



# THE RESTEK ADVANTAGE

Turning Visions into Reality™  
2005 vol. 2

## Rtx®-PCB: Unique Selectivity for PCBs

110 of 158 target PCB Congeners Elute Individually, Using GC/ECD

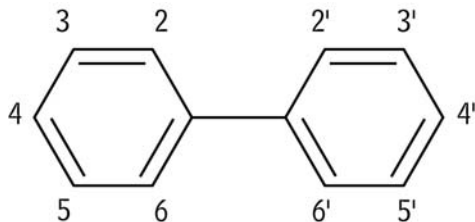
by Gary Stidsen, GC Columns Marketing Manager

- NEW low polarity, inert polymer phase provides distinct separations of PCB congeners.
- Unmatched selectivity and low bleed—a column of choice for trace analysis.
- Thermally stable to 340°C.

Rtx®-PCB columns show unique selectivity for polychlorinated biphenyl (PCB) congeners. In previous publications (lit.# 59925 and Advantage 2005v1, lit.# 59077, page 13), we discussed the excellent performance of this column and showed each of the European PCB congener indicator compounds - PCBs 28, 52, 101, 118, 138, 153, and 180 - can be resolved from other, interfering PCB congeners and quantified, using GC/MS.

“Weathering” of Aroclor® mixes that have been in the environment for more than 30 years, and changes in Aroclor® patterns in tissue samples, due to bioaccumulation, have dictated that PCBs now be reported as congeners, rather than as Aroclor® mixes. Consequently, many laboratories are analyzing longer lists of PCB congeners, using the data in determining specific congener patterns, in compiling congener results to obtain an accurate total PCB concentration, and in other ways.

**Figure 1** Biphenyl structure supports 209 PCB congeners, many with very similar retention characteristics.



The structure of the biphenyl molecule is shown in Figure 1. Identification and quantification of PCB congeners is chromatographically challenging because there are 209 possible combinations in which chlorine atoms can be added to the biphenyl structure, ranging from addition

of a single chlorine (monochlorobiphenyls) to addition at every available carbon atom (decachlorobiphenyl). In 1996, George Frame published work he performed in order to determine which PCB congeners are present in Aroclor® mixes.<sup>1</sup> Using this work as a guide, the list of 209 possible PCB congeners can be reduced to a target list of 158 congeners. This final target list includes PCB congeners found in Aroclor® mixes above 0.01%wt/wt, and a few compounds not detected in Aroclor® mixes, but detected in tissue due to

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### Restek Goes West!



Roy Lautamo gliding over Kings Canyon in the southern Sierra.

Restek celebrates continued growth in 2005 with the opening of Restek West, our new R&D facility in Shingle Springs, California. Roy Lautamo, Director of Innovative Research Chemistries, will manage the facility, focusing on R&D for our chromatography column product lines. Roy has an extensive range of experience in chromatography, acquired over more than a quarter of a century of research.

We welcome Roy and his staff into the Restek family!

### Correction

In *Advantage 2005v1: Fast GC/MS Analysis of Semivolatile Organic Compounds*, Figure 1 (page 14) the splitless hold time and pressure pulse time are reversed. The splitless hold time should be 0.15 min. and the pressure pulse time should be 0.20 min. We apologize for any inconvenience caused by this error.

**RESTEK** 20 YEARS

Turning Visions into Reality™

bioaccumulation (e.g., PCB#169). The list encompasses the seven European indicator compounds and the 12 most toxic congeners, according to the World Health Organization (WHO) list.<sup>2</sup>

Using a 40m x 0.18mm ID, 0.18 $\mu$ m film Rtx<sup>®</sup>-PCB column and the conditions listed in Figure 2, we evaluated a sample composed of Aroclor<sup>®</sup> PCB mixes 1242, 1254, and 1262. The 158 target PCB congeners were identified in the sample, eluted as 135 chromatographic peaks. Of the 158 congeners, 110 eluted singly and 48 were unresolved.

Produced through one of our newest polymer technologies, Rtx<sup>®</sup>-PCB columns undergo rigorous quality assurance measures, to ensure every column meets exacting standards and to give you highly reproducible performance, from column to column. If you are analyzing PCBs—as congeners, as Aroclor<sup>®</sup> mixtures, or as other mixtures (e.g., Kaneclor, Clophen, or Phenoclor mixtures)—we highly recommend using these new columns.

#### References

1. Frame, G., J. Cochran, and S. Bowadt, *Complete PCB Congener Distributions for 17 Aroclor Mixtures Determined by 3 HRGC Systems Optimized for Comprehensive, Quantitative, Congener-Specific Analysis* J. High Res. Chromatogr. 19, Dec. 1996, pp. 657-668.
2. Executive Summary, *Assessment of the Health Risk of Dioxins: Re-evaluation of the Tolerable Daily Intake (TDI)*, WHO Consultation, May 25-29, 1998, Geneva Switzerland. See: <http://www.who.int/ipcs/publications/en/exe-sum-final.pdf>

#### PCB Congener Standard #2

|                       |                            |
|-----------------------|----------------------------|
| 2,4,4' (BZ #28)       | 2,2',3,4,4',5' (BZ #138)   |
| 2,2',5,5' (BZ #52)    | 2,2',4,4',5,5' (BZ #153)   |
| 2,2',4,5,5' (BZ #101) | 2,2',3,4,4',5,5' (BZ #180) |
| 2,3',4,4',5 (BZ #118) |                            |

10 $\mu$ g/mL each in isoctane, 1mL/ampul  
cat. # 32294 (ea.)

For additional PCB congener mixes, and Aroclor<sup>®</sup> reference materials, please see our current catalog, or visit our website.

#### Rtx<sup>®</sup>-PCB (fused silica)

| ID     | df ( $\mu$ m) | temp. limits      | length   | cat. # |
|--------|---------------|-------------------|----------|--------|
| 0.18mm | 0.18          | 30°C to 320/340°C | 20-Meter | 41302  |
| 0.18mm | 0.18          | 30°C to 320/340°C | 60-Meter | 41304  |
| 0.25mm | 0.25          | 30°C to 320/340°C | 30-Meter | 13223  |
| 0.25mm | 0.25          | 30°C to 320/340°C | 60-Meter | 13226  |
| 0.32mm | 0.50          | 30°C to 320/340°C | 30-Meter | 13239  |

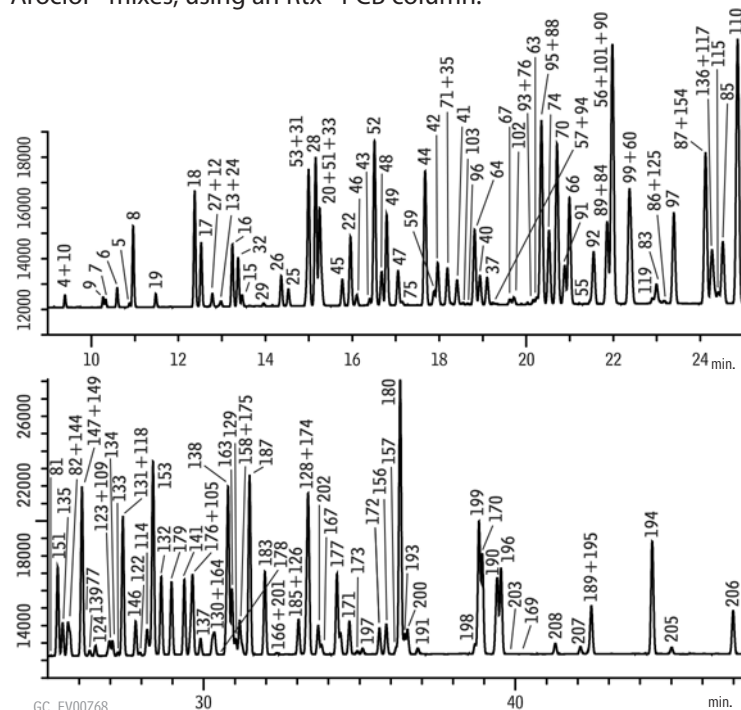
The maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

**Table I** 110 of 158 target PCB congeners in Aroclor<sup>®</sup> mixes are eluted singly from an Rtx<sup>®</sup>-PCB column.

| PCB# | s/m* | PCB# | s/m* | PCB# | s/m* | PCB# | s/m* | PCB# | s/m* |
|------|------|------|------|------|------|------|------|------|------|
| 1    | s    | 35   | s    | 82   | s    | 126  | s    | 173  | s    |
| 2    | s    | 37   | s    | 83   | s    | 128  | m    | 174  | m    |
| 3    | s    | 40   | s    | 84   | m    | 129  | s    | 175  | s    |
| 4    | m    | 41   | s    | 85   | s    | 130  | s    | 176  | m    |
| 5    | s    | 42   | s    | 86   | m    | 131  | m    | 177  | s    |
| 6    | s    | 43   | s    | 87   | m    | 132  | s    | 178  | s    |
| 7    | s    | 44   | s    | 88   | m    | 133  | s    | 179  | s    |
| 8    | s    | 45   | s    | 89   | m    | 134  | s    | 180  | s    |
| 9    | s    | 46   | s    | 90   | m    | 135  | s    | 183  | s    |
| 10   | m    | 47   | s    | 91   | s    | 136  | m    | 185  | s    |
| 11   | m    | 48   | s    | 92   | s    | 137  | s    | 187  | s    |
| 12   | m    | 49   | s    | 93   | m    | 138  | s    | 189  | s    |
| 13   | m    | 51   | m    | 94   | m    | 139  | s    | 190  | m    |
| 15   | s    | 52   | s    | 95   | m    | 141  | s    | 191  | s    |
| 16   | s    | 53   | m    | 96   | s    | 144  | s    | 193  | s    |
| 17   | m    | 54   | m    | 97   | s    | 146  | s    | 194  | s    |
| 18   | s    | 55   | s    | 99   | m    | 147  | m    | 195  | s    |
| 19   | s    | 56   | m    | 101  | m    | 149  | m    | 196  | m    |
| 20   | m    | 57   | m    | 102  | s    | 151  | s    | 197  | s    |
| 21   | m    | 59   | s    | 103  | s    | 153  | s    | 198  | s    |
| 22   | s    | 60   | m    | 105  | m    | 154  | m    | 199  | s    |
| 23   | m    | 63   | s    | 109  | m    | 156  | s    | 200  | s    |
| 24   | m    | 64   | s    | 110  | s    | 157  | s    | 201  | s    |
| 25   | s    | 66   | s    | 114  | s    | 158  | s    | 202  | s    |
| 26   | s    | 67   | s    | 115  | s    | 163  | s    | 203  | s    |
| 27   | m    | 70   | s    | 117  | m    | 164  | s    | 205  | s    |
| 28   | s    | 71   | s    | 118  | m    | 166  | s    | 206  | s    |
| 29   | s    | 74   | s    | 119  | s    | 167  | s    | 207  | s    |
| 31   | m    | 75   | s    | 122  | s    | 169  | s    | 208  | s    |
| 32   | s    | 76   | m    | 123  | m    | 170  | s    | 209  | s    |
| 33   | m    | 77   | m    | 124  | s    | 171  | s    |      |      |
| 34   | s    | 81   | s    | 125  | m    | 172  | s    |      |      |

\*s - compound eluted singly; m - compound eluted with one or more other congeners.

**Figure 2** Excellent separation and peak shape for PCBs in three Aroclor<sup>®</sup> mixes, using an Rtx<sup>®</sup>-PCB column.



Column: Rtx<sup>®</sup>-PCB 40m, 0.18mm ID, 0.18 $\mu$ m (cat.# 41303)  
 Sample: 300ng/mL Aroclor<sup>®</sup> 1242/1254/1262 in hexane: Aroclor<sup>®</sup> 1242 (cat.# 32009), Aroclor<sup>®</sup> 1254 (cat.# 32011), Aroclor<sup>®</sup> 1262 (cat.# 32409)  
 Inj.: 1.0 $\mu$ L splitless (hold 0.75 min.), 4mm single gooseneck inlet liner (cat.# 20983)  
 Inj. temp.: 230°C  
 Carrier gas: hydrogen, constant pressure  
 Linear velocity: 40cm/sec. @ 100°C  
 Oven temp.: 100°C (hold 1 min.) to 200°C @ 30°C/min., to 320°C @ 2°C/min. (hold 1 min.)  
 Det.: ECD @ 330°C

# Accurately Monitor Mercury-Sulfur-Nitrogen Compounds

## Siltek®/Sulfinert® Treatment Prevents Adsorption of Mercury, Sulfur Oxides, or Nitrous Oxides in Emission Monitoring Equipment

By Gary Barone, Restek Performance Coatings Division Manager, David Smith, RPC Chief Scientist, and Martin Higgins, RPC Chief Engineer

- Improved analytical reliability and sensitivity for mercury, SO<sub>x</sub>, or NO<sub>x</sub> compounds.
- Protection from corrosion—longer component lifetime.
- Apply to new or existing equipment.

The United States Environmental Protection Agency (US EPA) is actively developing regulations, limits, and control measures for monitoring and controlling mercury emissions from coal-fired power generators—one of the major sources of mercury emissions into the environment.<sup>1</sup> As these regulations and guidelines are developed and implemented, proper equipment will be needed for accurate sampling and analysis. Testing costs for mercury can be substantial (Table 1)<sup>2</sup>, so inaccurate analyses can have financial as well as environmental repercussions.

In flue streams from coal-fired power generators, mercury exists in three forms: elemental, the +2 oxidation state (Hg<sup>++</sup>), and attached to particulate matter. Hg<sup>++</sup> often reacts with sulfur compounds, nitrogen, chlorine, and/or oxygen, to produce sulfurous, nitrous, chloride, and oxide mercury species. Elemental and oxidized mercury can easily be lost to reactions and adsorption on the inner surfaces of monitoring equipment. In order to accurately sample and quantify mercury in all forms, it is important to use inert sample pathways. Laboratory testing and field results have proven that Sulfinert® treated sampling and testing equipment is essentially inert to active molecules<sup>3</sup>, including mercury.

Siltek®/Sulfinert® treatment can be applied to many of the components in a mercury sampling stream, including probe tubing, impingers, fittings, filters, housings, and transfer tubing (Figure 1). Treating all of the components of a stack or continuous emission monitoring system will greatly improve analytical reliability and sensitivity, which will be needed as regulations are brought on line and emission quotas are enforced. Fast and accurate testing, without re-work, can save a great deal of time and money.

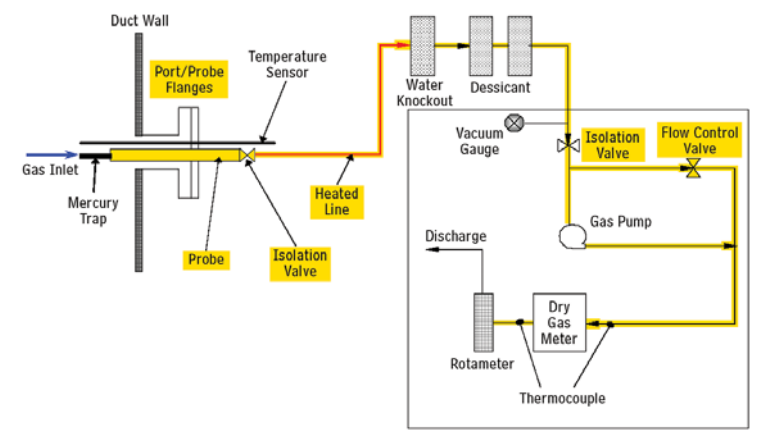
Similarly, a Siltek®/Sulfinert® treated sampling system will improve the reliability of data for sulfurous oxides and nitrous oxides (SO<sub>x</sub> and NO<sub>x</sub>). As with mercury, it is difficult to reliably transfer these compounds through untreated sampling equipment.

In addition to preventing adsorption of reactive compounds, Siltek®/Sulfinert® treatment will act as a barrier, protecting and prolonging the lifetime of treated equipment. The durable layer will withstand temperatures to 400°C.

We offer Siltek®/Sulfinert® treated tubing, sample cylinders, and other components from stock; to discuss custom treatment of system components, please contact the Restek Performance Coatings team.

Restek offers treated and untreated tubing, fittings, and valves, passive air sampling kits, air sampling canisters and miniature air canisters, sample loops, and more. For more information, request our catalog or visit us online. [www.restekcoatings.com](http://www.restekcoatings.com)

**Figure 1** Highlighted components of a mercury sampling train,<sup>4</sup> and all tubing in the system, can be Siltek®/Sulfinert® treated.



**Table 1** Typical costs of mercury sampling (U.S.).<sup>2</sup>

| Method        | Approx. Cost of Analysis |
|---------------|--------------------------|
| US EPA 29     | \$300                    |
| US EPA 101A   | \$100                    |
| ASTM D6784-02 | \$250                    |
| US EPA 324    | \$430                    |
| FAMS          | \$640                    |

#### References

1. Pottinger, M., S. Stecklow, and J.J. Fialka, *Invisible Export, A Hidden Cost of China's Growth: Mercury Migration* The Wall Street Journal Online, Dec. 17, 2004.
2. Serne, J.C., *An Overview and Comparison of Available Mercury Emission Test Methods for Boilers* Symposium on Air Quality Measurement; Methods and Technology 2005, San Francisco, CA; Air & Waste Management Association. paper no. 439, pg. 9.
3. Barone, G., M. Higgins, D. Smith, S. Rowan, W.J. Gross, and P. Harris, *The Surface for Sulfurs Hydrocarbon Engineering*, Dec. 2004, pp 47-50.
4. Proposed Method 324. *Determination of Vapor Phase Flue Gas Mercury Emissions from Stationary Sources Using Dry Sorbent Trap Sampling* United States Environmental Protection Agency. Washington, D.C. p. 5.





# Identify and Quantify Adulterants in Seized Cocaine

Using GC/MS (Rtx®-440 Column) and HPLC/RI (Pinnacle II™ Amino Column)

By Kristi Sellers, Clinical/Forensic Innovations Chemist, and Rick Morehead, R&D GC Column Group Leader

- Low bleed Rtx®-440 column improves resolution and inertness for adulterants by GC/MS.
- GC/MS provides positive identification for all adulterants except sugars; data can be used as evidence.
- HPLC is the preferred chromatographic method for identifying sugars as adulterants.

Illicit cocaine is commonly “cut” with adulterants or diluents to increase the amount of product available for sale. Because the composition of an illicit cocaine mixture can be specific to one dealer, identification of adulterants and diluents in seized cocaine is critical in determining the possible routes of distribution and sales.

Either GC or HPLC can be used to identify cocaine adulterants such as sugars, anesthetics, analgesics, and stimulants. GC is the most common analytical technique used for analyzing all cocaine adulterants except sugars. Although sugars can be derivatized for analysis by GC, they are more easily detected using HPLC.

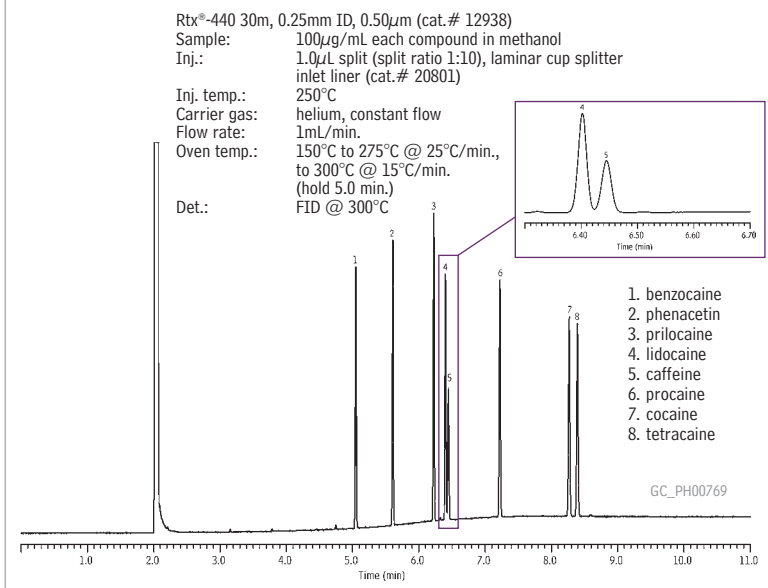
## GC

Cocaine mixture components can be detected using flame ionization detection (FID, Figure 1), nitrogen-phosphorus detection (NPD), or mass spectrometry (MS). Although FID or NPD provide good sensitivity for the adulterants, GC/MS is the most widely accepted detection method. MS is very sensitive, provides positive identifications based on mass spectra, and MS data are accepted as confirming evidence in courts of law.

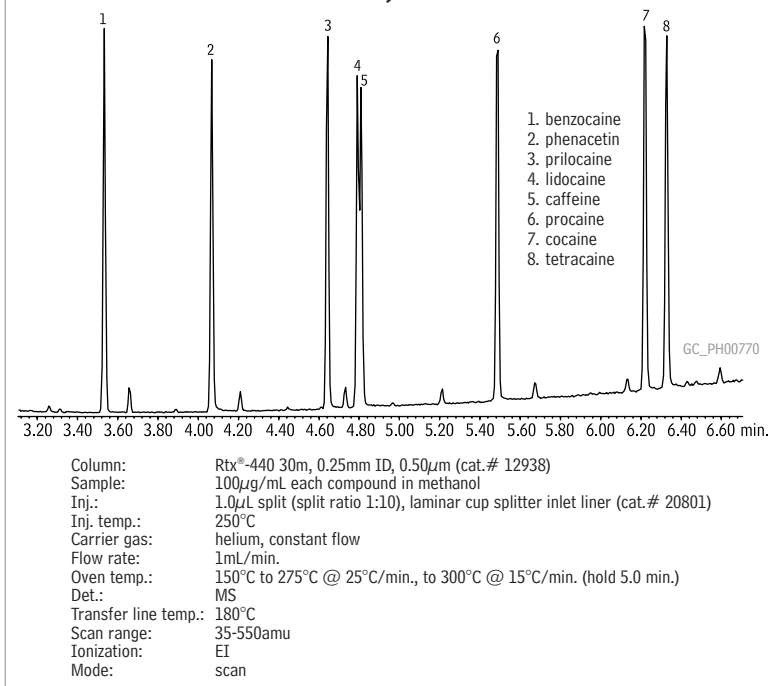
Among the column types we evaluated, only Rtx®440 columns resolved lidocaine and caffeine to baseline (Figure 1). To evaluate the columns, we prepared mock samples of illicit cocaine by adding equal concentrations of a variety of adulterants and diluents to cocaine hydrochloride. We used stimulants, including caffeine, local anesthetics, such as lidocaine, and over-the-counter analgesics, such as phenacetin, and followed a simple “dilute and shoot” sample preparation scheme to dissolve the samples for analysis.

We developed a GC/MS method that enabled us to identify each adulterant or diluent, focusing on maximizing resolution while minimizing total analysis time in order to increase sample throughput. In the optimized GC/MS method (Figure 2), total analysis time was 6.5 minutes. Unlike in the GC/FID analysis (Figure 1), caffeine and lidocaine were not resolved to baseline, but were resolved by approximately 40% (Figure 2), due to MS vacuum effects on sample flow through the column. Caffeine and lidocaine have very different mass spectra, however, and extracted ion analysis ensured

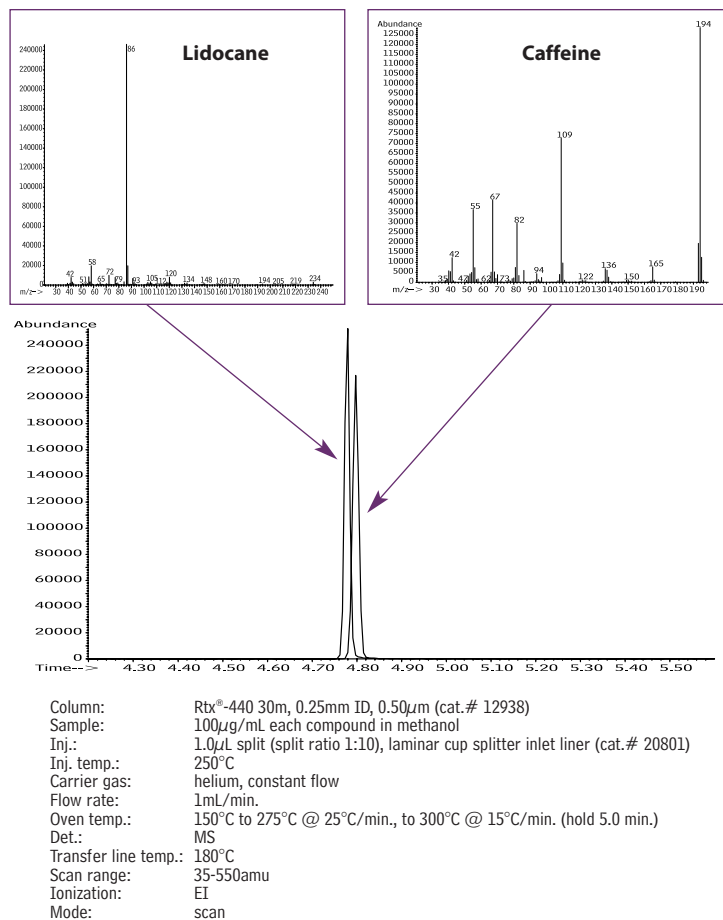
**Figure 1** An Rtx®-440 column resolves lidocaine/caffeine, and other cocaine adulterants, to baseline.



**Figure 2** Analyze cocaine adulterants in 6.5 minutes, using an Rtx®-440 column in a GC/MS analysis.



**Figure 3** Distinctive mass fragments ensure positive identification of lidocaine and caffeine.



positive identification and allowed quantification of each compound. Lidocaine and caffeine have distinctive mass fragments of 86m/z and 194m/z, respectively (Figure 3).

#### HPLC

Sugars are not easily volatilized and, therefore, are difficult to analyze by GC, making HPLC the better chromatographic approach for this analysis. Further, refractive index (RI) or evaporative light-scattering (ELS) detection must be used because sugars have no UV chromophore. HPLC/RI or HPLC/ELS provides reproducible retention times, adequate peak identification and good quantification for sugars, as shown in Figure 4.

HPLC/MS methods for simultaneous analysis of cocaine, sugars, and other classes of adulterants and diluents have not yet been developed, but such methods would enable analysts to evaluate street cocaine mixtures in one analysis. Column parameters and mobile phase composition will be critical parameters to optimize.

#### Conclusions

Cocaine samples can be “fingerprinted” by identifying and quantifying the adulterants and diluents mixed with the drug. GC/MS provides adequate quantitative information about the concentration of each additive, relative to the cocaine concentration, and provides undisputable identification of a substance (retention time and mass spectrum data). Therefore, GC/MS is the preferred chromatographic method for analyzing cocaine and most cocaine adulterants. Sugars are best analyzed by HPLC.

#### for more information

Smith, F.P, *Handbook of Forensic Drug Analysis*, pp.235-275, Elsevier, 2005.

Telepchak, M.J., T.F. August, and G. Chaney, *Forensic and Clinical Applications of Solid Phase Extraction*, pp.204-213, Humana Press, 2004.

#### Rtx®-440 (fused silica)

(proprietary intermediate-polarity Crossbond® phase)

| ID     | df (µm) | temp. limits      | length   | cat. # |
|--------|---------|-------------------|----------|--------|
| 0.25mm | 0.25    | 20°C to 320/340°C | 30-Meter | 12923  |
| 0.25mm | 0.50    | 20°C to 320/340°C | 30-Meter | 12938  |

#### Pinnacle II™ Amino

3µm Particles, 4.6mm ID cat. #  
150mm 9217365

#### Carbohydrate HPLC Performance Check Mix

Dry components in 4mL screw-cap vial.

Reconstitute in 1mL acetonitrile:water (75:25) to 2.0, 2.1, 4.4, 4.5, 4.0 mg/mL, respectively.

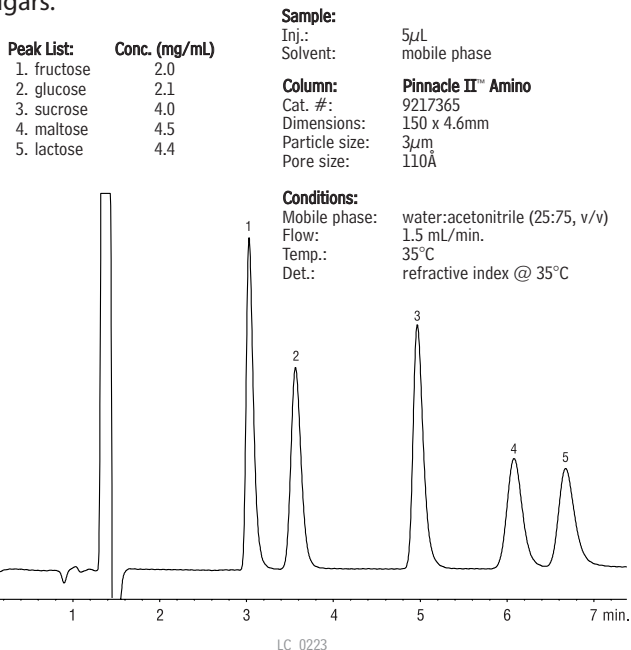
|                    |       |         |     |
|--------------------|-------|---------|-----|
| glucose            | 2.0mg | maltose | 4.5 |
| fructose           | 2.1   | sucrose | 4.0 |
| lactose            | 4.4   |         |     |
| cat. # 31809 (ea.) |       |         |     |

No data pack available.

#### tech tip

We recommend using an HPLC guard column for this application. For Trident™ guard column systems, refer to our catalog, or visit our website at [www.restek.com/hplc](http://www.restek.com/hplc).

**Figure 4** Pinnacle II™ Amino column provides fast, reliable analyses for sugars.



# New HPLC Confirmation Column for Explosives Analysis

## Introducing the Pinnacle II™ Biphenyl Column

By Becky Wittrig, Ph.D., HPLC Product Marketing Manager, Randy Romesberg, HPLC Applications Chemist, and Mike Wittrig, R&D Chemist

- Excellent resolution of US EPA Method 8330 explosives.
- Significantly different selectivity, relative to C18 columns; better resolution than cyano columns.
- Allows quantitative as well as qualitative confirmation.

Testing of residual materials is important when monitoring the disposal of expired or deteriorated munitions. US EPA Method 8330 was developed for quantifying 14 commonly monitored explosives. The method calls for reversed phase HPLC with UV detection, using a primary column and a confirmation column. The primary column contains a C18 stationary phase and, typically, the confirmation column contains a cyano stationary phase. Resolution of the target explosives is poor on cyano stationary phases, however, and the analysis provides qualitative confirmation only.

Restek chemists have developed a superior alternative to cyano phases for explosives analysis. The Pinnacle II™ Biphenyl column provides excellent resolution of EPA Method 8330 explosives, as shown in Figure 1. Further, selectivity is markedly different from that of a C18 column (Figure 2), making the Pinnacle II™ Biphenyl column a true, ideal, confirmation column. Separations on either column are accomplished with a simple, isocratic water:methanol mobile phase.

Restek offers a complete set of analytical reference materials for Method 8330. Our calibration materials for explosives analysis by HPLC are available in two options: as 1000ppm solutions of individual analytes, or as two 7-component mixtures, described on page 7. The internal standard, 3,4-dinitrotoluene, and the surrogate standard, 1,2-dinitrobenzene, also are available as described on page 7.

For superior data from your confirmation analysis for explosives, we highly recommend a Pinnacle II™ Biphenyl HPLC column.

### Pinnacle II™ Biphenyl

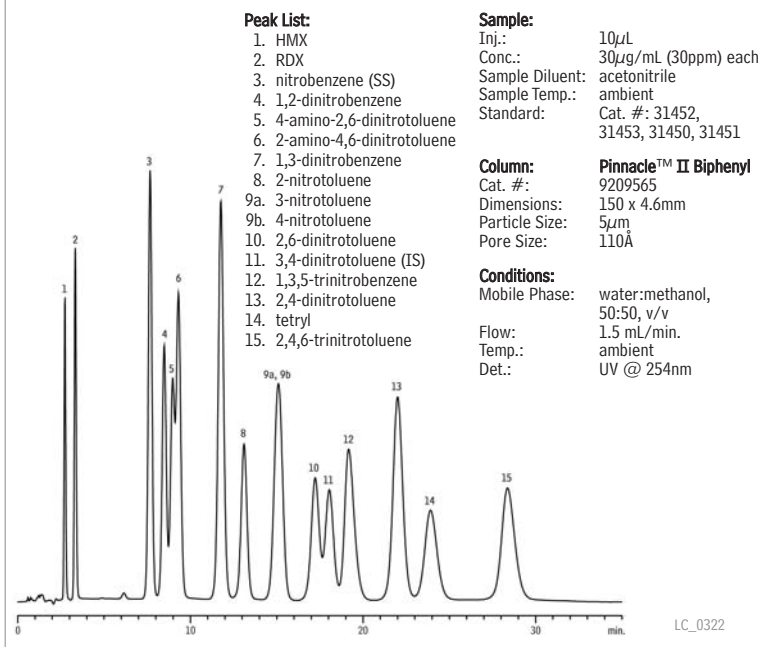
5µm Particles, 4.6mm ID  
150mm cat. #  
9209565

### Ultra C18 Columns

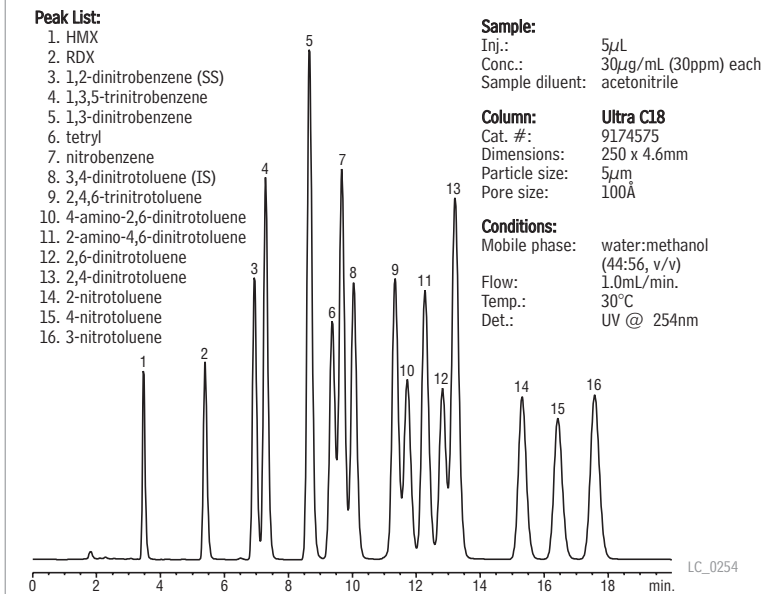
5µm Particles, 4.6mm ID  
250mm cat. #  
9174575

For individual solutions of EPA Method 8330 analytes, please see our catalog, or visit our website.

**Figure 1** Excellent resolution of US EPA Method 8330 explosives, using a Pinnacle II™ Biphenyl column.



**Figure 2** An Ultra C18 column is an outstanding primary column for explosives analysis.



# Analytical Reference Materials

## High-Purity Explosives - On-Line Data Packs - Custom Mixes

By Ken Herwehe, Analytical Reference Materials Product Marketing Manager

### High-Purity Reference Materials for Explosives

HPLC with UV detection is used to measure nitroaromatic and nitramine explosives and their degradation products in water and soil samples.<sup>1</sup> Obtaining pure, neat compounds for these standards can be very difficult. Some of these commercial-grade materials contain desensitizing agents such as beeswax, water, or other manufacturing by-products. Many are shipped wet and must be carefully dried before preparation. To ensure the highest quality standards, Restek chemists carefully purify or synthesize each compound to 98% pure or higher.

#### Reference

1 US Environmental Protection Agency. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. SW-846 Update III, Office of Solid Waste, Washington, DC, 1997.  
(Reference not available from Restek.)

#### 8330 Calibration Mix #1 (7 components)

|                    |                       |
|--------------------|-----------------------|
| 1,3-dinitrobenzene | RDX                   |
| 2,4-dinitrotoluene | 1,3,5-trinitrobenzene |
| HMX                | 2,4,6-trinitrotoluene |
| nitrobenzene       |                       |

1,000µg/mL each in acetonitrile, 1mL/ampul  
cat. # 31450 (ea.)

#### 8330 Calibration Mix #2 (7 components)

|                            |                |
|----------------------------|----------------|
| 2-amino-4,6-dinitrotoluene | 3-nitrotoluene |
| 4-amino-2,6-dinitrotoluene | 4-nitrotoluene |
| 2,6-dinitrotoluene         | tetryl         |
| 2-nitrotoluene             |                |

1,000µg/mL each in acetonitrile, 1mL/ampul  
cat. # 31451 (ea.)

#### 8330 Internal Standard

3,4-dinitrotoluene  
1,000µg/mL in methanol, 1mL/ampul  
cat. # 31452 (ea.)

#### 8330 Surrogate

1,2-dinitrobenzene  
1,000µg/mL in methanol, 1mL/ampul  
cat. # 31453 (ea.)

#### 8330 Nitroaromatics Kit

31450: 8330 Calibration Mix #1  
31451: 8330 Calibration Mix #2  
31452: 8330 Internal Standard Mix  
31453: 8330 Surrogate Mix

Contains 1mL each of these mixtures.  
cat. # 31454 (kit)

For individual solutions of these analytes, please see our catalog, or visit our website.

### free data packs

Restek now offers free downloadable data packs for analytical reference material products. Just visit our website at [www.restek.com/datapacks](http://www.restek.com/datapacks). Enter the catalog number and lot number for the product you ordered and obtain a printable PDF file.



### searching for the perfect solution?

Restek, "the company chromatographers trust™", should be your first choice for custom-made reference materials. Maximum convenience, maximum value, minimum time spent blending calibration mixtures in your laboratory.

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# Excellent Protein Separations from Viva™ HPLC Columns

## Best Performance Among Five Tested Wide Pore Columns

By Bruce Albright, HPLC Chemist; Vernon Bartlett, HPLC Manager; Julie Kowalski, Foods, Flavors, and Fragrances Innovations Chemist; and Becky Wittrig, Ph.D., HPLC Product Marketing Manager

- Best overall performance among five columns evaluated.
- Best resolution and peak symmetry for test proteins.
- C18, C8, C4, and silica columns available; other phases on request.

Reversed phase HPLC is an important technique for separating large biomolecules, such as proteins and peptides. Analysts generally employ C18 stationary phases, because these typically provide the best separations of related compounds, such as genetic variants of a protein or complex tryptic digests. However, limitations often are encountered when analyzing samples containing complex mixtures of closely related analytes. Columns containing wide pore silica (e.g., 300Å) are designed specifically for large molecule analyses, addressing this need for more resolving power.

Developed on Viva™ wide pore silica, Viva™ HPLC columns have ideal performance characteristics for separating large molecules and biomolecules. Using a reversed phase test mix, we compared column efficiency, peak asymmetry, and retention for Viva™ C18 columns and four other C18 wide pore HPLC columns. The Viva™ C18 column ranked highest in retention and selectivity and produced the best peak symmetry measurements (Table I).

To determine overall separating power, retention, and peak shape, we evaluated each column with a protein test mix. The Viva™ C18 column provided excellent resolution and peak shapes, as Figure 1 shows.

300Å silicas enhance resolution of similar or related analytes for several reasons. Large pore materials can provide greater retention because higher molecular weight analytes can enter more of the pores and access more surface area. Theoretically, the more surface to which an analyte has access, the longer the retention. For analytes with molecular weights greater than 3000, silica materials with pore diameters in the 250-350Å range yield the needed retention. Further, the mean pore diameter within the distribution (e.g., 250Å vs 350Å) can define the selectivity in some separations, by changing the elution order for certain analytes.

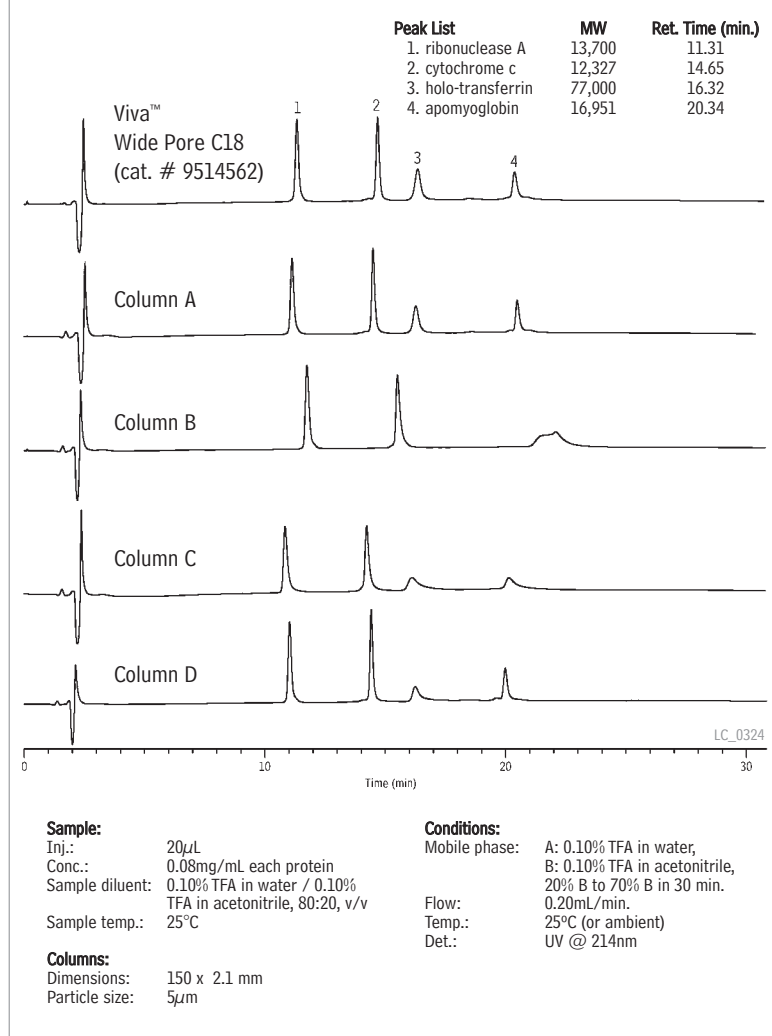
A 250-350Å mean pore diameter also is important because silicas with excessive numbers of pores smaller than 200Å can be more easily fouled by

**Table I** Viva™ wide pore C18 columns provide the best overall performance among five tested columns.

| Column        | Efficiency (plates/meter) | Asymmetry (biphenyl) | Retention Time (biphenyl) | Column Pressure (bar) |
|---------------|---------------------------|----------------------|---------------------------|-----------------------|
| Viva™ 300 C18 | >50,000                   | 1.16                 | 6.30                      | 60                    |
| Column A C18  | ~50,000                   | 1.46                 | 5.77                      | 72                    |
| Column B C18  | >50,000                   | 1.46                 | 4.96                      | 102                   |
| Column C C18  | >50,000                   | 1.30                 | 5.89                      | 66                    |
| Column D C18  | <50,000                   | 1.49                 | 3.79                      | 80                    |

Reversed phase test mix; 150 x 2.1mm C18 phase columns, 5µm particles

**Figure 1** Analysis of a four protein test mix shows the superior performance of the Viva™ C18 column.





larger molecular weight debris, and silicas with a high percentage of pores larger than 500Å can be impractically fragile for conventional HPLC applications. A narrow distribution around the mean pore diameter is advantageous; it better ensures that proper selectivity is maintained, and aids in separating closely related analytes that differ only slightly in hydrodynamic volume (molecular size in solution).

We developed and introduced Viva™ wide pore silica specifically to meet these challenging criteria. Among the materials we have tested, this new silica provides the greatest available surface area represented by 250-350 Å pores, with a highly desirable pore volume and pore diameter distribution (The Restek Advantage, 2005v1).

Superior physical characteristics and strong test performances show Viva™ HPLC columns are an excellent choice for analyzing proteins, peptides, or other large molecules or biomolecules. C18, C8, C4, and silica columns currently are available; other phases can be prepared on request. If you require a wide pore silica column for your analysis, we highly recommend new Viva™ columns.

### Viva™ Wide Pore HPLC Columns

- Excellent for separating peptides or proteins.
- Rugged, spherical particles, with 300Å pore size.
- High proportion of pore/surface area available to large molecules.

| Length                                     | 1.0mm ID |  | 2.1mm ID |  | 3.2mm ID |  | 4.6mm ID |  |
|--|----------|--|----------|--|----------|--|----------|--|
|  | cat.#    |  | cat.#    |  | cat.#    |  | cat.#    |  |
| <b>Viva™ Wide Pore C18 Columns, 5µm</b>    |          |  |          |  |          |  |          |  |
| 30mm                                       | 9514531  |  | 9514532  |  | 9514533  |  | 9514535  |  |
| 50mm                                       | 9514551  |  | 9514552  |  | 9514553  |  | 9514555  |  |
| 100mm                                      | 9514511  |  | 9514512  |  | 9514513  |  | 9514515  |  |
| 150mm                                      | 9514561  |  | 9514562  |  | 9514563  |  | 9514565  |  |
| 200mm                                      | 9514521  |  | 9514522  |  | 9514523  |  | 9514525  |  |
| 250mm                                      | 9514571  |  | 9514572  |  | 9514573  |  | 9514575  |  |
| <b>Viva™ Wide Pore Silica Columns, 5µm</b> |          |  |          |  |          |  |          |  |
| 30mm                                       | 9510531  |  | 9510532  |  | 9510533  |  | 9510535  |  |
| 50mm                                       | 9510551  |  | 9510552  |  | 9510553  |  | 9510555  |  |
| 100mm                                      | 9510511  |  | 9510512  |  | 9510513  |  | 9510515  |  |
| 150mm                                      | 9510561  |  | 9510562  |  | 9510563  |  | 9510565  |  |
| 200mm                                      | 9510521  |  | 9510522  |  | 9510523  |  | 9510525  |  |
| 250mm                                      | 9510571  |  | 9510572  |  | 9510573  |  | 9510575  |  |

### HPLC Reversed Phase Test Mix #1

Routine analysis using this product assists in determining the preferred performance of your system and system maintenance.

|         |           |             |      |
|---------|-----------|-------------|------|
| benzene | 3.00mg/mL | naphthalene | 0.50 |
| uracil  | 0.02      | biphenyl    | 0.06 |

In methanol:water (75:25), 1mL/ampul

cat. # 35005 (ea.)

No data pack available.

## Survival Kits for HPLC

Invaluable for Keeping Your System Running Smoothly!

By Becky Wittrig, Ph.D., HPLC Product Marketing Manager

- Tubing, fittings, and tools for system start-up or maintenance.
- Choose PEEK® or stainless steel components.
- More convenient and more economical than ordering components separately.

Restek HPLC survival kits contain practical selections of tubing, fittings, and tools for setting up or maintaining your HPLC system. The PEEK® Survival Kit contains PEEK® tubing, connectors, and elbows, Teflon® tubing, a tubing cutter and extra blades, a ValvTool wrench, open-end wrenches, and more. The Stainless Steel Survival Kit contains a selection of lengths and IDs of 1/16-inch tubing, plus nuts, ferrules, a ValvTool wrench, and a zero-dead-volume union.

### did you know?

Restek offers a wide range of HPLC columns, tools, and accessories, and many replacement parts for Agilent, Beckman, Hitachi, PerkinElmer, Shimadzu, Thermo Separation, and Waters instruments. Call us for a copy of our latest HPLC catalog (lit. cat.# 59241B), or visit us on line.

### PEEK® Survival Kit for HPLC

The PEEK® Survival Kit is an invaluable parts kit that contains tools and supplies essential for setting up and maintaining your HPLC system.

| Description                 | qty. | cat.# |
|-----------------------------|------|-------|
| PEEK® Survival Kit for HPLC | kit  | 25322 |



### Stainless Steel Survival Kit for HPLC

Contains a wide range of stainless steel tubing, plus fittings and a ValvTool wrench.

| Description                           | qty. | cat.# |
|---------------------------------------|------|-------|
| Stainless Steel Survival Kit for HPLC | kit  | 25097 |



# Improve Storage Stability for Sulfur Compounds

## Using Sulfinert® Treated Sample Cylinders

By Neil Mosesman, Air Monitoring Product Marketing Manager

- Eliminate sample-surface reactions in sample cylinders, collect and store active compounds.
- Obtain accurate data for active sulfur compounds at ppb levels.
- Many treated system components available from stock.

High-pressure sample cylinders are commonly used for collecting and storing refinery and natural gas samples containing trace amounts of sulfur compounds. These highly active compounds degrade very rapidly in stainless steel sample cylinders, making accurate determination of sulfur compounds virtually impossible. Restek's exclusive Sulfinert® surface treatment eliminates the reactivity of high-pressure sample cylinders and allows collection and stable storage of sulfur compounds, even at ppb levels. Figure 1 shows the recovery of 17ppbv hydrogen sulfide, carbonyl sulfide, methyl mercaptan, ethyl mercaptan, and dimethyl disulfide after 60 hours of storage in a Sulfinert® treated sample cylinder. The data show that these active compounds were unaffected by long-term storage in the Sulfinert® treated cylinder.

In addition to Sulfinert® treated cylinders, we also offer Sulfinert® treated valves, tubing, and sample loops to ensure the entire sample pathway is inert. Custom treatment is available for a wide range of items.

### Sulfinert®-Treated Sample Cylinders

- Stable storage of low concentrations of sulfur compounds.
- D.O.T. rated to 1800psi at room temperature.
- 316 stainless steel, 1/4" female NPT threads on both ends.

| Size   | qty. | cat.# |
|--------|------|-------|
| 75cc   | ea.  | 24130 |
| 150cc  | ea.  | 24131 |
| 300cc  | ea.  | 24132 |
| 500cc  | ea.  | 24133 |
| 1000cc | ea.  | 24134 |
| 2250cc | ea.  | 21394 |

### Sulfinert®-Treated Sample Cylinder Valves and Rupture Discs

- All "wetted" valve parts are Sulfinert®-treated.
- Maximum pressure rating, 5000psi.

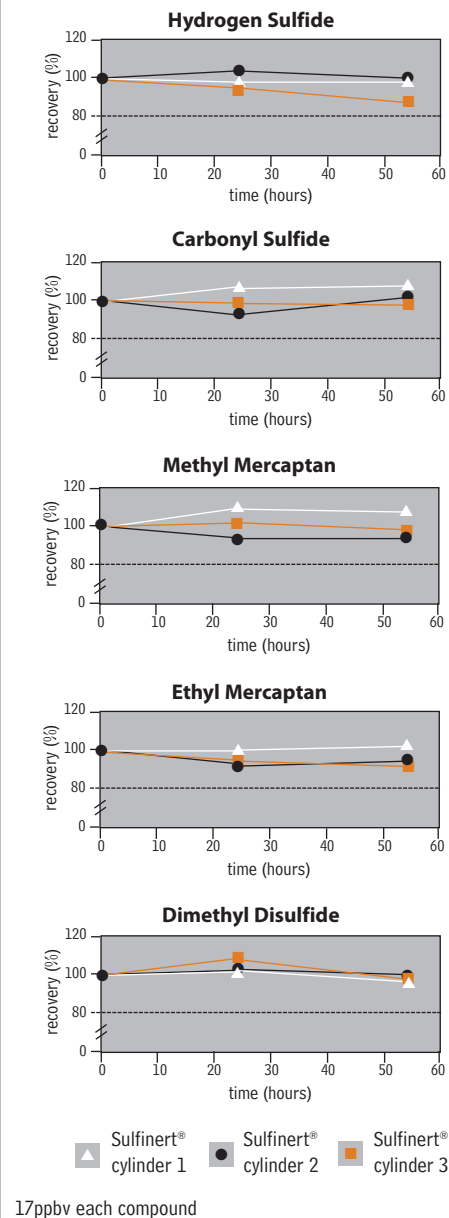
| Description                                    | qty. | cat.# |
|--|------|-------|
| 1/4" NPT Exit, Kel-F® Stem Tip                 | ea.  | 24127 |
| 1/4" Compression Exit, Kel-F® Stem Tip         | ea.  | 24128 |
| 1/4" Female NPT Outlet (built-in rupture disc) | ea.  | 21395 |

### Sulfinert®-Treated Gas Sample Loops

- Ideal for samples containing low concentrations of sulfur compounds.
- Sizes from 5µL to 5cc.; 1/16" fittings, for "W Type" valves.

| Size  | qty. | cat.# |
|-------|------|-------|
| 5µL   | ea.  | 22840 |
| 10µL  | ea.  | 22841 |
| 20µL  | ea.  | 22842 |
| 25µL  | ea.  | 22843 |
| 50µL  | ea.  | 22844 |
| 100µL | ea.  | 22845 |
| 250µL | ea.  | 22846 |
| 500µL | ea.  | 22847 |
| 1cc   | ea.  | 22848 |
| 2cc   | ea.  | 22849 |
| 5cc   | ea.  | 22850 |

**Figure 1** Active sulfur compounds are stable in Sulfinert® treated sample cylinders.



for **more info**

Please request Applications Note **#59164B** for more details about storing sulfur compounds in Sulfinert® treated cylinders.

# Analysis of Nitrofurans in Honey

## Using LC/MS/MS and an Ultra C18 Column

By Eberhardt Kuhn, Ph.D.; International Marketing Specialist; and Becky Wittrig, Ph.D., HPLC Product Marketing Manager

- Sensitive detection of antibiotic metabolites in a complex matrix.
- Ultra C18 column assures the resolution needed for the LC/MS/MS method.
- Excellent peak shape at sub-ppb levels.

Nitrofurans are a class of veterinary antibiotics used to increase growth rate and prevent or treat disease in animals. Animals have been treated with antibiotics since the 1950s and, currently, about 45% of the antibiotics produced each year in the U.S. are administered to livestock. In Europe, this practice is illegal, because the inadvertent consumption of residual antibiotics in animal tissue, such as meat or liver, can lead to increased drug resistance or allergies in humans.

Nitrofurans have been detected not only in treated animals, but also in animal products, including honey. The low levels of these compounds and the complexity of honey as a matrix present challenges for the analysis of nitrofurans. In addition, nitrofurans are unstable and metabolize rapidly *in vivo*. Any analysis method for nitrofurans, therefore, must be able to separate and detect these metabolites. In the analysis of honey, it is of interest to quantify four nitrofurans: furazolidone, furaltadone, nitrofurazone, and nitrofurantoin, through their respective metabolites, 3-amino-2-oxazolidone (AOZ), 5-mofolinomethylmethyl-3-amino-2-oxazolidone (AMOZ), semicarbazide (SC) and 1-aminhydantoin (AHD). The method of choice for the analysis of nitrofurans and nitrofurans metabolites in honey is LC/MS/MS, with separation on a C18 column.

In this study, honey samples treated with the four nitrofurans metabolites were dissolved in water, then extracted with ethyl acetate. After centrifugation, the extract was evaporated and reconstituted in 125mM HCl, then derivatized with 2-nitrobenzaldehyde. After two liquid-liquid extractions with ethyl acetate, the extract was evaporated and reconstituted with mobile phase, filtered, and injected into the LC/MS/MS system. The column used for the analysis was a 100 x 2.1 mm, 3µm Ultra C18 column. For maximum sensitivity and specificity, a triple quadrupole analyzer was used, with electrospray ionization and selected reaction monitoring (SRM).

Results from the analysis of 0.3ppb nitrofurans metabolites in honey are shown in Figure 1. The Ultra C18 HPLC column is an excellent choice for this analysis. As a reliable general purpose column based on a high-purity, base-deactivated silica, its utility extends to other compounds that might be present in animal-derived matrixes, such as steroids and vitamins.

In analyses for nitrofurans antibiotics, an Ultra C18 HPLC column is an excellent choice, especially for analyzing trace levels of these compounds in a complex sample matrix.

### Acknowledgement

We are grateful to EIDOMET SRL, Restek distributor in Argentina, and application chemist Dr. Alejandro Albornoz, for the analytical work discussed in this article.

### Ultra C18 Column

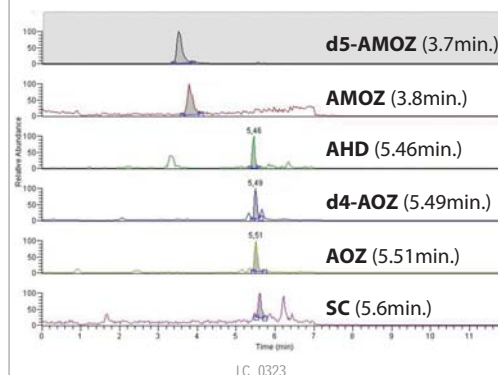
3µm Particles, 2.1mm ID  
100mm

cat. #  
9174312

For many other dimensions, refer to our catalog or visit our website.



**Figure 1** Nitrofurans metabolites in honey detected at 0.3ppb by LC/MS/MS, using an Ultra C18 column.



**Column:** Ultra C18  
Cat. #: 9174312  
Dimensions: 100 x 2.1mm  
Particle Size: 3µm  
Pore Size: 100Å

**Conditions:**  
Mobile phase: A: 0.05% formic acid in methanol  
B: 0.05% formic acid –  
5 mM NH<sub>4</sub> formate in water

| Time (min) | %B |
|------------|----|
| 0          | 90 |
| 2.5        | 90 |
| 5          | 10 |
| 10         | 10 |
| 12         | 90 |
| 15         | 90 |

Sample: 0.3ppb each analyte  
Flow: 200µL/min.  
Temp.: 30°C  
Det.: MS/MS triple quadrupoles  
(Thermo Finnigan Discovery)

### Analyzer Parameters:

Ion source: ESI (electrospray ionization)  
Only segment: 15 min.  
Polarity: positive  
Data type: centroid  
Scan mode: SRM product  
Scan width (m/z): 0.7  
Scan time (s): 0.25  
Peak width: Q1: within 0.7  
Q2: 0.7  
Collision gas pressure (mTorr): 1.5 (argon)  
Divert valve: active, with 3 positions  
Positions: 1° 2 min., 2° 8 min., 3° 5 min.

| Analyte | Prec. Ion | Prod. Ion | Collision E | Tube Lens |
|---------|-----------|-----------|-------------|-----------|
| AOZ     | 236       | 134       | 12 V        | 120       |
| AMOZ    | 335       | 291       | 10 V        | 100       |
| SC      | 209       | 166       | 12 V        | 80        |
| AHD     | 249       | 134       | 12 V        | 110       |

AMOZ = 3-amino-5-morpholinomethyl-2-oxazolidinone  
AHD = 1-aminohydantoin hydrochloride  
ADZ = 3-amino-2-oxazolidinone  
SC = semicarbazide

Data courtesy of Dr. Alejandro Albornoz, EIDOMET SRL, Buenos Aires.

## FID Gas Stations: FID-1000 & New FID-2500

### Convenient, Safe Source of Zero Air and Pure Hydrogen

By Kelli Ventura, GC Accessories Associate Product Manager

- Single unit produces zero grade air and 99.9995% pure hydrogen.
- Eliminates inconvenient and dangerous cylinders.
- Silent operation, minimal operator attention required.

Parker Balston FID-1000 and FID-2500 Gas Stations provide both UHP grade hydrogen fuel gas and zero grade air (<0.1ppm total hydrocarbons) for flame ionization detectors on gas chromatographs. The system is designed specifically to supply fuel gas to FIDs and to support flame thermionic and flame photometric detectors. The units produce zero air by purifying compressed air to a total hydrocarbon concentration of 0.1 ppm or less (measured as methane). The hydrogen generators produce hydrogen gas from deionized water, using the principle of electrolytic dissociation of water and hydrogen proton conduction through a proton exchange membrane cell.

These units are designed for universal operation. When ordering an FID Gas Station for use in countries other than the United States, simply add the appropriate international power cord suffix to the catalog number for the gas station.

| Description   | qty. | cat. # |
|---|------|--------|
| Model FID-1000 Gas Station (ideal for 1 - 2 FIDs)   | ea.  | 20177  |
| Model FID-2500 Gas Station (ideal for 5 - 6 FIDs)   | ea.  | 24913  |
| <b>Replacement Components for FID Gas Stations</b>  |      |        |
| Resin Bed Cartridge for FID-1000 and FID-2500 Hydrogen Generators   | ea.  | 24914  |
| Desiccant Cartridge   | ea.  | 21671  |
| FID Gas Station Maintenance Kit (Includes 1 desiccant cartridge, 1 resin bed cartridge, 1 filter cartridge) | ea.  | 24915  |
| <b>International Power Cord Sets</b>  |      |        |
| United Kingdom (230VAC, 50/50Hz)  | ea.  | -550   |
| European (230VAC, 50/60Hz)  | ea.  | -551   |
| IEC Connector Only (230VAC, 50/60Hz)  | ea.  | -552   |
| Japanese (200VAC, 50/60Hz)  | ea.  | -556   |
| Japanese for Zero Air (100VAC, 50/60Hz)   | ea.  | -553   |
| Japanese for Hydrogen (100VAC, 50/60Hz)   | ea.  | -554   |
| Japanese for Nitrogen (100VAC, 50/60Hz)   | ea.  | -555   |

Just add the proper suffix to the catalog number for the gas generator you are ordering.



#### Specifications - FID Gas Stations:

|                          |   |
|--------------------------|---|
| Hydrogen Purity:         | 99.9995%  |
| Zero Air Purity:         | FID-1000:<br>< 0.1ppm total hydrocarbons as methane<br>FID-2500:<br>< 0.05ppm total hydrocarbons as methane |
| Max. Hydrogen Flow Rate: | FID-1000: 90cc/min.<br>FID-2500: 250cc/min.   |
| Max. Zero Air Flow Rate: | FID-1000: 1000cc/min.<br>FID-2500: 2500cc/min.  |
| Power:                   | 120VAC/amp, 60Hz, 400 watts   |

|                           |   |
|---------------------------|---|
| Hydrogen Outlet Pressure: | 60 psig   |
| Zero Air Outlet Pressure: | 40-125 psig*                                      |
| Inlet Connection:         | 1/4" NPT (female)                                 |
| Outlet:                   | 1/8" compression                                  |
| Dimensions:               | 16.5" h x 10.5" w x 17" d<br>(42cm x 27cm x 43cm) |
| Weight:                   | 53 lbs. (24kg)                                    |

\*Zero air inlet requires minimum of 40psig compressed air pressure.

### built to international standards

Produced and supported by an ISO 9001 registered organization, Parker Balston hydrogen generators are built to meet the toughest laboratory standards - CSA, UL, CE, and IEC 1010.

## Volatile Organic Compounds by GC/MS

### Columns and Reference Mixes for US EPA 524.2 Revision IV.

By Christopher English, GC Innovations Group Leader, and Joseph Moodler, Analytical Reference Materials Technical Supervisor

- All 84 compounds listed in Method 524.2 resolved in 12 minutes, using an Rtx®-VMS column.
- MegaMix™ reference mix includes 73 compounds in stable solution.
- Three reference mixes include all 84 compounds.

Initially, US Environmental Protection Agency Method 524.2, a purge and trap, capillary GC/MS method, was used to identify 60 volatile aromatic and halogenated hydrocarbons in municipal drinking water. Revision 4.0 (1992) added 24 polar compounds and, in 2003, California allowed the addition of *tert*-amyl methyl ether (TAME), *tert*-butyl alcohol (TBA), ethyl-*tert*-butyl ether (ETBE), and 1,1,2-trichlorotrifluoroethane (Freon® 113) to the list of target compounds.

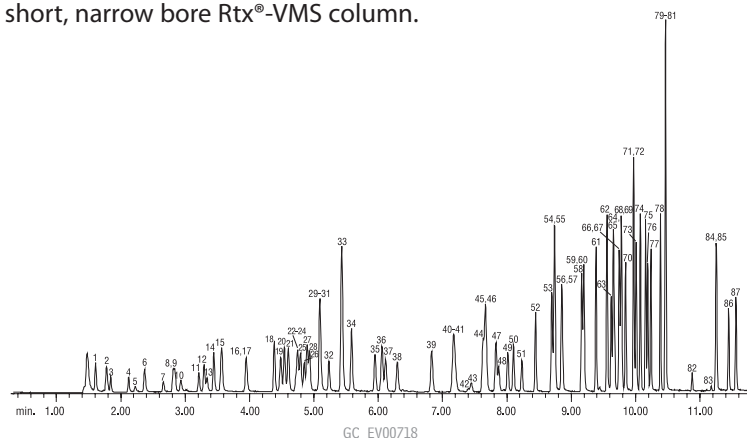
Of these 84 compounds, only the six gases and five ketones are not included in Drinking Water VOA MegaMix™, 524.2 Rev 4.1. To help ensure long-term stability of the mixes, we offer the ketones separately, in purge & trap methanol/water (90:10, v/v). This solvent system protects the keto groups and prevents acetal formation, which is more likely to occur in 100% methanol.



**Table 1** Method 524.2 analytes and internal standards.

|                                      |                                       |   |
|--------------------------------------|---------------------------------------|---|
| 1. dichlorodifluoromethane           | 30. methacrylonitrile                 | 59. styrene                             |
| 2. chloromethane                     | 31. benzene                           | 60. bromoform                           |
| 3. vinyl chloride                    | 32. 1,2-dichloroethane                | 61. isopropylbenzene                    |
| 4. bromomethane                      | 33. fluorobenzene (IS)                | 62. 4-bromofluorobenzene (IS)           |
| 5. chloroethane                      | 34. trichloroethene                   | 63. bromobenzene                        |
| 6. trichlorofluoromethane            | 35. dibromomethane                    | 64. <i>n</i> -propylbenzene             |
| 7. diethyl ether                     | 36. 1,2-dichloropropane               | 65. 1,1,2,2-tetrachloroethane           |
| 8. 1,1-dichloroethene                | 37. bromodichloromethane              | 66. 2-chlorotoluene                     |
| 9. carbon disulfide                  | 38. methyl methacrylate               | 67. 1,2,3-trichloropropane              |
| 10. iodomethane                      | 39. <i>cis</i> -1,3-dichloropropene   | 68. 1,3,5-trimethylbenzene              |
| 11. allyl chloride                   | 40. toluene                           | 69. <i>trans</i> -1,4-dichloro-2-butene |
| 12. methylene chloride               | 41. chloroacetonitrile                | 70. 4-chlorotoluene                     |
| 13. acetone                          | 42. 2-nitropropane                    | 71. <i>tert</i> -butylbenzene           |
| 14. <i>trans</i> -1,2-dichloroethene | 43. 1,1-dichloro-2-propanone          | 72. pentachloroethane                   |
| 15. methyl <i>tert</i> -butyl ether  | 44. 4-methyl-2-pentanone              | 73. 1,2,4-trimethylbenzene              |
| 16. 1,1-dichloroethane               | 45. tetrachloroethene                 | 74. <i>sec</i> -butylbenzene            |
| 17. acrylonitrile                    | 46. <i>trans</i> -1,3-dichloropropene | 75. <i>p</i> -isopropyltoluene          |
| 18. <i>cis</i> -1,2-dichloroethene   | 47. 1,1,2-trichloroethane             | 76. 1,3-dichlorobenzene                 |
| 19. 2,2-dichloropropane              | 48. ethyl methacrylate                | 77. 1,4-dichlorobenzene                 |
| 20. bromochloromethane               | 49. dibromochloromethane              | 78. <i>n</i> -butylbenzene              |
| 21. chloroform                       | 50. 1,3-dichloropropane               | 79. hexachloroethane                    |
| 22. methyl acrylate                  | 51. 1,2-dibromoethane                 | 80. 1,2-dichlorobenzene-d4 (IS)         |
| 23. carbon tetrachloride             | 52. 2-hexanone                        | 81. 1,2-dichlorobenzene                 |
| 24. tetrahydrofuran                  | 53. chlorobenzene                     | 82. 1,2-dibromo-3-chloropropane         |
| 25. 1,1,1-trichloroethane            | 54. ethylbenzene                      | 83. nitrobenzene                        |
| 26. 2-butanone                       | 55. 1,1,1,2-tetrachloroethane         | 84. hexachlorobutadiene                 |
| 27. 1,1-dichloropropene              | 56. <i>m</i> -xylene                  | 85. 1,2,4-trichlorobenzene              |
| 28. 1-chlorobutane                   | 57. <i>p</i> -xylene                  | 86. naphthalene                         |
| 29. propionitrile                    | 58. <i>o</i> -xylene                  | 87. 1,2,3-trichlorobenzene              |

Peaks 1-6 are components of cat. # 30439; peaks 13,26,43,44,52 are components of cat. # 30602; all other analytes except IS are components of cat. # 30601.

**Figure 1** 12-minute analysis of 84 volatile compounds, using a short, narrow bore Rtx®-VMS column.**Purge and Trap Conditions:**

Concentrator: Tekmar LSC-3100 purge and trap  
 Trap: Vocarb 3000 (type K)  
 Purge: 11 min. @ 40 mL/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: Silcosteel® transfer line  
 1:30 split at injection port. 1mm ID split injection sleeve (cat.# 20972).  
 Column: Rtx®-VMS, 30m, 0.25mm ID, 1.4µm (cat.# 19915)  
 Sample: 502.2 Calibration Mix #1 (cat.# 30042)  
 Drinking Water VOA MegaMix™, 524.2 Rev 4 (cat.# 30601)  
 524 Internal Standard/Surrogate Mix (cat.# 30201)  
 Ketone Mix, EPA Method 524.2 Rev 4.1 (cat.# 30602)  
 Compounds at 20 ppb each in 5mL RO water  
 (ketones at 50ppb; internal standards at 40ppb)  
 Inj. temp.: 250°C  
 Carrier gas: helium, constant flow  
 Flow rate: 1.1mL/min.  
 Dead time: 1.48 min. @ 40°C  
 Oven temp.: 40°C (hold 2 min.) to 85°C @ 14°C/min. (hold 2 min.)  
 to 220°C @ 30°C/min. (hold 4 min.).  
 Det: Agilent 5971A GC/MS  
 Transfer line temp.: 280°C  
 Scan range: 35-300 amu  
 Tune: PFTBA/BFB  
 Ionization: EI

A 30m, 0.25mm ID, 1.4µm Rtx®-VMS capillary column (cat.# 19915) is an excellent choice for analyzing the 84 target compounds (Figure 1). This narrow bore column improves resolution of traditionally coeluting compounds, such as carbon tetrachloride / 1,1,1-trichloroethane, while shortening the analysis time. Analysis time is less than 12 minutes, and the cycle time is 16 minutes, which is well below the cycle time of a standard purge and trap system. This allows the fastest run-time attainable using a Tekmar 3100 purge and trap unit coupled to a single GC. A slower initial temperature ramp rate makes additional resolution possible.

We recommend using the 30m, 0.25mm ID column for best resolution of the target gases. At 20ppb in 5mL water, the gases are better than 90% resolved, using an initial temperature of 40°C (Figure 1). We encourage laboratories using either dual purge and trap technology or newer purge and trap systems with rapid cycle times to use a 20m, 0.18mm ID, 1.0µm Rtx®-VMS column for sub-10 minute runtimes.<sup>1,2</sup> Whatever your system for analyzing volatiles, we offer the columns, analytical standards, GC accessories, and technical knowledge to get your laboratory running these analyses quickly and accurately.

**References**

- Butler J.C., E. Phillips, and M. Conoley Application Note AN9197, Thermo Electron Corporation, 2215 Grand Avenue Parkway, Austin, TX., 2003.
- A.L. Hilling and G. Smith, Environmental Testing & Analysis, 10 (3),15-19, 2001.

**Rtx®-VMS (fused silica)**

| ID     | df (µm) | temp. limits     | length   | cat. # |
|--------|---------|------------------|----------|--------|
| 0.18mm | 1.00    | -40 to 240/260°C | 20-Meter | 49914  |
| 0.25mm | 1.40    | -40 to 240/260°C | 30-Meter | 19915  |

**Drinking Water VOA MegaMix™, 524.2 Rev. 4.1**

(73 components—see Table 1)

2,000µg/mL each in P&T methanol, 1mL/ampul  
 cat. # 30601 (ea.)

**Ketones Mix, 524.2 Rev. 4.1** (5 components)

acetone 2-hexanone  
 2-butanone (MEK) 4-methyl-2-pentanone (MIBK)  
 1,1-dichloro-2-propanone  
 5,000µg/mL each in 90% P&T methanol:10% water, 1mL/ampul  
 cat. # 30602 (ea.)

**502.2 Calibration Mix #1 (gases)**

bromomethane dichlorodifluoromethane  
 chloroethane trichlorofluoromethane  
 chloromethane vinyl chloride  
 200µg/mL each in P&T methanol, 1mL/ampul  
 cat. # 30439 (ea.)  
 2,000µg/mL each in P&T methanol, 1mL/ampul  
 cat. # 30042 (ea.)

For individual solutions of *tert*-amyl methyl ether, *tert*-butyl alcohol, ethyl-*tert*-butyl ether, and 1,1,1-trichlorotrifluoroethane (Freon® 113), and for internal and surrogate standards, please see our catalog, or visit our website.

# Instrument Innovations!

Restek is your #1 source for GC consumables and supplies

by Donna Lidgett, GC Accessories Product Marketing Manager

## From Injector to Detector

Restek designs, develops, and markets OEM-equivalent parts and supplies for Agilent, PerkinElmer, Shimadzu, Thermo Finnigan or Varian GC systems. Restek consumables and parts meet or exceed OEM performance, helping you maintain optimum system performance and giving you the convenience and economy of one-stop shopping for all your GC needs.

Check out some of our newest consumables. . .



### new! EPC Test Kit for Agilent 6890 GCs

- Kit includes 3 o-rings, 2 plugs, 1 mounting screw and 1 test block.

| Description                       | Similar to Agilent part # | qty. | cat.# |
|-----------------------------------|---------------------------|------|-------|
| EPC Test Kit for Agilent 6890 GCs | G1530-60960*              | kit  | 24323 |

\*Similar to Agilent part # G1530-60960, but not exact equivalent. Kits differ in parts.



Clear anodized aluminum and high-quality stainless steel.

### new! Septum Nut for Shimadzu 17A & 2010 GCs

- One piece design for ease of installation and removal.

| Description                            | Similar to Shimadzu part # | qty. | cat.# |
|--|----------------------------|------|-------|
| Septum Nut for Shimadzu 17A & 2010 GCs | 221-41286-00               | ea.  | 22079 |
|  | 221-44584-00               |      |       |



High quality stainless steel.

### new! FID Jet for PerkinElmer Auto SYS™ XL

| Description                          | Similar to PE part # | qty. | cat.# |
|--------------------------------------|----------------------|------|-------|
| FID Jet for PerkinElmer Auto SYS™ XL | N6100361             | ea.  | 23038 |



High quality stainless steel.

### new! FID Capillary Column Adaptor for PerkinElmer Auto SYS™ XL

| Description   | Similar to PE part # | qty. | cat.# |
|---|----------------------|------|-------|
| <b>For use with PE style capillary nuts</b>               |                      |      |       |
| FID Capillary Column Adaptor for PerkinElmer Auto SYS™ XL | N6120020             | ea.  | 22608 |
| <b>For use with 1/8" compression style nuts</b>           |                      |      |       |
| FID Capillary Column Adaptor for PerkinElmer Auto SYS™ XL | —                    | ea.  | 22609 |



Clear anodized aluminum/  
High quality stainless steel.

### new! Septum Cap for PerkinElmer Auto SYS™ XL

| Description                             | Similar to PE part # | qty. | cat.# |
|---|----------------------|------|-------|
| Septum Cap for PerkinElmer Auto SYS™ XL | N6100153             | ea.  | 22322 |



High quality stainless steel.

### new! Injector Adaptor for PerkinElmer Auto SYS™ XL

| Description   | Similar to PE part # | qty. | cat.# |
|---|----------------------|------|-------|
| <b>For use with PE style capillary nuts</b>                   |                      |      |       |
| Injector Adaptor for PerkinElmer Auto SYS™ XL                 | N6100157             | ea.  | 22318 |
| Siltek®-Treated Injector Adaptor for PerkinElmer Auto SYS™ XL | —                    | ea.  | 22320 |
| <b>For use with 1/8" compression style nuts</b>               |                      |      |       |
| Injector Adaptor for PerkinElmer Auto SYS™ XL                 | —                    | ea.  | 22319 |
| Siltek®-Treated Injector Adaptor for PerkinElmer Auto SYS™ XL | —                    | ea.  | 22321 |



Siltek®-treated version for increased inertness.

# COOL TOOLS!

Restek Innovations Save You Time and Money

## A Clean Square Cut...

The key to obtaining a leak-tight seal in a Press-Tight® connector—or in other connecting devices that make a compression seal with the end of the column—is a clean, right angle cut at the end of the column. If you use an unsuitable device to cut your columns, you run the risk of angled cuts or chipped or jagged edges that will not seal effectively, or even crushing the end of the column. We offer a selection of scoring tools that will help you properly cut your columns.



Make a clean, square cut for optimum performance. The cut on the right will produce a poor seal.

## Scoring Wafer with Handle

- Ceramic wafer is serrated on one side and straight-edged on the other to cut both fused silica and metal tubing cleanly.
- Unique, ergonomic handle is made of soft, comfortable rubber.



Hold tubing firmly in one hand, allowing about two inches to extend freely. Hold the scoring wafer at a 45° angle to the tubing. Exert just enough pressure to put a slight arc in the tubing. Pull perpendicularly across the tubing.



The tubing should fall off on its own, or it should easily break at the score with a slight tap of the wafer.



Check the cut against the white of the scoring wafer. Look for a clean, square cut.



Make clean, square cuts!

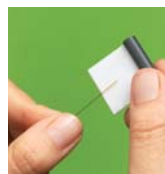
| Description               | qty.  | cat.# |
|---------------------------|-------|-------|
| Scoring Wafer with Handle | 2-pk. | 23015 |

## Ceramic Scoring Wafers

- Four straight scoring edges for cutting fused silica tubing and four serrated edges for cutting MXT® metal capillary columns.
- Sure-grip handle included.



Exert just enough pressure to put a slight arc in the tubing. The tubing should fall off or break with a slight tap of the wafer.



Check the cut against the white of the scoring wafer. Look for a clean, square cut.



| Description            | qty.  | cat.# |
|------------------------|-------|-------|
| Ceramic Scoring Wafers | 5-pk. | 20116 |

## Sapphire Scribe

- Cuts fused silica tubing.
- Produces a clean, square cut.



One quick stroke...



...and tap leaves a clean, square end.



| Description     | qty. | cat.# |
|-----------------|------|-------|
| Sapphire Scribe | ea.  | 20182 |

## Shortix™ Capillary GC Column Cutter

- Consistently make precise, clean, square cuts with a diamond blade.
- Built-in magnifier to verify square cut.
- Use with 0.25mm ID to 0.53mm ID tubing (0.78mm OD max.).
- Maintenance kit includes diamond cutting wheel, O-rings, and a tool to open the column cutter.



Maintenance Kit for Shortix™ Capillary GC Column Cutter



| Description  | qty. | cat.# |
|--|------|-------|
| Shortix Capillary GC Column Cutter                     | ea.  | 23026 |
| Maintenance Kit for Shortix Capillary GC Column Cutter | kit  | 23027 |

## Items of Interest

Compiled by the Advantage Staff

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### Detecting Adulterants in Butter



Investigators in the Dipartimento di Scienza degli Alimenti, Università degli Studi di Napoli "Federico II" (Via Università, 100-80055 Portici (NA), Italy) have proposed a new analytical method for detecting extraneous animal fats or vegetable oils added to butter.

Application of a HRGC Method on Capillary Column Rtx® 65-TG for Triglyceride Analysis to Monitor Butter Purity, by Daniele Naviglio and Carlo Raia, was published in Analytical Letters, Vol. 36, No. 14, 2003 (pages 3063-3094). Relative to the official EU method for detecting added animal fats, such as lard or tallow, the authors propose the new method is simple, rapid, and precise, even when quantities of added fats are minimal. The new method is easy to follow, even by nonspecialists. Naviglio and Raia developed their method using a 30m, 0.25mm ID, 0.25um Rtx®-65TG Restek column.

Contact the authors at their university address, above, or by e-mail: [danielenaviglio@inwind.it](mailto:danielenaviglio@inwind.it)

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