

# RESTEK

## The Advantage

### Allure C18 HPLC Column

#### Specialized Columns for LC-MS

by Keith Duff & Dave Bell

Innovators of  
High Resolution  
Chromatography  
Products

#### in this issue

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

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

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

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

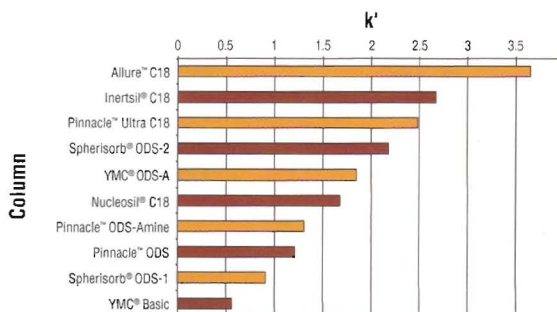
Table I shows increased retention for neutral compounds obtained on the Allure™ C18 phase, as compared to other

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

Continued on page 2.

Table I:

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



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

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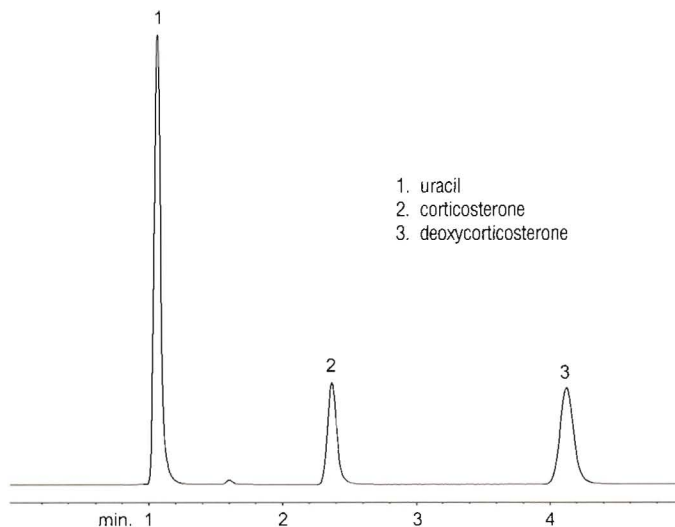


# Allure™ C18 HPLC Column

Continued from page 1.

Figure 1:

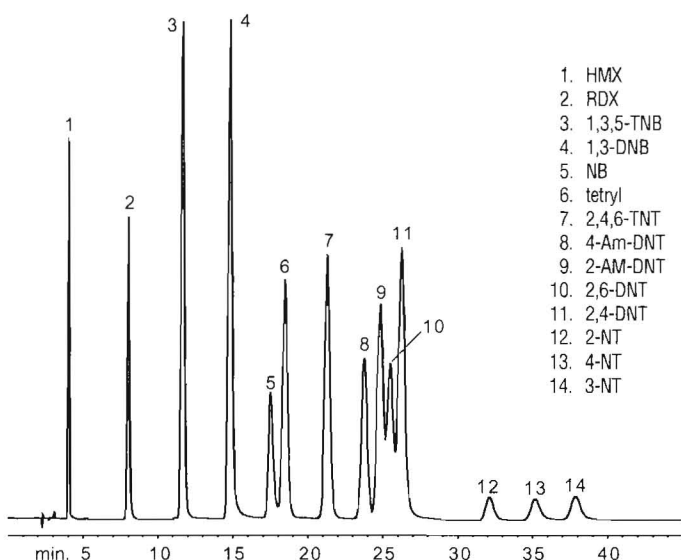
Corticosteroids on Allure™ C18 Column Show Higher Retention of Less Polar Compounds



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

Figure 2:

All EPA-8330 Explosives are Separated on One Column



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

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

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

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

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

## Product Listing:

### Allure™ C18 HPLC Columns

5µm particles, 60Å pore size

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



# 100% Bonded, Unbreakable MXT®-PLOT Columns

by Andy Schuyler

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



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

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

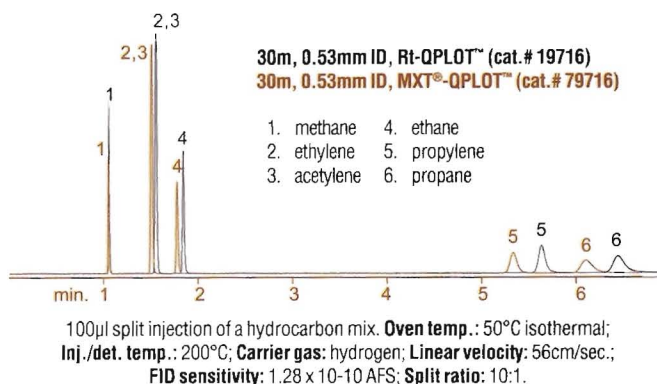
mally stable, easy to handle, and will not break.

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

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

**Figure 1:**

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



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

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

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

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

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

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

**Table I:**

## Ideal Uses of MXT®-PLOT Columns.

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

## Product List

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

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# Optimizing Massachusetts Volatile Petroleum Hydrocarbon GC Analysis

by Christopher English and Eric Steindl

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

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

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

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

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

## Difficulties with the Analysis

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

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

## Column Selection

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

Figure 1:

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

Figure 1A: PID

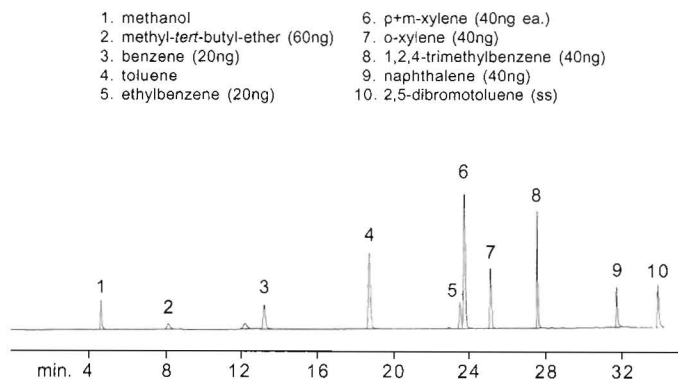
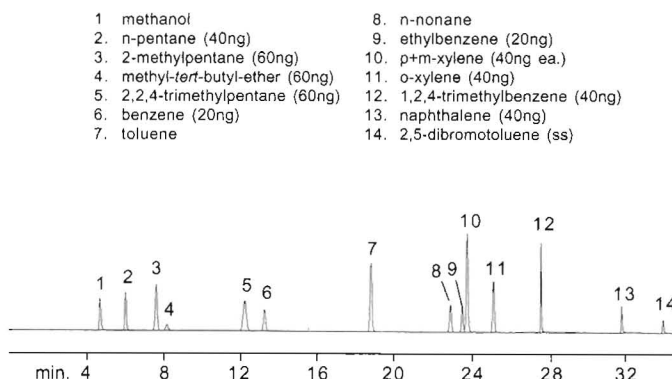


Figure 1B: FID



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



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

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

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

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

#### Standards

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

#### Product Listing:

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

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

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

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

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

#### Analytical Reference Materials

##### MA VPH Standard

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

in P&T methanol, 1ml/ampul

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

##### MA VPH Standard with Surrogate

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

in P&T methanol, 1ml/ampul

\*surrogate

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

##### MA VPH Surrogate Standard

2,5-dibromotoluene

1000µg/ml in P&T methanol, 1ml/ampul

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

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

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

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

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

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

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

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# GC-MS Analysis of Alkyl Phenols and Bisphenol A Endocrine Disrupters\*

by Frank Dorman and Chris Cox

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

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

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

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

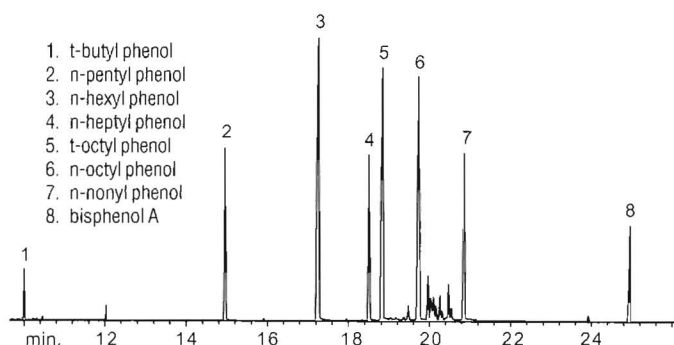
and US EPA Methods 3540 (soxhlet) or 3550 (sonication) for solid matrices. Alternatively, SPE, microwave, and pressurized fluid extraction also could be used. These methods produce a methylene chloride extract, which is then dried using granular sodium sulfate and concentrated to a final volume. For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

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

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

**Figure 1:**

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



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

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

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

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

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

## References

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

## Product Listing:

### Rtx®-5MS Column

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

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

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# Koni's Korner

## Do We Need a Chromatography School?

by Dr. Konrad Grob

**Sometimes things  
need to be said  
in a pointed way.  
The following is  
one of those things.  
If even less than 50%  
is applicable, then  
it is enough to  
sound an alarm.**

### *The Slow Sinking of Chromatography*

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

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

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

### *Lab Work Degrades*

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

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

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

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

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

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

### *A Need for Better Education*

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

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

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

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



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

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

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

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

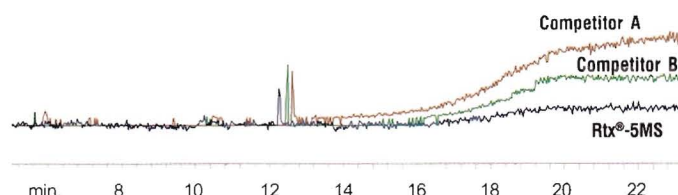
The level of column bleed affects the sensitivity of any MS, especially ion trap instruments, which utilize automatic gain control. As the level of column bleed increases, so does the signal—from bleed ions in the

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

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

Figure 1:

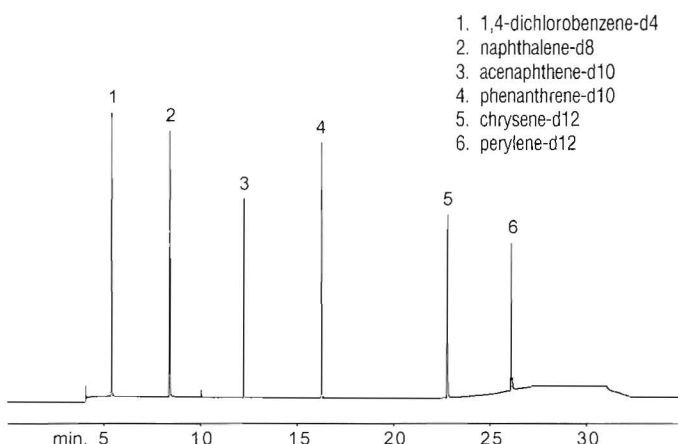
The Restek Rtx®-5MS Exhibits the Lowest Bleed.



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

Figure 2:

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



30m, 0.25mm ID, 0.25µm Rtx®-200MS (cat.# 15623); 1.0µl split injection of a semi-volatile internal standard; On-column conc.: 5ng; Oven temp.: 40°C to 300°C @ 10°C/min. (hold 5 min.); Inj. program: 40°C to 280°C @ 50°C/min.; Det. type: Ion Trap GC/MS; Ionization: EI; Carrier gas: Helium; Scan range: 50-400m/z; Linear velocity: 27cm/sec. @ 40°C; Split ratio: 40:1.

In addition to Restek's new chemistry, our rigorous QA testing includes MS polymer batch testing and individual column testing for inertness, efficiency, and bleed using a

special test mix designed for environmental analysis. This combination ensures that each MS column exceeds requirements of the most sensitive mass spectrometers.



### Rtx®-1MS

*Best for GC/MS Analysis of Air Pollutants*

| ID<br>(mm) | df<br>(µm) | temp. limits<br>(°C) | length      |             |
|------------|------------|----------------------|-------------|-------------|
|            |            |                      | 15-meter    | 30-meter    |
| 0.25       | 0.10       | -60 to 330/350       | 11605 \$270 | 11608 \$425 |
|            | 0.25       | -60 to 330/350       | 11620 \$260 | 11623 \$415 |
|            | 0.50       | -60 to 330/350       | 11635 \$260 | 11638 \$415 |
|            | 1.00       | -60 to 325/350       | 11650 \$260 | 11653 \$415 |
| 0.32       | 0.10       | -60 to 330/350       | 11606 \$290 | 11609 \$455 |
|            | 0.25       | -60 to 330/350       | 11621 \$280 | 11624 \$445 |
|            | 0.50       | -60 to 330/350       | 11636 \$280 | 11639 \$445 |
|            | 1.00       | -60 to 325/350       | 11651 \$280 | 11654 \$445 |
| 0.53       | 0.50       | -60 to 320/340       | 11637 \$285 | 11640 \$495 |
|            | 1.00       | -60 to 320/340       | 11652 \$285 | 11655 \$495 |
|            | 1.50       | -60 to 310/330       | 11667 \$285 | 11670 \$495 |

### Rtx®-5MS

*Ideal for Semi-Volatile Analyses*

| ID<br>(mm) | df<br>(µm) | temp. limits<br>(°C) | length             |             |
|------------|------------|----------------------|--------------------|-------------|
|            |            |                      | 15-meter           | 30-meter    |
| 0.25       | 0.10       | -60 to 330/350       | 12605 \$270        | 12608 \$425 |
|            | 0.25       | -60 to 330/350       | 12620 \$260        | 12623 \$415 |
|            | 0.50       | -60 to 330/350       | 12635 \$260        | 12638 \$415 |
|            | 1.00       | -60 to 325/350       | 12650 \$260        | 12653 \$415 |
| 0.32       | 0.10       | -60 to 330/350       | 12606 \$290        | 12609 \$455 |
|            | 0.25       | -60 to 330/350       | 12621 \$280        | 12624 \$445 |
|            | 0.50       | -60 to 330/350       | 12636 \$280        | 12639 \$445 |
|            | 1.00       | -60 to 325/350       | 12651 \$280        | 12654 \$445 |
| 0.53       | 0.50       | -60 to 320/340       | 12637 \$285        | 12640 \$495 |
|            | 1.00       | -60 to 320/340       | 12652 \$285        | 12655 \$495 |
|            | 1.50       | -60 to 310/330       | 12667 \$285        | 12670 \$495 |
| ID<br>(mm) | df<br>(µm) | temp. limits<br>(°C) | length<br>60-meter |             |
| 0.25       | 0.10       | -60 to 330/350       | 12611              | \$665       |
|            | 0.25       | -60 to 330/350       | 12626              | \$665       |
|            | 0.50       | -60 to 330/350       | 12641              | \$665       |
| 0.32       | 0.10       | -60 to 330/350       | 12612              | \$725       |
|            | 0.25       | -60 to 330/350°C     | 12627              | \$725       |
|            | 0.50       | -60 to 330/350°C     | 12642              | \$725       |

### Rtx®-35MS

*Excellent Confirmational Column for MS Analyses*

| ID<br>(mm) | df<br>(µm) | temp. limits<br>(°C) | length      |             |
|------------|------------|----------------------|-------------|-------------|
|            |            |                      | 15-meter    | 30-meter    |
| 0.25       | 0.10       | -60 to 330/350       | 14605 \$270 | 14608 \$425 |
|            | 0.25       | -60 to 330/350       | 14620 \$260 | 14623 \$415 |
|            | 0.50       | -60 to 330/350       | 14635 \$260 | 14638 \$415 |
|            | 1.00       | -60 to 325/350       | 14650 \$260 | 14653 \$415 |
| 0.32       | 0.10       | -60 to 330/350       | 14606 \$290 | 14609 \$455 |
|            | 0.25       | -60 to 330/350       | 14621 \$280 | 14624 \$445 |
|            | 0.50       | -60 to 330/350       | 14636 \$280 | 14639 \$445 |
|            | 1.00       | -60 to 325/350       | 14651 \$280 | 14654 \$445 |
| 0.53       | 0.50       | -60 to 320/340       | 14637 \$285 | 14640 \$495 |
|            | 1.00       | -60 to 320/340       | 14652 \$285 | 14655 \$495 |
|            | 1.50       | -60 to 310/330       | 14667 \$285 | 14670 \$495 |

### Rtx®-200MS

*Preferred for Ion Trap Analyses*

| ID<br>(mm) | df<br>(µm) | temp. limits<br>(°C) | length      |             |
|------------|------------|----------------------|-------------|-------------|
|            |            |                      | 15-meter    | 30-meter    |
| 0.25       | 0.10       | -20 to 320/340       | 15605 \$270 | 15608 \$425 |
|            | 0.25       | -20 to 320/340       | 15620 \$260 | 15623 \$415 |
|            | 0.50       | -20 to 310/330       | 15635 \$260 | 15638 \$415 |
|            | 1.00       | -20 to 290/310       | 15650 \$260 | 15653 \$415 |
| 0.32       | 0.10       | -20 to 320/340       | 15606 \$290 | 15609 \$455 |
|            | 0.25       | -20 to 320/340       | 15621 \$280 | 15624 \$445 |
|            | 0.50       | -20 to 310/330       | 15636 \$280 | 15639 \$445 |
|            | 1.00       | -20 to 290/310       | 15651 \$280 | 15654 \$445 |
| 0.53       | 0.50       | -20 to 300/320       | 15637 \$285 | 15640 \$495 |
|            | 1.00       | -20 to 290/310       | 15652 \$285 | 15655 \$495 |
|            | 1.50       | -20 to 280/300       | 15667 \$285 | 15670 \$495 |

**"We are confident that our MS columns will perform to the level of quality you've come to expect from Restek. Call Restek, or contact your local distributor, and ask about a 30-day free evaluation!"**

**Rick Crago**  
Fused Silica  
Product Line Manager



**Restek Trademarks:** Allure, Diskcover, IceBlue, Leak Detective, MXT, MXT-QPLOT, Pinnacle, RedLite, Resprep, Res-Sil, Rt-QPLOT, Rtx, Silcosteel, Stabilwax, Thermolite, and the Restek logo.

**Restek Service Marks:** Restek Analytical Services and The Future of Chromatography.

**Other Trademarks & Registered Trademarks:** BioRex (Bio-Rad Laboratories), Carbowax (Union Carbide Corp.), Durapak (Waters), Hyperbond (Hypersil), Hypersil (Hypersil), HyPurity (Hypersil), Inertsil (GL Sciences), KEL-F (3M Co., Chemical Div.), µBondapak (Waters), Microseal (Merlin Instrument Co.), Nucleosil (Macherey-Nagel), Porasil (Waters), Spherisorb (Phase Separations), Tekmar (Tekmar, Co.), VOCARB (Supelco, Inc.), and YMC (Waters).

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# Restek and Hypersil™ Announce Strategic Partnership

- Expanded column line gives Restek customers "one-stop shopping."
- Expanded technical support for Hypersil™ column purchasers.
- RAS cGMP method development and validation services.
- Laboratory and regulatory services.
- Continuous, aggressive R&D to develop new phases.

Restek Corporation is proud to formally announce a strategic partnership with Hypersil®. By forming this relationship, Restek can now offer the entire line of superior-quality Hypersil® columns. We are especially proud to offer the new HyPurity™ Elite columns, which combine the unrivaled chromatographic performance of a robust bonded phase on a high-purity silica.

## Hyperbond™ Columns

*Manufactured by Hypersil*

**Chromatographic Properties:** Since  $\mu$ Bondapak™ is only available from one manufacturer, a demand has grown for a more cost-effective column of equivalent performance from an alternative source. The Hyperbond™ C18 column provides equivalent performance and selectivity, at a lower cost.

**Physical Characteristics:** Particle size: 10 $\mu$ m, irregular; Not end-capped; Pore size: 150Å; pH Range: 2.5 to 7.5; Average carbon load: 10%

| C18           |             |            |       |
|---------------|-------------|------------|-------|
| Particle size | Dimensions  | cat.#      | price |
| 10 $\mu$ m    | 300 x 3.9mm | 9260000301 | \$328 |
| Guard Column  |             |            |       |
| 10 $\mu$ m    | 30 x 3.9mm  | 9260000307 | \$210 |

## HyPurity™ Elite Columns

*Manufactured by Hypersil*

**Chromatographic Properties:** The HyPurity™ Elite C18 column is the first column to be based upon the new HyPurity™ silica\*. By bonding this unique, new HyPurity™ silica using a specialized procedure, universally applicable HyPurity™ Elite C18 columns are produced. These columns are then scrutinized using the extensive testing procedure established specifically for Hypersil® Elite columns. The HyPurity™ Elite column is the ultimate in chromatographic validation.

**Physical Characteristics:** Particle size: 5 $\mu$ m, spherical; Pore size: 180Å; pH Range: 0.9 to 9.0; Average carbon load: 13%

| Elite C18       |             |            |             |
|-----------------|-------------|------------|-------------|
| Particle size   | Dimensions  | cat.#      | price       |
| 5 $\mu$ m       | 30 x 4.6mm  | 9222105066 | \$350       |
| 5 $\mu$ m       | 50 x 4.6mm  | 9222105065 | \$350       |
| 5 $\mu$ m       | 50 x 2.1mm  | 9222105089 | \$350       |
| 5 $\mu$ m       | 100 x 3.0mm | 9222105078 | \$350       |
| 5 $\mu$ m       | 100 x 4.6mm | 9222105064 | \$350       |
| 5 $\mu$ m       | 100 x 2.1mm | 9222105088 | \$350       |
| 5 $\mu$ m       | 125 x 4.0mm | 9222105071 | \$375       |
| 5 $\mu$ m       | 150 x 3.0mm | 9222105077 | \$375       |
| 5 $\mu$ m       | 150 x 4.6mm | 9222105062 | \$375       |
| 5 $\mu$ m       | 150 x 2.1mm | 9222105087 | \$375       |
| 5 $\mu$ m       | 250 x 3.0mm | 9222105075 | \$400       |
| 5 $\mu$ m       | 250 x 4.6mm | 9222105060 | \$400       |
| 5 $\mu$ m       | 250 x 2.1mm | 9222105085 | \$400       |
| 5 $\mu$ m       | 250 x 4.0mm | 9222105070 | \$400       |
| Guard Cartridge |             |            |             |
| 5 $\mu$ m       | 10 x 4.6mm  | 9222105105 | \$370/4-pk. |

\*This pure silica is essentially metal-free, and the C18 phase is exceptionally stable even at pH extremes. The HyPurity™ manufacturing process ensures the highest levels of reproducibility and quality, and the homogeneous surface ensures uniform bonding coverage. Only the purest grade solvents and raw materials are used, and each component is quality assured.



Restek Analytical Services<sup>SM</sup> (RAS) is a unique chromatography laboratory designed to deliver both quality services and quality products. The ultimate goal of RAS is to provide total security to the customer by being the sole product-service supplier, beginning with stationary phase raw material and ending with a validated chromatographic method. By offering this customized product-service package to our clients, RAS redefines the industry and truly becomes "The Future of Chromatography<sup>SM</sup>."

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## Hypersil® HPLC Columns

Manufactured by Hypersil

**Chromatographic Properties:** Hypersil® stationary phases are widely recognized as an industry standard in HPLC. Hypersil® is manufactured under strict quality guidelines and standards as indicated by BS 5750 part 1 and ISO 9001 accreditation.

**Physical Characteristics:** Particle size: 5µm, spherical; Fully end-capped; Pore size: 120Å (130Å for BDS C18); pH Range: 2.5 to 7.5; Average carbon load: [ODS 10%] [BDS C18 12%] [C8 7%] [BDS C8 7%] [CPS-2 4%] [APS-2 2%] [Phenyl-2 5%] [SAS 3%]

| ODS             |              |            |             |
|-----------------|--------------|------------|-------------|
| Particle size   | Dimensions   | cat.#      | price       |
| 3µm             | 100 x 4.6mm  | 9230103064 | \$305       |
| 3µm             | 150 x 4.6mm  | 9230103062 | \$315       |
| 5µm             | 150 x 4.6mm  | 9230105062 | \$285       |
| 5µm             | 250 x 4.6mm  | 9230105060 | \$310       |
| 5µm             | 100 x 10mm   | 9230105122 | \$800       |
| 5µm             | 100 x 21.2mm | 9230105127 | \$1800      |
| 5µm             | 150 x 10mm   | 9230105121 | \$900       |
| 5µm             | 150 x 21.2mm | 9230105126 | \$2000      |
| 5µm             | 250 x 10mm   | 9230105120 | \$1000      |
| 5µm             | 250 x 21.2mm | 9230105125 | \$2000      |
| Guard Cartridge |              |            |             |
| 5µm             | 10 x 4.6mm   | 9230105105 | \$140/4-pk. |
| BDS C18         |              |            |             |
| 3µm             | 50 x 4.6mm   | 9228103123 | \$330       |
| 3µm             | 100 x 4.6mm  | 9228103064 | \$330       |
| 3µm             | 150 x 4.6mm  | 9228103062 | \$350       |
| 5µm             | 100 x 10mm   | 9228105122 | \$800       |
| 5µm             | 100 x 20mm   | 9228105127 | \$1000      |
| 5µm             | 150 x 10mm   | 9228105121 | \$1500      |
| 5µm             | 150 x 21.2mm | 9228105126 | \$3000      |
| 5µm             | 250 x 10mm   | 9228105120 | \$2000      |
| 5µm             | 250 x 21.2mm | 9228105125 | \$4500      |
| Guard Cartridge |              |            |             |
| 5µm             | 10 x 4.6mm   | 9228105105 | \$140/4-pk. |
| MOS-2 (C8)      |              |            |             |
| 3µm             | 100 x 4.6mm  | 9230303064 | \$305       |
| 3µm             | 150 x 4.6mm  | 9230303062 | \$315       |
| 5µm             | 150 x 4.6mm  | 9230305062 | \$285       |
| 5µm             | 250 x 4.6mm  | 9230305060 | \$310       |
| BDS (C8)        |              |            |             |
| 3µm             | 100 x 4.6mm  | 9228203064 | \$330       |
| 3µm             | 150 x 4.6mm  | 9228203062 | \$350       |
| 5µm             | 150 x 4.6mm  | 9228205062 | \$325       |
| 5µm             | 250 x 4.6mm  | 9228205060 | \$350       |



Please call to request the new HPLC products and services catalog (lit. cat.# 59968).

### Hypersil® HPLC Columns (cont.)

| CPS-2 (Cyano)                    |             |            |       |
|----------------------------------|-------------|------------|-------|
| 5µm                              | 150 x 4.6mm | 9231805062 | \$285 |
| 5µm                              | 250 x 4.6mm | 9231805060 | \$310 |
| APS-2 (Amino)                    |             |            |       |
| 3µm                              | 100 x 4.6mm | 9230703064 | \$305 |
| 3µm                              | 150 x 4.6mm | 9230703062 | \$315 |
| 5µm                              | 150 x 4.6mm | 9230705062 | \$285 |
| 5µm                              | 250 x 4.6mm | 9230705060 | \$310 |
| Phenyl-2                         |             |            |       |
| 5µm                              | 150 x 4.6mm | 9231905062 | \$285 |
| 5µm                              | 250 x 4.6mm | 9231905060 | \$310 |
| Silica                           |             |            |       |
| 3µm                              | 100 x 4.6mm | 9230003064 | \$295 |
| 3µm                              | 150 x 4.6mm | 9230003062 | \$305 |
| 5µm                              | 150 x 4.6mm | 9230005062 | \$275 |
| 5µm                              | 250 x 4.6mm | 9230005060 | \$300 |
| SAS Methyl                       |             |            |       |
| 3µm                              | 100 x 4.6mm | 9230503064 | \$305 |
| 3µm                              | 150 x 4.6mm | 9230503062 | \$315 |
| 5µm                              | 150 x 4.6mm | 9230505062 | \$285 |
| 5µm                              | 250 x 4.6mm | 9230505060 | \$310 |
| Carbamate                        |             |            |       |
| 5µm                              | 250 x 4.6mm | 9231305060 | \$375 |
| Hypersil® Guard Cartridge Holder |             |            |       |
|                                  | 10 x 4.6mm  | 25064      | \$75  |

Note: Other phases and column dimensions are available.

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# Sample Cylinders & Valves

## Silcosteel®-Treated Sample Cylinders

- Sizes range from 75cc to 1000cc.
- Made from 304 stainless steel.
- Department of Transportation rated to 1800psi at room temperature.
- Silcosteel®-treated for use with active samples.

Whether you collect hydrocarbons in refineries or reactive compounds in chemical reaction vessels, use Silcosteel®-treated sample cylinders to ensure sample integrity for sulfur compounds and other trace reactive compounds.

Restek's Silcosteel® process deposits a thin, inert layer on the stainless steel, resulting in a very inert surface. Even trace levels of sulfur compounds can be successfully collected and stored in these cylinders without significant loss (**Figure 1**). Silcosteel®-treated sample cylinders combine the inertness of glass with the strength of stainless steel and are ideal for the analysis of difficult sulfur compounds.

### Product Listing:

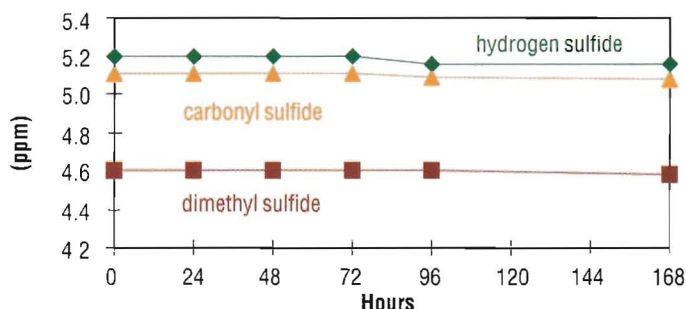
#### Silcosteel®-Treated Sample Canisters

| Volume | cat.# | price |
|--------|-------|-------|
| 75cc   | 24271 | \$125 |
| 150cc  | 24272 | \$140 |
| 300cc  | 24273 | \$150 |
| 500cc  | 24274 | \$180 |
| 1000cc | 24275 | \$320 |



**Figure 1:**

Storage of sulfur compounds at ~5.0ppm in a Silcosteel®-treated stainless steel cylinder for seven days shows no significant compound loss.



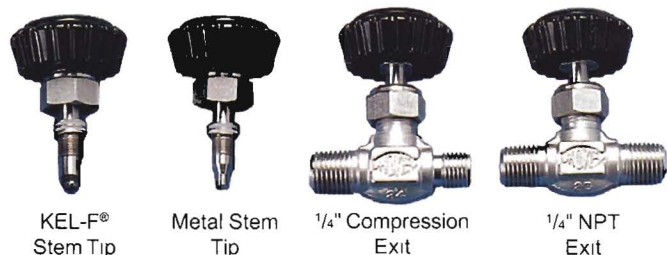
## Silcosteel®-Treated Hoke Sample Cylinder Valves for Maximum Inertness

Hoke valves (to use with the above sample cylinders) are now offered with Silcosteel®-treatment of all parts that come in contact with the sample. The metal stem tip is Hoke model #3712, and the KEL-F® stem tip is Hoke model #3752. Both valves are offered with either a 1/4-inch NPT outlet fitting or a 1/4-inch compression fitting. Maximum operating pressure is 5000 psig. The temperature range for the metal stem tip is -65°F to 450°F, and the KEL-F® stem tip is -20°F to 250°F.

### Product Listing:

#### Silcosteel®-Treated Hoke Sample Cylinder Valves

| Description                           | cat.# | price |
|---------------------------------------|-------|-------|
| KEL-F stem tip: 1/4" male NPT exit    | 24278 | \$130 |
| KEL-F stem tip: 1/4" compression exit | 24279 | \$130 |
| Metal stem tip: 1/4" male NPT exit    | 24276 | \$120 |
| Metal stem tip: 1/4" compression exit | 24277 | \$120 |





# Advances in GC Septa

by Douglas Elliott

## One Chromatographer's Observations—

John Lidgett, QA Specialist, is responsible for using five GCs and one GC-MS (from four different manufacturers) to assure the quality and purity of Restek's reference chemicals. In a typical month he analyzes over 1,000 samples—there can be no margin of error, no inaccurate data or equipment downtime!

When I approached John last Fall and asked him to beta-test the Merlin Microseal™ septa and the new IceBlue™ septa, he was eager to participate. John installed and used a Merlin Microseal™ septum and several IceBlue™ septa in his equipment. His objective was to observe the quality of the chromatography and report on the performance of these septa during routine use.

## Merlin Microseal™ Septa The Long-Life Alternative



After 10 months and thousands of injections on an HP 7673 Autosampler, the Merlin Microseal™ septum still looks and performs like new.

Routine maintenance involved weekly checks with a Restek

Leak Detective™ electronic leak detector (cat. # 21607). An increased carrier gas leak from the septum nut signaled that the septum needed to be examined and cleaned. To do this, John removed, disassembled, and cleaned the septum and nut. Cleaning involved sonicating the two pieces in methanol, air drying, then reassembling them. (The new Merlin 400 series septa have a removable tab that exposes an internal area designed to trap potentially interfering sample media and retain them so that the sealing surface remains clean for a long time.)

John did add one step that Merlin does not recommend; he removed the return support spring in the injection port to allow more solvent to enter the needle path. This is a delicate and potentially risky maneuver but still possible.

John said that he found it hard to "wear-out" the Microseal™ septum if it was maintained and installed properly (remember that it is as important not to over-tighten the septum nut when using a Microseal™ septum as when using another septum).

When it came to chromatographic performance, John noticed no septum bleed and no septum particles shedding into his inlet liner. This second observation led to the realization that he could use a laminar cup inlet liner. Laminar cup liners are recognized for providing excellent vaporization and homogenization of sample vapors. Their disadvantage is that they accumulate septum particles, which is a source of sample adsorption and bleed, and are difficult to clean. With the Microseal™ septum, John was able to use a laminar

cup splitter for better chromatography and not worry about septum particle build-up.

## IceBlue™ Septa They're COOL!



John also installed the new Restek IceBlue™ septa in several injection ports. I figured the Microseal™ septa endorsement would be hard to top, but he found himself even more impressed by the IceBlue™ septa!

The IceBlue™ septa showed no signs of bleed, even when he occasionally set his injection port to 300°C. The manual injections were very smooth and the septum displayed low needle resistance. Resealing after injections was very good up to the time of replacement at 300 injections. John said that the performance was as good as he had seen in any other septa.

The real surprises started when he changed the used IceBlue™ septum for a new one. The septum did not stick to the injection port as he had seen with other septa. Also, there was no oily film under the septum. He had seen this in other septa that had been evaluated. When he removed the inlet liner for cleaning, there were NO septum particles! This meant that he could also use more complex inlet liners, like a laminar cup, for better sample vaporization and mixing. John was surprised that a silicone septum offered the chromatographic performance of a Microseal™ septum.

Restek has long held a good reputation for high-performance, high-temperature Thermolite® and RedLite™ septa. Now Restek is offering you Merlin Microseal™ septa, the finest mechanically engineered septa, and the new IceBlue™ septa, the finest general purpose silicone septa.

For a detailed discussion on septum performance including bleed, bleed impostors, operating variables, and troubleshooting, please request *The Guide to Minimizing Septa Problems* (lit. cat# 59886).

## IceBlue™\* Septa

| Septa size | 50-Pack<br>cat.#/price | 100-Pack<br>cat.#/price |
|------------|------------------------|-------------------------|
| 9.5mm      | 22388 \$35             | 22389 \$65              |
| 10mm       | 22390 \$35             | 22391 \$65              |
| 11mm       | 22392 \$35             | 22393 \$65              |

## Merlin Microseal® High-Pressure, 400 Series Septa\*

| Description                                   | Merlin # | cat.# | price |
|---|----------|-------|-------|
| Nut Kit (1 nut, fits 300 & 400 series septa): | 403      | 22809 | \$100 |
| Standard Kit (1 nut, 2 high-pressure septa):  | 404      | 22810 | \$340 |
| Starter Kit (1 nut, 1 high-pressure septum):  | 405      | 22811 | \$240 |
| High-Pressure Replacement Septa (1 septum):   | 410      | 22812 | \$140 |

\*Only the most popular sizes are listed. For a complete product listing, call to request the Genuine Restek Replacement Parts for HP GCs catalog (lit. cat.# 59627).

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# New Resprep™ SPE Tubes, Disks, and Accessories

## US EPA Drinking Water Methods

We have expanded our solid phase extraction

(SPE) product line to

include many of your

favorite packings,

tubes and disk sizes,

and specialized

products for EPA

Methods!\* Call to ask

for free SPE tube or

disk samples, or to re-

ceive our "Look In-

side" brochure listing

these new SPE prod-

ucts (lit. cat.# 59623).

\* See the applicable method for specific procedural and product requirements.

| Method | Analytes                         | Product description | cat.# | price          |
|--------|----------------------------------|---------------------|-------|----------------|
| 525.1  | Semi-volatile organic compounds  | C18, 6mL, 0.5g      | 24052 | \$60, 30-pk.   |
|        |                                  | C18, 6mL, 1.0g      | 24051 | \$85, 30-pk.   |
|        |                                  | C18, 47mm disk      | 24004 | \$95, 20-pk.   |
| 508    | Organochlorine pesticides        | C18, 6mL, 0.5g tube | 24052 | \$60, 30-pk.   |
|        |                                  | C18, 6mL, 1.0g tube | 24051 | \$85, 30-pk.   |
| 515    | Chlorinated herbicides           | C18, 6mL, 0.5g tube | 24052 | \$60, 30-pk.   |
| 515.2  |                                  | Resin, 47mm disk    | 26023 | \$130, 20-pk.  |
| 548.1  | Endothal                         | BioRex® 5, 6mL tube | 26063 | \$110, 30-pk.  |
| 548.2  |                                  |                     |       |                |
| 549.1  | Diquat & paraquat                | C8, 6mL, 0.5g tube  | 26039 | \$60, 30-pk.   |
|        |                                  | C8, 47mm disk       | 24048 | \$95, 24-pk.   |
| 550.1  | PAHs                             | C18, 6mL, 1.0g tube | 24051 | \$85, 30-pk.   |
|        |                                  | C18, 47mm disk      | 24004 | \$95, 20-pk.   |
| 552.1  | Haloacetic acids & dalapon       | AG 1x8, 1mL tube    | 26064 | \$145, 100-pk. |
| 553    | Benzidines & nitrogen pesticides | C18, 6mL, 0.5g tube | 24052 | \$60, 30-pk.   |
|        |                                  | C18, 6mL, 1.0g tube | 24051 | \$85, 30-pk.   |
| 554    | Carbonyl compounds               | C18, 6mL, 0.5g tube | 24052 | \$60, 30-pk.   |

## US EPA Solid Waste Methods

| Method | Analytes  | Product description        | cat.# | price        |
|--------|---|----------------------------|-------|--------------|
| 3535   | Organochlorine pesticides   | C-18, 47mm disk            | 24004 | \$95, 20-pk. |
| 3610B  | Nitrosamines  | Alumina B, 6mL, 1.0g tubes | 55101 | \$80, 30-pk. |
|        | Phthalate esters  | Alumina N, 6mL, 1.0g tubes | 55099 | \$80, 30-pk. |
| 3620B  | Chlorinated hydrocarbons, Organochlorine pesticides, PCBs, Phthalate esters | Florisil, 6mL, 1.0g tubes  | 24034 | \$70, 30-pk. |
| 3630C  | PCBs, Organochlorine pesticides, PFBB derivatized phenols                   | Silica, 6mL, 1.0g tubes    | 24038 | \$70, 30-pk. |

## Special Methods

| Method                | Analytes                        | Product description | cat.# | price        |
|-----------------------|---------------------------------|---------------------|-------|--------------|
| CLP                   | Chlorinated pesticides          | Florisil, 6mL, 0.5g | 55100 | \$80, 30-pk. |
| Pesticides & Arochlor | Arochlor                        | Florisil, 6mL, 1.0g | 24034 | \$70, 30-pk. |
| 1664 update           | Oil & grease                    | Special, 47mm disk  | 26022 | \$75, 20-pk. |
|                       |                                 | Reprep™ Flowfilter  | 26024 | \$14, 20-pk. |
| Mass. EPA for EPH/VPH | Hexane extractable hydrocarbons | Silica, 20mL, 5.0g  | 26065 | \$95, 20-pk. |

To receive a complete SPE products information

packet, request

lit. cat.# 59664.

## New Resprep™ SPE Manifolds

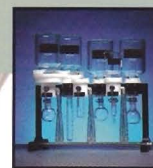
Restek now offers many versatile SPE manifold models to better suit your processing needs. Our 6D manifold is compact, inexpensive, and enables you to process 6 disk samples simultaneously. Samples are automatically drawn directly from collection

containers or can be transferred into our optional 125mL Diskcover™-47 reservoirs.

For methods that require large collection volumes, our new Maxi-Manifold accepts a wide variety of large volume receiving vessels and can easily be

expanded from one to all six positions as your budget allows or work-load requires.

We also offer both 12- and 24-position manifolds to streamline your SPE tube extraction processes. Ask for help to decide the right model for your needs! Or call for our hardware brochure (lit. cat.# 59621).



Resprep™  
Maxi-Manifold



Resprep™  
12- & 24-Port  
Manifolds

Visit us at EAS to talk about your sample preparation needs!



# Peak Performers

## Genuine Restek Replacement Parts for HP 5890/6890 GCs

- Meets or exceeds original equipment manufacturer specifications.
- Free technical service.
- Fast delivery.

Request our new catalog,  
Genuine Restek Replacement Parts  
for HP GCs (lit. cat.# 59627).



**Replacement FID Jets  
for HP 5890 GCs**

*Standard or Silcosteel®-treated  
high-performance version*

(0.011-inch ID tip)  
(Similar to HP part #19244-80560)

**Standard Version:**

cat.# 20670, \$45 ea.  
cat.# 20671, \$122/3-pk.

**High Performance:**

cat.# 20672, \$54 ea.  
cat.# 20673, \$155/3-pk.



**Replacement FID Jets  
for HP 6890 GCs**

*Standard or Silcosteel®-treated  
high-performance version*

(Similar to HP part #G1531-80560)

**Standard Version:**

cat.# 21621, \$36 ea.  
cat.# 21682, \$100/3-pk.

**High Performance:**

cat.# 21620, \$45 ea.  
cat.# 21683, \$125/3-pk.



**MSD Source Nut**

cat.# 20643, \$18/2-pk.



**MSD Conversion Fitting**

cat.# 21314, \$40 ea.



**FID/NPD Capillary Adaptor  
for HP 5890/6890 GCs**

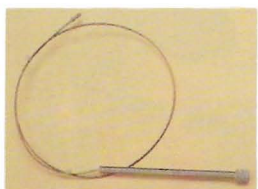
(Similar to HP part #19244-80610)

cat.# 22077, \$52/ea.



**Detector Plug Nut**

cat.# 21883, \$24/2-pk.



**Replacement Chemical Trap  
for HP 5890 GCs**

(Similar to HP part # 05890-61260)

cat. # 21610, \$82 ea.



**Silcosteel®-Treated Septum-  
Purged Packed Column  
Port Weldment**

(Similar to HP part # 19243-80570)

cat.# 21691, \$99/ea.



**Low Volume Injector for HP  
Split/Splitless GC Inlets**

cat.# 21692, \$375/kit



**Stainless Steel Capillary Nuts  
for HP 5890/6890 GCs**

For use with standard ferrules:

cat.# 20883, \$25/2-pk.

For use with "short"  
HP-type ferrules:

(Similar to HP part #5181-8830)

cat.# 21884, \$25/2-pk.

**New! Replacement Split Trap  
Assembly for HP 6890 GCs**

(Similar to HP part # G1544-80520)

cat. # 22820, \$70 ea.-not shown

**Replacement 24-inch Split  
Vent Line for HP**

**5890 GCs**

(Similar to HP part # 5020-8262)

cat. #22800, \$36/2-pk.-not shown



**Restek's Leak Detective™**

(110 volts) cat.# 21607, \$595/ea.

(220 volts) cat.# 21609, \$645/ea.



**Air Diverter for HP 5890 GCs**

(Similar to HP part # 19247-60510)

cat.# 22076, \$120/ea.



**Reducing Nut for HP  
5890/6890 GCs**

(Similar to HP part # 18740-20800)

cat.# 22078, \$35/ea.

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# A Heart of Gold Lives On...

In September, 1997

Erik D. Steindl, certified personal fitness trainer at Restek, a senior at The Pennsylvania State University (PSU), a Staff Sergeant in the Pennsylvania Air National Guard, and son of Eric Steindl (also an employee of Restek), was diagnosed with a germ-cell cancer. The initial prognosis was excellent.

At the time of diagnosis, the softball-sized tumor had already invaded Erik's left bronchial tube, restricting air and blood flow to his left lung. It also was believed to be dangerously close to his heart. Surgery at this point was not an option. The doctors moved quickly to begin a very aggressive chemotherapy treatment, which they hoped would shrink the size of the tumor and restore blood and air flow to Erik's left lung. Then they would remove the remaining cancerous tissue by surgery. While Erik's family and friends struggled to accept that a strong, otherwise healthy 24-year-old could have developed such a serious medical condition, Erik continued with his studies at PSU and planned for his future.

Initial chemotherapy treatments appeared to have a positive effect, and although chemotherapy would exhaust Erik, he never lost sight of his dreams or forgot his family and friends. He continued to write, phone, and visit people whenever possible. Too sick and tired to

drive himself, his family would take him to visit his friends. Erik would always say "I need to walk in and show people I'm OK so they don't worry so much." At Restek, Erik would walk throughout the entire facility, talking to people about his hopes and plans for the future. To many, he would say "I'm going to beat this cancer and write a book about it."

In October and November, Erik's condition became more complicated. His heart rate would suddenly and dangerously increase, requiring him to be flown by Life Flight helicopter to Hershey Medical Center, PA. He developed severe headaches, and it was discovered that blood clots had formed in several major veins in his brain. While in the hospital, family and friends from across the country came to visit. Each time a person entered Erik's hospital room, Erik would sit up, try to relieve the visitor's worry, and entertain them as if nothing was wrong. Despite his declining health, Erik maintained contact with friends at Restek and PSU.

In December, Erik's younger brother Greg graduated from PSU. Too sick to attend the graduation ceremony, Erik stayed at home with several friends, talking and planning for 1998.

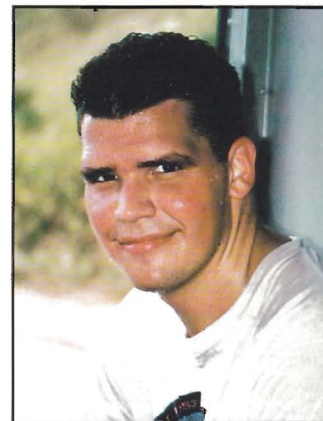
After four chemotherapy treatments and numerous tests, Erik's doctors felt the cancer was finally operable. During

surgery, the doctors found the cancer had spread into the heart tissue itself, part of the left lung, and the chest wall. The night after surgery, Erik suffered a stroke from the blood clots in his brain. Gravely ill and unable to speak because of the ventilator, Erik was still fighting and writing notes to ask if his family and friends were alright. On February 14, Valentine's Day, this young man who touched so many hearts, lost his fight against cancer.

On June 19, what would have been Erik's 25 birthday, a memorial ceremony was held at Restek. Over 100 family members and friends attended from across the country. Erik's parents and brother were presented with several awards for Erik. Restek presented its highest honor posthumously, "The President's Award," to Erik for his courage and strength, which was an example to us all. Also, Restek dedicated an outdoor memorial and renamed the fitness center "Corazon de Oro," meaning "Heart of Gold" in Spanish, a language Erik had studied for many years in the Air Force and at PSU. PSU presented Erik's posthumous diploma, awarded because of Erik's fight to continue with his studies in spite of his illness. The Pennsylvania Air National Guard, 112<sup>th</sup> Air Control Squadron, by order of the President of the United States, awarded Erik the "Air Force Meritorious Service Award." This is the highest award given by the Air Force to

a non-combatant servicemen. The members of the squadron also presented Erik's family with a shadow box containing all the awards, rank insignia, and unit emblems Erik had earned during his military service.

Restek deeply misses Erik and will actively preserve his legacy here and in the community. To ensure Erik's dedication and spirit lives on, a memorial scholarship fund has been established at Penn State University. This scholarship, the "Erik D. Steindl Spanish Scholarship Fund" will be awarded to a student in the Spanish Department who demonstrates outstanding academic excellence and community spirit. If you would like to make a contribution to this fund, please send your tax-deductible donation to: **Erik D. Steindl Spanish Scholarship Fund, The Pennsylvania State University, College of Liberal Arts, 101 Sparks Bldg., University Park, PA 16802.**



**Erik D. Steindl**  
*Deeply missed by all.*



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