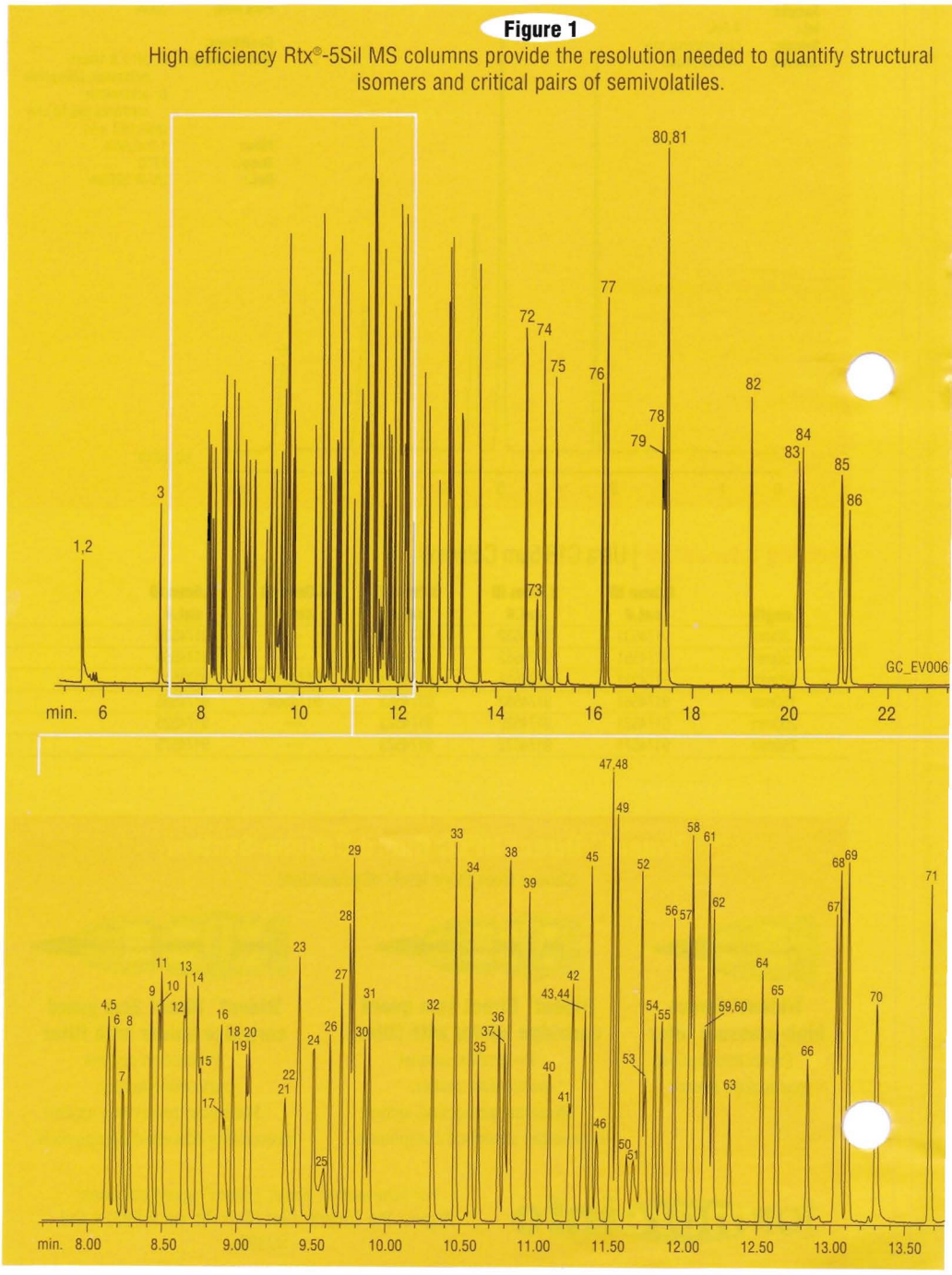
*For more information about Integra-Guard™ columns, request lit. cat.# 59441.

Our 8270 MegaMix™ eliminates mixing and minimizes preparation time for calibration and laboratory control samples—it combines all current target analytes in EPA Method 8270D. 8270 MegaMix™ components are indicated in bold in the list of analytes in Figure 1. A unique feature of this mix is the inclusion of 3-methyl- and 4-methylphenol at 0.5x the concentration of the other components, so you won't have to adjust reporting limits when analyzing for these compounds. A long shelf life for unopened ampuls of 8270 MegaMix™ minimizes ordering and inventory problems.

If you are monitoring semivolatile analytes according to US EPA Method 8270D, or similar methods, trust Restek Rtx®-5Sil MS columns and 8270 MegaMix™ to help you obtain reliable, consistent results.

**Figure 1**

High efficiency Rtx®-5Sil MS columns provide the resolution needed to quantify structural isomers and critical pairs of semivolatiles.



1. **N-nitrosodimethylamine**
2. **pyridine**
3. 2-fluorophenol
4. phenol-d6
5. **phenol**
6. **aniline**
7. **bis(2-chloroethyl)ether**
8. **2-chlorophenol**
9. **1,3-dichlorobenzene**
10. 1,4-dichlorobenzene-d4
11. **1,4-dichlorobenzene**
12. **benzyl alcohol**
13. **1,2-dichlorobenzene**
14. **2-methylphenol**
15. **bis(2-chloroisopropyl)ether**
16. **4-methylphenol/ 3-methylphenol***
17. **N-nitroso-di-n-propylamine**
18. **hexachloroethane**
19. nitrobenzene-d5
20. **nitrobenzene**
21. **isophorone**
22. **2-nitrophenol**
23. **2,4-dimethylphenol**
24. **bis(2-chloroethoxy)methane**
25. benzoic acid
26. **2,4-dichlorophenol**
27. **1,2,4-trichlorobenzene**
28. naphthalene-d8
29. **naphthalene**
30. **4-chloroanaline**
31. **hexachlorobutadiene**
32. **4-chloro-3-methylphenol**
33. **2-methylnaphthalene**
34. **1-methylnaphthalene**
35. **hexachlorocyclopenta-diene**
36. **2,4,6-trichlorophenol**
37. **2,4,5-trichlorophenol**
38. 2-fluorobiphenyl
39. **2-chloronaphthalene**
40. **2-nitroaniline**
41. **1,4-dinitrobenzene**
42. **dimethylphthalate**
43. **1,3-dinitrobenzene**
44. **2,6-dinitrotoluene**
45. **acenaphthylene**
46. **1,2-dinitrobenzene**
47. **3-nitroaniline**
48. acenaphthene-d10
49. **acenaphthene**
50. **2,4-dinitrophenol**
51. **4-nitrophenol**
52. **dibenzofuran**
53. **2,4-dinitrotoluene**
54. **2,3,4,6-tetrachlorophenol**
55. **2,3,5,6-tetrachlorophenol**
56. **diethyl phthalate**
57. **4-chlorophenyl phenyl ether**
58. **fluorene**
59. **4-nitroaniline**
60. **4,6-dinitro-2-methylphenol**
61. **diphenylamine****
62. **azobenzene*****
63. 2,4,6-tribromophenol
64. **4-bromophenyl phenyl ether**
65. **hexachlorobenzene**
66. **pentachlorophenol**
67. phenanthrene-d10
68. **phenanthrene**
69. **anthracene**
70. **carbazole**
71. **di-*n*-butylphthalate**
72. **fluoranthene**
73. benzidine
74. **pyrene**
75. *p*-terphenyl-d14
76. **butyl benzyl phthalate**
77. **bis(2-ethylhexyl)adipate**
78. **benzo(a)anthracene**
79. chrysene-d12
80. **chrysene**
81. **bis(2-ethylhexyl)phthalate**
82. **di-*n*-octyl phthalate**
83. **benzo(b)fluoranthene**
84. **benzo(k)fluoranthene**
85. **benzo(a)pyrene**
86. perylene-d12
87. **indeno(1,2,3-cd)pyrene**
88. **dibenzo(a,h)anthracene**
89. **benzo(ghi)perylene**

**Bold indicates a component of the 8270D MegaMix™**


| | |
|---|---|
| **Column:** | Rtx®-5Sil MS w/ 5-meter Integra-Guard™ 30m, 0.25mm ID, 0.25µm (cat.# 12723-124) |
| **Instrument:** | Agilent 5973 GC/MS |
| **Sample:** | US EPA Method 8270D Mix 1µL, 16 ppm each component (16ng on column) 8270 MegaMix™ (cat.# 31686) Benzoic Acid Standard (cat.# 31415) Benzidine Standard (cat.# 31441) Acid Surrogate Mix (4/89 SOW)(cat.# 31063) B/N Surrogate Standard Mix (4/89 SOW) (cat.# 31062) SV Internal Standard Mix (cat.# 31006) |
| **Solvent:** | dichloromethane |
| **Inj.:** | 1.0µL splitless (hold 0.3 min.), 4mm Drilled Uniliner® (cat.# 21055) |
| **Inj. temp.:** | 300°C |
| **Carrier gas:** | helium, constant flow |
| **Flow rate:** | 1.1mL/min. |
| **Dead Time:** | 1.8 minutes @ 35°C |
| **Oven temp.:** | 35°C (hold 4 min.) to 245°C @ 25°C/min. (no hold) to 330°C @ 6°C/min.(hold 3 min.) |
| **Det:** | GC/MS |
| **Transfer line temp.:** | 280°C |
| **Scan range:** | 35–550 amu |
| **Solvent Delay:** | 5 min. |
| **Tune:** | DFTPP |
| **Ionization:** | EI |

**Each at 0.5x concentration of other components.*

***N-nitrosodiphenylamine (8270-listed analyte) decomposes to diphenylamine (mix component).*

****1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).*

GC_EV00629

4.00

## Ordering Information | Rtx®-5Sil MS Columns (Fused Silica)

*(Equivalent selectivity of Crossbond 5% diphenyl/95% dimethyl polysiloxane) Stable to 360°C*

| 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 |

### 8270 Matrix Spike Mix (76 components)

See **bold** compounds in Figure 1 peak list.

*200µg/mL each (except noted) in methanol:methylene chloride:benzene (80:15:5), 5mL/ampul*

| Each | 5-pk. | 10-pk. |
|---|---|---|
| 31687 | 31687-510 | — |
| | with data pack | |
| 31687-500 | 31687-520 | 31787 |

### 8270 MegaMix™ (76 components)

See **bold** compounds in Figure 1 peak list.

*1,000µg/mL each (except noted) in methylene chloride:benzene (75:25), 1mL/ampul*

| Each | 5-pk. | 10-pk. |
|---|---|---|
| 31686 | 31686-510 | — |
| | with data pack | |
| 31686-500 | 31686-520 | 31786 |

# New Analytical Reference Materials

## FAMEs, Acetates, BTEX, Glycols

### Food Industry FAME Mix (37 components)

- Includes *trans* FAMEs.

| Chain | % by Weight |
|---|---|
| C4:0 | 4.0 |
| C6:0 | 4.0 |
| C8:0 | 4.0 |
| C10:0 | 4.0 |
| C11:0 | 2.0 |
| C12:0 | 4.0 |
| C13: | 2.0 |
| C14:0 | 4.0 |
| C14:1(*cis*-9) | 2.0 |
| C15:0 | 2.0 |
| C15:1(*cis*-10) | 2.0 |
| C16:0 | 6.0 |
| C16:1(*cis*-9) | 2.0 |
| C17:0 | 2.0 |
| C17:1(*cis*-10) | 2.0 |
| C18:0 | 4.0 |
| C18:1(*trans*-9) | 2.0 |
| C18:1(*cis*-9) | 4.0 |
| C18:2(all-*trans*-9.12) | 2.0 |
| C18:2(all-*cis*-9,12) | 2.0 |
| C18:3(all-*cis* 6,9,12) | 2.0 |
| C18:3(all-*cis* 9,12,15) | 2.0 |
| C20:0 | 4.0 |
| C20:1(*cis*-11) | 2.0 |
| C20:2(all-*cis* 11,14) | 2.0 |
| C20:3 (all-*cis* 8,11,14) | 2.0 |
| C20:3(all-*cis* 11,14,17) | 2.0 |
| C20:4(all-*cis* 5,8,11,14) | 2.0 |
| C20:5(all-*cis* 5,8,11,14,17) | 2.0 |
| C21:0 | 2.0 |
| C22:0 | 4.0 |
| C22:1(*cis* 13) | 2.0 |
| C22:2(all-*cis* 13,16) | 2.0 |
| 22:6(all-*cis* 4,7,10,13,16,19) | 2.0 |
| C23:0 | 2.0 |
| C24:0 | 4.0 |
| C24:1(*cis*-15) | 2.0 |

*30mg/mL in methylene chloride, 1mL/ampul*

| ea. |
|---|
| 35077 |

### 8260B Acetate Mix (7 components)

- Includes methyl acetate and n-amyl acetate.

*n*-amyl acetate — methyl acetate
butyl acetate — propyl acetate
ethyl acetate — vinyl acetate
isopropyl acetate

*2,000µg/mL each in P&T methanol, 1mL/ampul*

| | Each | 5-pk. | 10-pk. |
|---|---|---|---|
| | 30489 | 30489-510 | |
| w/data pack | 30489-500 | 30489-520 | 30589 |

### BTEX Standard (6 components)

- m- and p-xylene at 1/2 concentration.
- Contact Restek for future formulations.

benzene — *m*-xylene*
ethylbenzene — *o*-xylene
toluene — *p*-xylene*

*2,000µg/mL each in P&T methanol, 1mL/ampul*

| | Each | 5-pk. | 10-pk. |
|---|---|---|---|
| | 30488 | 30488-510 | |
| w/data pack | 30488-500 | 30488-520 | 30588 |

*1,000µg/mL

### Glycols Standard

- Assay for de-icing compounds.

ethylene glycol — propylene glycol

*50,000µg/mL each in DI water, 1mL/ampul*

| | Each | 5-pk. | 10-pk. |
|---|---|---|---|
| | 30471 | 30471-510 | |
| w/data pack | 30471-500 | 30471-520 | 30571 |

# Scotty® Transportables

## For Laboratory or Field Application

by Donna Lidgett, Air Monitoring Product Marketing Manager

- ✔ Portability makes your job easier.
- ✔ 4-Liter, 14-liter, and 48-liter sizes.
- ✔ Long shelf life.

Restek now offers a broad selection of Scotty® Transportables, ranging from pure gases to multi-component mixes. These standards have found many uses in the analytical lab, including purging, calibrating, and peak identification.

We offer three sizes to choose from. The 4-liter container has a delivery tube for purge gas or connection to sample loop or bag. The 14-liter container has a CGA 160 fitting for connection to an analytical system. The 48-liter cylinder has a CGA 165 connection, and can deliver large volumes of sample.


### Regulators for Scott Transportable Gases

**Specifications**
Maximum Inlet Pressure: 300psig
Outlet Pressure Range: 2–10psig
Operating Temperature Range: 35°F to 150°F (2°C to 65°C)
Outlet Connection: ¼" Female NPT

**Materials of Construction**
Body: Brass
Diaphragm: Viton®
Seat: Acetal
Seal: Viton®

| Description | qty. | cat.# |
|---|---|---|
| Regulator with CGA 160 inlet connection for 14L Scott container | ea. | 22690 |
| Regulator with CGA 165 inlet connection for 48L Scott container | ea. | 22691 |

| Description | Shelf Life | Scotty 4 (4 Liter) cat.# | Scotty 14 (14 Liter) cat.# | Scotty 48 (48 Liter) cat.# |
|---|---|---|---|---|
| **Pure Gases** | | | | |
| Air, zero (THC <1ppm) | 2 yrs. | 34447 | 34448 | 34449 |
| Argon, 99.995% | 2 yrs. | 34456 | 34457 | — |
| Carbon dioxide, 99.80% | 2 yrs. | 34450 | 34451 | 34452 |
| Hydrogen, 99.99% | 2 yrs. | — | 34453 | — |
| Methane, 99.00% | 2 yrs. | — | 34454 | — |
| Oxygen, 99.60% | 2 yrs. | — | 34455 | — |
| **Two-Component Mixtures** | | | | |
| Benzene in air (1ppm) | 1 yr. | — | — | 34458 |
| Benzene in air (100ppm) | 1 yr. | — | — | 34459 |
| 1,3-Butadiene in nitrogen (10ppm) | 2 yrs. | — | 34460 | 34461 |
| Carbon dioxide in helium (100ppm) | 2 yrs. | — | 34462 | — |
| Carbon dioxide in nitrogen (100ppm) | 2 yrs. | — | 34463 | 34464 |
| Carbon dioxide in nitrogen (1000ppm) | 2 yrs. | — | 34465 | 34466 |
| Ethylene in air (8-10ppm) | 2 yrs. | — | 34467 | 34468 |
| Ethylene in helium (100ppm) | 2 yrs. | — | 34489 | — |
| Hydrogen in helium (100ppm) | 2 yrs. | — | 34469 | — |
| Hydrogen in nitrogen (1%) | 2 yrs. | 34470 | 34471 | 34472 |
| Hydrogen in nitrogen (100ppm) | 2 yrs. | — | 34473 | 34474 |
| Methane in helium (100ppm) | 2 yrs. | 34475 | 34476 | 34477 |
| Methane in nitrogen (100ppm) | 2 yrs. | — | 34478 | — |
| Methane in nitrogen (1%) | 2 yrs. | 34481 | 34482 | 34483 |
| Nitrogen in helium (100ppm) | 2 yrs. | — | 34479 | — |
| Nitrous oxide in nitrogen (1ppm) | 2 yrs. | — | 34484 | 34485 |
| Oxygen in helium (100ppm) | 2 yrs. | — | 34480 | — |
| Oxygen in nitrogen (2%) | 2 yrs. | 34486 | 34487 | 34488 |
| Oxygen in nitrogen (6%) | 2 yrs. | 34490 | 34491 | 34492 |
| 1,1,1-Trichloroethane in nitrogen (10ppm) | 2 yrs. | — | — | 34493 |
| Trichloroethylene in nitrogen (10ppm) | 2 yrs. | — | 34494 | 34495 |
| Vinyl chloride in nitrogen (1ppm) | 2 yrs. | — | 34496 | 34497 |
| Vinyl chloride in nitrogen (10ppm) | 2 yrs. | — | 34498 | 34499 |
| Vinyl chloride in nitrogen (50ppm) | 2 yrs. | — | 34500 | — |
| Vinyl chloride in nitrogen (100ppm) | 2 yrs. | — | 34501 | — |
| Vinyl chloride in nitrogen (1000ppm) | 2 yrs. | — | 34502 | — |
| **Multi-Component Mixtures** | | | | |
| Carbon monoxide, carbon dioxide, hydrogen and oxygen in nitrogen (0.5% each) | 2 yrs. | 34503 | 34504 | 34505 |
| Carbon monoxide, carbon dioxide, hydrogen and oxygen in nitrogen (1% each) | 2 yrs. | 34506 | 34507 | 34508 |
| Carbon monoxide, carbon dioxide, methane, ethane, ethylene and acetylene in nitrogen (1% each) | 1 yr. | 34509 | 34510 | 34511 |
| Carbon monoxide, carbon dioxide, nitrogen, and oxygen, (5% each) and methane and hydrogen (4% each) in helium | 2 yrs. | — | 34512 | — |
| Carbon monoxide (7%), carbon dioxide (15%) and oxygen (5%) in nitrogen | 2 yrs. | 34513 | 34514 | — |
| Carbon monoxide (7%), oxygen (7%), carbon dioxide (15%) and methane (4.5%) in nitrogen | 2 yrs. | — | 34515 | 34516 |
| C1-C6 *n*-Paraffins: methane, ethane, propane, butane, pentane, hexane in nitrogen (15ppm each) | 2 yrs. | 34517 | 34518 | 34519 |
| C1-C6 *n*-Paraffins: methane, ethane, propane, butane, pentane, hexane in helium (100ppm each) | 2 yrs. | 34520 | 34521 | 34522 |
| C1-C6 *n*-Paraffins: methane, ethane, propane, butane, pentane, hexane in helium (1000ppm each) | 2 yrs. | 34523 | 34524 | 34525 |
| C1-C6 *n*-Paraffins: methane, ethane, propane, butane, pentane, hexane in nitrogen (100ppm each) | 2 yrs. | 34526 | 34527 | 34528 |
| C2-C4 Alkynes: acetylene, propylene, 1-butylene, 2-butylene in nitrogen (15ppm each) | 2 yrs. | 34535 | — | — |
| C2-C6 Olefins: ethylene, propylene, 1-butene, 1-pentene, 1-hexene in helium (100ppm each) | 2 yrs. | — | 34529 | 34530 |
| C2-C6 Olefins: ethylene, propylene, 1-butene, 1-pentene, 1-hexene in nitrogen (100ppm each) | 2 yrs. | — | 34531 | 34532 |
| Branched Paraffins: 2,2-dimethylbutane, 2,2-dimethylpropane, iso-butane, 2-methylbutane, 2-methylpentane, 3-methylpentane in nitrogen (15ppm each) | 2 yrs. | 34533 | 34534 | — |
| Methane, ethane, ethylene, acetylene, propane, propylene, *n*-butane in nitrogen (15ppm each) | 1 yr. | 34536 | — | 34537 |
| *n*-butane, iso-butane, *cis*-2-butene, *trans*-2-butene, 1-butene, iso-butylene, 1,3-butadiene, ethyl acetylene in nitrogen (15ppm each) | 1 yr. | 34538 | — | 34539 |

# European Pharmacopoeia Reference Materials

## Formulated for ICH Solvent Guidelines

by Ken Herwehe, Analytical Reference Materials Marketing Manager

✔ All ICH Class 1 and Class 2 solvents included.
✔ Class 2 solvents separated into 3 mixtures for less complex chromatograms.

The analysis of residual solvents in pharmaceutical products has changed, particularly for products being sold into Europe. The International Conference on Harmonization (ICH) Guidelines for Residual Solvents is becoming the international standard and is being adopted by more Pharmacopoeias, including the United States Pharmacopoeia, every year.

The ICH method and compound list is more extensive than any method previously used and poses new challenges. Compounds in Class 1 are solvents considered to be of highest risk and to be avoided in pharmaceutical manufacturing. Use of Class 2 compounds is to be limited, as they pose a lower, but present, threat to health. Compounds in Class 3 pose the lowest toxic potential and may be used routinely in manufacturing.

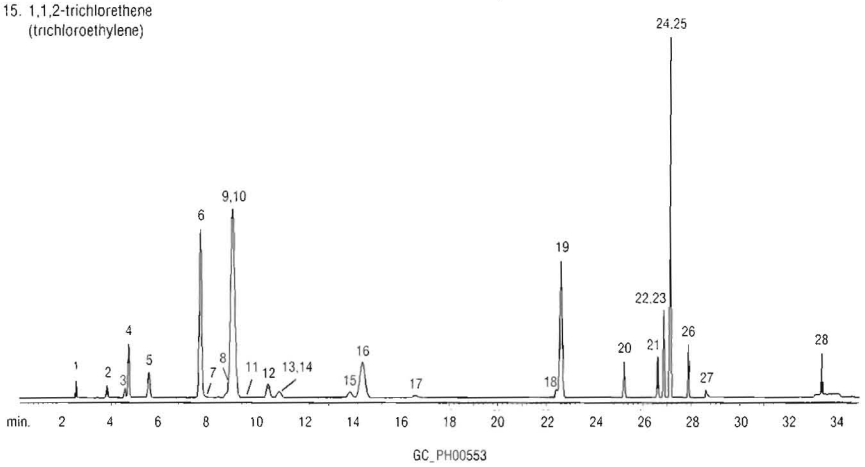
Restek manufactures Rtx®-1301 (cat.# 16085) and Stabilwax® (cat.# 10640) capillary columns and formulated these analytical reference materials to meet the new requirements.* Please request cat.# 59107, *European Pharmacopoeia Analysis of Residual Solvents* for more information.

**Figure 1**

Excellent resolution of most European Pharmacopoeia Class 1 and Class 2 compounds at the regulation limit concentration, using an Rtx®-1301 column.

1. methanol
2. 1,1-dichloroethene
3. acetonitrile
4. dichloromethane (methylene chloride)
5. hexane (C6)
6. *cis*-1,2-dichloroethene
7. nitromethane
8. chloroform
9. cyclohexane
10. 1,1,1-trichloroethane
11. carbon tetrachloride
12. benzene
13. 1,2-dimethoxyethane
14. 1,2-dichloroethane
15. 1,1,2-trichlorethene (trichloroethylene)
16. methylcyclohexane
17. 1,4-dioxane
18. pyridine
19. toluene
20. 2-hexanone
21. chlorobenzene
22. DMF
23. ethylbenzene
24. *m*-xylene
25. *p*-xylene
26. *o*-xylene
27. N,N-dimethylacetamide
28. 1,2,3,4-tetrahydronaphthalene

**Column:** 30m, 0.53mm ID x 3.0µm Rtx®-1301 (cat.# 16085)
**Inj:** Headspace injection of 28 Class 1 and 2 residual solvents for pharmaceutical processing. Prepared at the regulatory limit concentration, using samples shaken and heated at 80°C for 15 minutes. 1mL headspace injection.
**Oven temp.:** 40°C (hold 20 min.) to 240°C @ 10°C/min. (hold 20 min.)
**Inj./det. temp.:** 200°C/250°C
**FID sensitivity:** 1.1 x $10^{-11}$ AFS
**Carrier gas:** hydrogen @ 35cm/sec.
**Split ratio:** 2:1




Restek reference materials are formulated to correspond to the permissable daily exposure.

### European Pharmacopoeia/ ICH Class 1 Mix

| | |
|---|---|
| benzene | 2µg/mL |
| carbon tetrachloride | 4 |
| 1,2-dichloroethane | 5 |
| 1,1-dichloroethene | 8 |
| 1,1,1-trichloroethane | 1500 |

*In water:dimethylsulfoxide (90:10), 1mL/ampul*

| Each | 5-pk. | 10-pk. |
|---|---|---|
| 36228 | 36228-510 | 36328 |

### European Pharmacopoeia/ ICH Class 2 Mix A

**(13 components)**

| | |
|---|---|
| chlorobenzene | 360µg/mL |
| cyclohexane | 3,880 |
| *cis*-1,2-dichloroethene | 1,870 |
| dichloromethane | 600 |
| N,N-dimethylformamide | 880 |
| ethylbenzene | 369 |
| hexane | 290 |
| methylcyclohexane | 1,180 |
| toluene | 890 |
| 1,1,2-trichloroethene | 80 |
| *m*-xylene | 1,302 |
| *o*-xylene | 195 |
| *p*-xylene | 304 |

*In dimethylsulfoxide, 1mL/ampul*

| Each | 5-pk. | 10-pk. |
|---|---|---|
| 36229 | 36229-510 | 36329 |

### European Pharmacopoeia/ ICH Class 2 Mix B

**(10 components)**

| | |
|---|---|
| acetonitrile | 410µg/mL |
| chloroform | 60 |
| 1,2-dimethoxyethane | 100 |
| N,N-dimethylacetamide | 1,090 |
| 1,4-dioxane | 380 |
| 2-hexanone | 50 |
| methanol | 3,000 |
| nitromethane | 50 |
| pyridine | 200 |
| 1,2,3,4-tetrahydronaphthalene (tetraline) | 100 |

*In water:dimethylsulfoxide (90:10), 1mL/ampul*

| Each | 5-pk. | 10-pk. |
|---|---|---|
| 36230 | 36230-510 | 36330 |

### European Pharmacopoeia/ ICH Class 2 Mix C

| | |
|---|---|
| 2-ethoxyethanol | 160µg/mL |
| ethylene glycol | 620 |
| formamide | 220 |
| 2-methoxyethanol | 50 |
| N-methylpyrrolidone | 4,840 |
| sulfolane | 160 |

*In water, 1mL/ampul*

| Each | 5-pk. | 10-pk. |
|---|---|---|
| 36231 | 36231-510 | 36331 |

# Enhanced Retention of Polar Analytes by HPLC

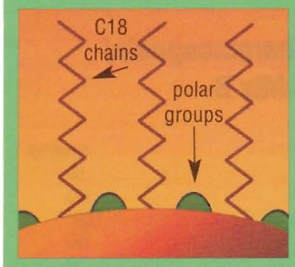
## Using the New Ultra Aqueous C18 Column

by Terrence Reid, HPLC Applications Team Chemist

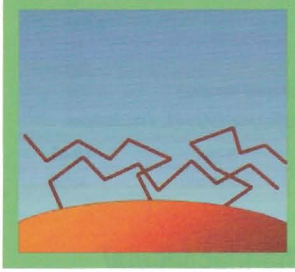
✔ Excellent peak shape for basic analytes.
✔ Compatible with 100% aqueous to 100% organic mobile phases.
✔ Compatible with MS detection.

The newest addition to our selection of HPLC columns, the Ultra Aqueous C18 column, is designed to enhance the retention of polar compounds by reversed phase HPLC. The Ultra Aqueous C18 stationary phase is a true C18 chain (USP L1), but it is immobilized on the silica surface through a unique chemistry that creates polar groups on the silica surface, between the C18 chains (Figure 1). This secondary polar character has several benefits. First, polar analytes that are insufficiently retained on a conventional C18 column interact with the polar groups in an Ultra Aqueous C18 column, producing enhanced retention. Second, the polar groups aid the retention of polar compounds by keeping the stationary phase completely wetted, even in 100% aqueous mobile phases. In theory, eliminating organic solvent from the mobile phase should maximize retention in reversed phase HPLC, and this is true for Ultra Aqueous C18 columns. In contrast, many conventional C18 columns lose ability to retain analytes in highly aqueous mobile phases because the C18 chains self-associate or fold down on the silica (Figure 1), a phenomenon sometimes referred to as chain folding. Third, the polar groups on the Ultra Aqueous C18 stationary phase shield analytes from active silanol sites on the silica surface, ensuring excellent peak shape for basic analytes (Figure 2).

**Figure 1**
An Ultra Aqueous C18 column is compatible with mobile phases from 100% organic content to 100% aqueous.




Ultra Aqueous C18 phase is stable in highly organic or highly aqueous mobile phase



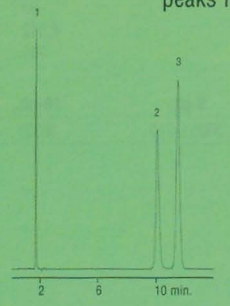
Conventional C18 chains collapse and lose capacity for retention in highly aqueous mobile phases

Although they were designed to be used with highly aqueous mobile phases, Ultra Aqueous C18 columns also are completely compatible with highly organic mobile phases. The ability to cover the full range of mobile phase composition, from 100% aqueous to 100% organic, is useful for developing gradient methods for analyzing samples containing both highly polar and nonpolar analytes. For example, Figure 3 shows water-soluble vitamins are eluted from an Ultra Aqueous C18 column with excellent resolution and as sharp, symmetric peaks.

Ultra Aqueous C18 columns also are compatible with MS detection-the minimal noise generated by an Ultra Aqueous C18 column is comparable to background in a blank analysis with no column in line (Figure 4). LC/MS is the analytical approach for a steadily increasing variety of analytes, and the versatile Ultra Aqueous C18 column is an obvious column choice.

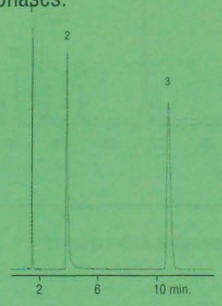
If you are analyzing samples containing polar analytes, or mixtures of polar and nonpolar analytes, and are contending with unsatisfactory resolution, insufficient retention of polar compounds, poorly shaped peaks, and/or complicated mobile phases, an Ultra Aqueous C18 column can be the solution to your problems.

**Figure 2**
Silanol-shielding polar groups on the Ultra Aqueous C18 stationary phase ensure symmetric peaks for basic analytes, compared to other C18 phases.




| | |
|---|---|
| 1. uracil | 5.0µg/mL |
| 2. pyridine | 0.1µL/mL |
| 3. phenol | 1.86mg/mL |

**Sample:** 5µL
**Mobile Phase:** 20mM potassium phosphate pH 7.0: acetonitrile (80:20)
**Flow:** 1.0mL/min.
**Temp:** 25°C
**Det.:** UV @ 254nm




**Column:** Ultra Aqueous C18 (cat.# 9178565)
**Dimensions:** 150 x 4.6mm; **Particle Size:** 5µm
**Pore Size:** 100Å

LC_0143

**Column:** Base-deactivated C18
**Dimensions:** 150 x 4.6mm; **Particle Size:** 5µm
**Pore Size:** 100Å

**Figure 3**
Excellent resolution and peak shapes for water-soluble vitamins eluted from an Ultra Aqueous C18 column.


| Peak | Conc. (mg/mL) |
|---|---|
| 1. thiamin (B1) | 250 |
| 2. ascorbic acid (C) | 1000 |
| 3. unknown | n/a |
| 4. nicotinic acid (B3) | 1000 |
| 5. unknown | n/a |
| 6. pantothenic acid (B5) | 1000 |
| 7. folic acid (B9) | 500 |
| 8. riboflavin (B2) | 250 |
| 9. methyl paraben | 0.2 |

**Sample:** Analytes in water; initial dilutions of B1 and B2 basified with ammonium hydroxide
**Column:** Ultra Aqueous C18 (cat.# 9178575 )
**Dimensions:** 250 x 4.6mm
**Particle size:** 5µm
**Pore size:** 100Å

**Mobile Phase:** A: 25mM potassium phosphate, pH 2.00: methanol (95:5, v/v)
B: methanol:25mM potassium phosphate, pH 3.5 (60:40, v/v)

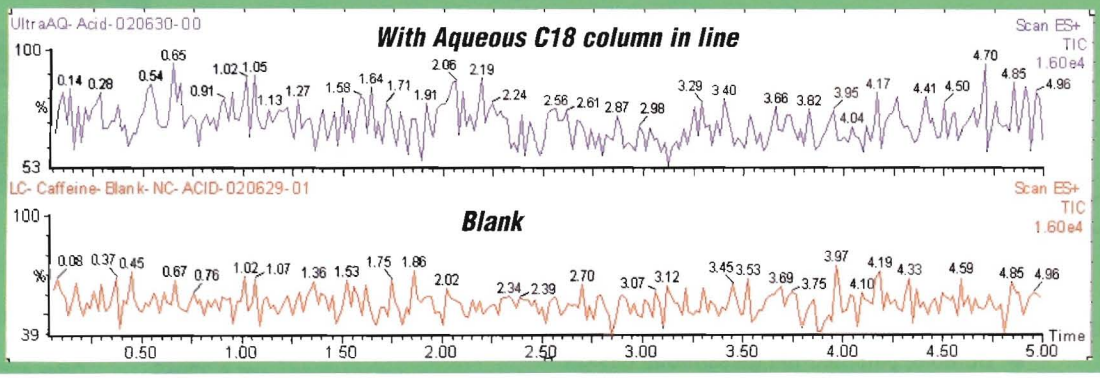
| Time (min.) | %B |
|---|---|
| 0-6 | Hold 0 |
| 6.01 | Step to 25 |
| 6.01-11 | 25-100 |
| 11-16 | Hold 100 |

**Flow:** 1.0mL/min.
**Temp.:** 27°C
**Det.:** UV @ 254nm

LC_0141

**Figure 4**

Minimal background makes Ultra Aqueous C18 columns ideal for LC/MS.




## Ordering Information | Ultra Aqueous C18 5µm Columns

| 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 |

### FREE HPLC poster!

This helpful poster features step-by-step instructions on HPLC setup and storage and many useful reference charts. Request lit. cat.# 59894.

# Replacement Parts for Waters HPLC Instruments

## Designed to Meet Original Specifications

by Greg France, HPLC Product Marketing Manager

Our replacement parts for Waters, Agilent, and Shimadzu instruments are built to meet or exceed the original equipment manufacturers' (OEM) specifications. To ensure instrument compatibility and the quality you expect from an OEM product, we obtain many of the components from the same suppliers the OEMs use. Currently we offer detector, autosampler, and pump parts. As we learn what additional needs you have we will continue to expand this line. Listed here is a partial selection of Waters parts. For other Waters parts, or for Agilent or Shimadzu parts, please visit our website or call customer service or HPLC technical service. If we don't stock what you need, just ask-we'll do our best to get it for you.

### HPLC Piston Seal Insertion Tool

- Simplify your pump maintenance
- One end removes old piston seal, other easily and securely installs new seal

| Description | qty. | cat.# |
|---|---|---|
| HPLC Piston Seal Insertion Tool | ea. | 21356 |

| Description | Waters Instrument Model # | Waters™ part # | Equivalent Restek Part qty. | cat.# |
|---|---|---|---|---|
| Inlet Check Valve Assembly | M6KA, 501, 510, 515, 590, 600E | 33679, 25214 | ea. | 25360 |
| Inlet Check Valve Housing | M6KA, 501, 510, 515, 590, 600E | 25203 | ea. | 25361 |
| Inlet Check Valve Rebuild Kit | M6KA, 501, 510, 515, 590, 600E | 60495 | 2-pk. | 25362 |
| Outlet Check Valve Assembly (Actuator Style) | M6KA, 501, 510, 515, 590, 600E | 25030 | ea. | 25363 |
| Outlet Check Valve Housing (Actuator Style) | M6KA, 501, 510, 515, 590, 600E | 25212 | ea. | 25364 |
| Outlet Check Valve Rebuild Kit (Actuator Style) | M6KA, 501, 510, 515, 590, 600E | 26016 | 2-pk. | 25365 |
| Outlet Check Valve Assembly (Ball & Seat Style) | M6KA, 501, 510, 515, 590, 600E | 25216 | ea. | 25366 |
| Outlet Check Valve Housing (Ball & Seat Style) | M6KA, 501, 510, 515, 590, 600E | 25207 | ea. | 25367 |
| Outlet Check Valve Rebuild Kit (Ball & Seat Style) | M6KA, 501, 510, 515, 590, 600E | 26014 | 2-pk. | 25368 |
| Inlet Check Valve Assembly, 225µL (Extended Flow) | M6KA, 501, 510, 515, 590, 600E | 60307 | ea. | 25369 |
| PerformancePLUS™ Check Valve Housing | M6KA, 501, 510, 515, 590, 600E | 700000254 | 2-pk. | 25370 |
| Check Valve Rebuild Kit (Extended Flow) | M6KA, 501, 510, 515, 590, 600E | 88223 | 2-pk. | 25371 |
| PerformancePLUS™ Check Valve Housing | M6KA, 501, 510, 515, 590, 600E | — | ea. | 25372 |
| Round Pump Head w/ Actuator Outlets | M6KA, 510, 590, 600 | 60058 | ea. | 25413 |
| Round Pump Head, Ball & Seat Check Valves | M6KA, M45, 501 | — | ea. | 25414 |
| Check Valve Cartridges | Alliance™ | WAT270941 | 2-pk. | 25373 |
| Super Seal™ for Analytical Heads | M6KA, 501, 510, 515, 590, 600E | 22946, 22934 | ea. | 25374 |
| Plunger Seal, Gold (Analytical Heads) | M6KA, 501, 510, 515, 590, 600E | 22934 | ea. | 25375 |
| Plunger Seal, Tan | M6KA, 501, 510, 515, 590, 600E | 25384 | ea. | 25376 |
| Plunger Seal, Black | M6KA, 501, 510, 515, 590, 600E | 26613 | ea. | 25378 |
| Plunger Seal, Gold (EF Heads) | 510, 590, 600E | 26644 | ea. | 25380 |
| Seal Wash Plunger Seal | Alliance™ | WAT271018 | 2-pk. | 25386 |
| Head Plunger Seal Kit | Alliance™ | WAT270938 | 2-pk. | 25387 |
| Insert Seal Parts Kit | M6KA, 501, 510, 515, 590, 600E | 60012 | kit | 25389 |
| Sapphire Plunger | M6KA, 510, 590, 600 | 25656 | ea. | 25381 |
| Sapphire Plunger | M45, M501 | 26524 | ea. | 25383 |
| Sapphire Plunger | M515 | WAT207069 | ea. | 25384 |
| Sapphire Plunger | 616, 625, 626 | 31788 | ea. | 25420 |
| Sapphire Plunger | Alliance™ | WAT270959 | ea. | 25385 |
| Single Solvent Inlet Manifold | 600E | 60034, 60042 | ea. | 25390 |
| Gradient Proportioning Valve, 12Volt | 600E | 62037 | ea. | 25419 |
| Wash Face Seal | Alliance™ 2690 | WAT271017 | ea. | 25428 |
| Wash Tube Seal Kit | Alliance™ 2690 | WAT270940 | 4-pk. | 25429 |
| Proportioning Valve | Alliance™ 2690 | WAT270927 | ea. | 25430 |
| Xenon Lamp | 474 | — | ea. | 25405 |
| Deuterium Lamp (UV/Vis) | 484 | 80357 | ea. | 25406 |
| Deuterium Lamp | 996, 2996 | WAT052586 | ea. | 25408 |
| Deuterium Lamp | 2487 | WAS081142 | ea. | 25409 |

# Super-Clean™ Gas-Trapping System for LC/MS

## New Quick-Change System Removes Hydrocarbon Impurities from Nitrogen

by Donna Lidgett, GC Accessories Product Marketing Manager

✔ Fast, easy cartridge changes—no tools, no purge time, no potential contamination.
✔ Durable, patented full glass/metal design.

The Super-Clean™ Gas-Trapping System is the latest technology in cartridge systems for gas purification, and is ideal for purifying nitrogen for LC/MS systems. Changing cartridges is quick and easy. A two-position base plate, installed in the gas line, allows you to exchange cartridges without introducing oxygen into the system: spring-loaded check valves seal when a cartridge is removed and open only when a new cartridge has been locked in place. There is no need for loosening and tightening fittings every time you change cartridges, and your system cannot become contaminated during the changing process.

To meet the high flow needs of an LC/MS system, the two cartridges are positioned and connected in parallel. The incoming gas stream is split equally between the two cartridges, and the two streams are rejoined after purification but before the gas exits the base plate. This approach allows longer contact between the nitrogen and the charcoal adsorbent, ensuring higher gas purity. The system can deliver up to 20L of 99.9999% pure nitrogen per minute, at a maximum pressure of 11 bar (160psi). Estimated cartridge lifetime is 3 to 6 months.

**Special Offer**
Two Super-Clean™ filtration systems:
(2) 2-Position baseplates & (4) charcoal filters
cat.# 22063 Offer ends 12/31/02.

**Ordering Information** | Super-Clean™ Gas-Trapping for LC/MS

| Description | qty. | cat.# |
|---|---|---|
| LC/MS 2-Position Base Plate with 1/4" Fittings | ea. | 22060 |
| Charcoal Replacement Filters | 2-pk. | 22061 |
| Super-Clean™ Gas-Trapping System (includes 2-position base plate and 2 charcoal replacement filters) | ea. | 22062 |

# New Reference Books

## Higher Education Doesn't have to be Expensive

by Jack Crissman, Seminar and Educational Products Manager

***For review of these books, visit our website.***

**The Merck Index. 13th Edition:** *S. Budvari, Merck, 2001, 2560pp., ISBN 0-91-191013-1 cat.# 21383 (ea.)*

**A Century of Separation Science:** *Haleem J Issaq, Marcel Dekker, Inc., 2002, 755pp , ISBN 0-8247-0576-9* cat.# 20473 (ea.)

**Chromatography Theory:** *Jack Cazes and Raymond P.W Scott, Marcel Dekker, Inc , 2002, 475pp., ISBN 0-8247-0778-8 cat.# 21573 (ea.)*

**HPLC of Biological Macromolecules: Second Edition, Revised and Expanded:** *Karen M. Gooding and Fred E. Regnier, Marcel Dekker, Inc., 2002, 777pp., ISBN 0-8247-0665-X cat.# 21574 (ea.)*

**Advances in Chromatography, Volume 41:** *Phyllis R. Brown and Eli Grushka, Marcel Dekker, Inc., 2001, 425pp., ISBN 0-8247-0509-2* cat.# 21575 (ea.)

**Milestones in the Evolution of Chromatography:** *Leslie S. Ettre, ChromSource, Inc , 2002, 220pp , ISBN 0-9717144-0-1 cat.# 20472 (ea.)*

**Chromatography in Food Science and Technology:** *Tibor Cserháti and Esther Forgács, CRC Press, LLC, 1999, 552pp , ISBN 1-56676-749-0* cat.# 21492 (ea.)

**Gas Chromatographic Techniques and Applications:** *Alan J Handley and Edward R. Ardlard, CRC Press, LLC, 2001, 320pp., ISBN 0-8493-0521-7* cat.# 21491 (ea.)

**Handbook of Chemistry and Physics, 83rd Edition:** *D. R. Lide, CRC Press, LLC, 2002, 2,672pp., ISBN 0-8493-0483-0* cat.# 21442 (ea )

**Multidimensional Chromatography:** *L. Mondello, A. C. Lewis and K. D. Bartle, John Wiley, 2002, 436pp., ISBN 0-471-98869-3* cat.# 21443 (ea.)

**Modern Derivatization Methods for Separation Sciences:** *T. Toyo'oka, John Wiley, 1999, 298pp., ISBN 0-471-98364-0* cat.# 21444 (ea.)

## A Wealth of Practical Chromatography Experience, at a Location Near You

Again this year the chromatography wizards from Restek are presenting comprehensive seminars designed to help you minimize down-time and obtain the results you want. We keep these seminars to one day, and hold them at sites all around the country, to minimize the time you spend away from your lab and conserve your travel budget. The low cost of the seminar is an investment that can be quickly returned, because you will improve your lab throughput and spend less time dealing with problems. Choose the topic that suits your work:

***Comprehensive Capillary GC***
***Environmental GC Analysis***
***Comprehensive HPLC***
***Food, Flavor, and Fragrance Analysis***

Our brochure *2002 Seminars* (lit. cat.# 59282A) provides details about these seminars and lists dates and locations. Call, fax, or e-mail your request for the seminars brochure today, or view it on our website. We look forward to meeting you.

**Special Offer!**
**Capillary Column Installation Video (CD-ROM)**
Covers the critical points in installing a capillary GC column: instrument preparation, setting gas flows, leak checks, etc., produced by the technical wizards of Restek.
cat.# 20499, (ea.)

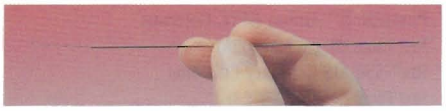
# cool tools

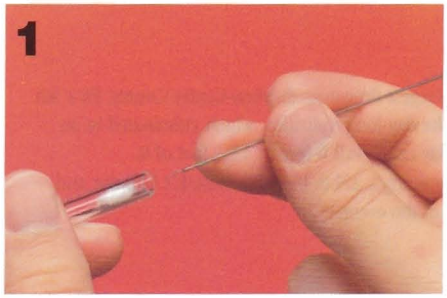
For Easier GC Maintenance Try These New Tools from Restek

by Brad Rightnour and Michael Goss, Instrument Innovations Team

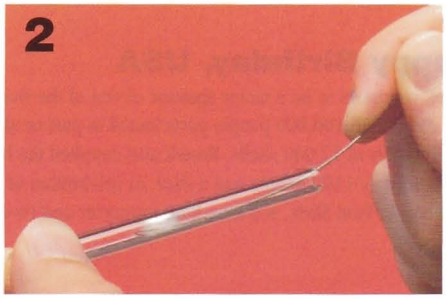
## Mini Wool Puller/Inserter

A wool plug that is incorrectly positioned or contaminated with finger oils can be more hindrance than help. This inexpensive little tool greatly simplifies the chore of consistently placing contaminant-free wool plugs in an inlet liner, and retrieving a plug when its time to replace it. We suggest you order several packages-or be ready to spend time trying to find out who's borrowed yours.
cat.# 20114, (2-pk.)






Place a 1cm plug of loosely bound wool in the liner. Adjust its position with the puller/inserter tool.




Use the hooked end of the puller/inserter to retrieve the plug when it's time to replace it.

*Use with conventional 2mm ID or 4mm ID liners and most other liner configurations, but not with double gooseneck liners.*

## MS Installation Gauge

Easily pre-seat ferrules for consistent installations in Agilent 5973 MS!

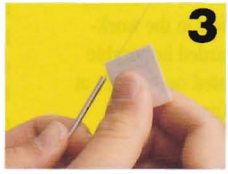
- ✔ Prevents damage to the column end
- ✔ Ensures leak-free connection

cat.# 21894, (ea.)

new


Install the nut and ferrule onto the column, then insert the column through the installation tool, exposing several centimeters at the exit end.


Tighten the nut.




Score and remove the exposed end of the column making sure of a clean, square cut, then loosen the nut.


The ferrule will be properly seated and should remain in place when light force is applied. Install the column into the GC/MS interface.

# Pre-Cleaned Sample Vials

## Ready to Use for Volatiles Analyses

by Donna Lidgett, GC Accessories Product Marketing Manager

new

- ✔ Container, liner, and closure cleaned, assembled, and ready to use.
- ✔ Open-top caps.
- ✔ Teflon®-faced 0.125" silicone septa.
- ✔ Each case lot numbered.
- ✔ Available in clear or amber.

| Description | qty. | cat.# |
|---|---|---|
| 20ml CLEAR Pre-Cleaned VOA Vials | 72-pk. | 21798 |
| 20ml AMBER Pre-Cleaned VOA Vials | 72-pk. | 21799 |
| 0ml CLEAR Pre-Cleaned VOA Vials | 72-pk. | 21796 |
| 40ml AMBER Pre-Cleaned VOA Vials | 72-pk. | 21797 |

# RESTEK Behind the Scenes

## Happy Birthday, USA

Restek continues to be a major sponsor of one of the world's largest fireworks displays-Centre County, PA's 4th Fest. More than 100,000 people participated in part or all of the day-long festivities, which culminated in an aerial display of 12,000 shells. Restek also supplied the birthday cake—all 6 feet by 12½ feet of it. Approximately 1100 people had a slice, in celebration of our country's 226th birthday. Restek's founder and head coach, Paul Silvis, is a long-time supporter and co-chair of the event.

## Restek Wins Healthy Workplace Award

Restek Corporation has been awarded the Healthy Workplace Award for Small Sized Companies by the Pennsylvania Psychological Association. This award is given annually to companies that demonstrate a commitment to family support, employee development, employee involvement, community involvement, and health and safety in the workplace. Last year, Restek was awarded honorable mention. The award was presented on June 21 at an official award ceremony in Lancaster.

Restek offers numerous employee-friendly benefits, including contributions toward child care costs, reimbursement for continuing education and development, on-site fitness and recreational facilities with subsidized personal trainers, 401k and employee stock ownership programs, open-book management, and a casual dress code. For the last 2 years, the company has won awards for being among the 100 Best Companies to Work for in Pennsylvania.

## Restek Employees Prove They're Healthy and Involved

Proving our Healthy Workplace Award was no accident, two Restek employees, Becky Wittrig (Innovations Team chemist) and Matt Reilly (ATM technician) took first place in their divisions in a 5K run benefiting a local library, in June. Becky's son, James, Matt's daughter, Leena, and Alex Reid, son of HPLC Applications Team chemist Terry Reid, ran in the children's divisions. Our Employee Action Group made a $100 sponsorship donation.

Also in June, Restek's Relay for Life team won the Bronze Award at the local Relay event by raising more than $1500 for the American Cancer Society.

## New Literature

- ✔ Narcotics / Acetaminophen by HPLC
  Applications Note (lit. cat.# 59453)
- ✔ Organophosphorus Pesticides by Capillary GC
  Applications Note (lit. cat.# 59359)
- ✔ Calibration Standards for ASTM Method D2887-01
  Fast Facts (lit. cat.# 59383A)
- ✔ Certified PAHs in Diesel Fuel #2
  Fast Facts (lit. cat.# 59384A)
- ✔ Environmental Gas Standards
  Fast Facts (lit. cat.# 59276)
- ✔ Pesticide Reference Materials
  Fast Facts (lit. cat.# 59446)
- ✔ Pinnacle II™ Amino HPLC Columns
  Fast Facts (lit. cat.# 59385A)
- ✔ Rtx®-5Sil MS Capillary Columns
  Fast Facts (lit. cat.# 59323)
- ✔ UST Products for Massachusetts
  Fast Facts (lit. cat.# 59391)
- ✔ UST Products for the Northwest Region
  Fast Facts (lit. cat.# 59396)
- ✔ UST Products for Wisconsin
  Fast Facts (lit. cat.# 59392)
- ✔ Rtx®-VMS Capillary Columns
  New Product Flyer (lit. cat.# 59209A)
- ✔ Bonded PLOT Columns—Flyer (lit. cat.# 59456)
- ✔ Integra-Guard™ Capillary Columns
  Flyer (lit. cat.# 59441)
- ✔ Review of Restek Literature for Pharmaceuticals Analyses—Flyer (lit. cat.# 59450)
- ✔ Genuine Restek Replacement Parts for Agilent GCs—Catalog (lit. cat.# 59627C)


**Restek USA**: Phone: 800-356-1688 or 814-353-1300 • Fax: 814-353-1309 • www.restekcorp.com
**Restek France**: Phone: 01 60 78 32 10 • Fax: 01 60 78 70 90 • restekfr@club-internet.fr
**Restek GmbH**: Phone: 49 06172 2797 0 • Fax: 49 06172 2797 77 • RESTEK-GMBH@t-online.de
**Restek Ireland**: Phone: 44 28 9081 4576 • Fax: 44 28 9081 4576 • restekeurope@aol.com
**Thames Restek UK LTD**: Phone: 01753 624111 • Fax: 01753 624666 • Sales@Thamesrestek.co.uk



ISO 9001 REGISTERED

**Lit. Cat.# 59461-INT**





Please direct your comments on this publication to Carrie Sprout, Graphic Designer, at carrie@restekcorp.com or call 814-353-1300, ext. 2151