

The Advantage

**Innovators of
High Resolution
Chromatography
Products**

Pharmaceutical Researchers, The Future of ChromatographySM Arrives Today!

by David Bliesner

in this issue

Restek Analytical ServicesSM:

- **cGMP, HPLC, and GC method development and validation**
- **Stationary phase development**
- **Full lines of HPLC and GC columns and accessories**
- **Innovative hardware and accessory designs**
- **Unsurpassed HPLC and GC technical support**
- **HPLC and GC educational services**

Restek Corporation is proud to announce the grand opening of Restek Analytical ServicesSM (RAS). RAS is a unique and innovative, current Good Manufacturing Practices (cGMP)-compliant chromatography laboratory, designed to deliver quality products and services for the pharmaceutical market. No other column manufacturer can provide such complete services to support their products. Along with a full line of both HPLC and GC columns, RAS offers analytical method development and validation, HPLC/GC education and training, custom stationary phase design, and cGMP/regulatory services and support.

RAS has assembled seasoned researchers and analysts who

have extensive experience in cGMP method development and validation. These experts are a perfect complement to our existing manufacturing staff. As a group, we understand the pressures and time constraints associated with pharmaceutical research and have practical experience analyzing many of the common drugs currently under development. The entire RAS staff is dedicated to helping you achieve your goals in a timely and compliant fashion.

In addition to offering a complete selection of quality HPLC and GC columns, we can help design tools to fit your unique requirements. We continue to develop new stationary phases and applications in response to customer

inquiries and needs. We can design a product and service package specially for you.

For further information, please request the RAS fulfillment folder (cat.# 59630). This folder includes details on quality assurance, research and development, analytical development and validation, laboratory and regulatory service, and HPLC columns and accessories.

Or, feel free to contact the director of the RAS laboratory, Dr. David Bliesner, to discuss your specific applications. He can be reached at 800-356-1688 or 814-353-1300, ext. 2193. Call us today and find out why RAS is truly the **future of chromatography**.

Restek Analytical Services
...pg. 1

**Chiral Separation of
Underivitized
Pharmaceuticals**
...pg. 3

Azo Dye Analysis
...pg. 4

Tribute to Dr. Kováts
pgs. 5 & 11

New Rt-U PLOT Columns
...pg. 6

OrganoTin Mixes
...pg. 7

**Why Should You use Rtx®-
CLPesticides Columns?**
...pg. 8

**Silcosteel®-Treated
Injection Ports**
...pg. 9

Ultra-Clean XAD®-2 Resin
...pg. 9

**Rtx®-624 Column for
Analysis of Rum & Scotch**
...pg. 10

Koni's Korner
...pg. 12

Peak Performers
...pg. 14

News From Restek
...pg. 16



Spring/Summer
98



**Macclesfield
Cheshire SK11 6PJ
UK**

Restek Analytical ServicesSM

continued from page 1.

RAS has assembled some of the most talented, customer-oriented analysts in the HPLC and pharmaceutical industries. Meet the leaders of RAS:



Dr. David M. Bliesner,
RAS Director

Expertise:

- HPLC method development and validation in a cGMP environment
 - Qualification and operation of cGMP analytical laboratories
 - Thin layer chromatography
 - cGMP auditing and data review
 - 15 years combined R&D and leadership experience
- dbliesner@restekcorp.com**



David S. Bell,
RAS Senior Researcher

Expertise:

- HPLC method development and validation in a cGMP environment
 - Thin layer chromatography
 - 8 years of experience
- dbell@restekcorp.com**



Keith J. Duff,
RAS R&D Group Leader

Expertise:

- HPLC bonded phase synthesis
 - Synthetic chemistry
 - HPLC method development
 - 16 years of experience
- keithd@restekcorp.com**



Larry T. Peters,
RAS Senior HPLC Technician

Expertise:

- HPLC column packing
 - HPLC column bonding
 - 16 years of experience
- larryp@restekcorp.com**



Randy L. Romesberg,
RAS Production Group Leader

Expertise:

- R&D and HPLC packing materials and hardware production
 - 9 years of experience
- rrome@restekcorp.com**

RAS Products & Services:

Development & Validation

- HPLC and GC method development and validation
- Ruggedness testing
- Robustness testing
- HPLC column crossover validation
- Method troubleshooting and optimizing

Laboratory & Regulatory Services

- HPLC & GC Educational Services
- Assistance in preparation with CMC sections for NDAs
- Laboratory auditing services
- On-site data review and troubleshooting
- Pre-approval inspection (PAI) preparation assistance

Research & Development

- Developing custom stationary phases
- Designing new hardware and accessories
- Investigating new separation technologies
- Improving existing products

Quality Assurance

- Full compliance with cGMPs
- QA testing is an integrated part of the RAS system

HPLC Columns & Accessories

- Pinnacle®, Hypersil®, Inertsil®, Kromasil®, and Nucleosil® HPLC columns
- Unsurpassed technical support
- Quality HPLC accessories
- 30-day "no questions asked" return policy
- Stationary phase and hardware design

"In the modern chromatography laboratory, you want one thing — reliable solutions to your chromatographic problems. Previously, when a method could not be reproduced, questions such as, 'Is it the column or did something change in the sample workup?' could not be answered by one source. By combining product development and method development under one umbrella, you can be assured that your problem will be solved with full accountability. Any method validation done by Restek is supported by a team of dedicated scientists with your success in mind. RAS is a fully cGMP compliant laboratory that provides personalized services dependent on your needs. We can provide training, method development and validation, or custom design stationary phases to speed up your analysis or achieve a difficult separation. Please give us a call and let us help you."

David M. Bliesner, Ph.D.
Director, RASSM



**Restek
Analytical
Services-**

The Advantage

Restek's Silcosteel® HPLC Columns

the strength of steel and inertness of glass

by Matt Piserchio

Combine the ruggedness and pressure limits of stainless steel with the inertness of PEEK or GLT™.

- Provide a shield between the metal surface and active species.
- Applicable to all column dimensions.

When analyzing active components, GC and HPLC sample pathways must be inert to prevent adsorption and poor chromatography. Unfortunately, GC sample pathways are constructed from metal to achieve and maintain the high temperatures needed for sample vaporization. In HPLC, columns and sample pathways require materials that can withstand high pressures. Contact of an active analyte

with metal surfaces can result in adsorption, sample degradation, and inaccurate quantitation.

Silcosteel® is a proprietary process developed by Restek that applies a thin layer of material to the surface of metals. This coating acts as a passivation layer by providing a shield between the metal surface and the active species, eliminating detrimental interactions.

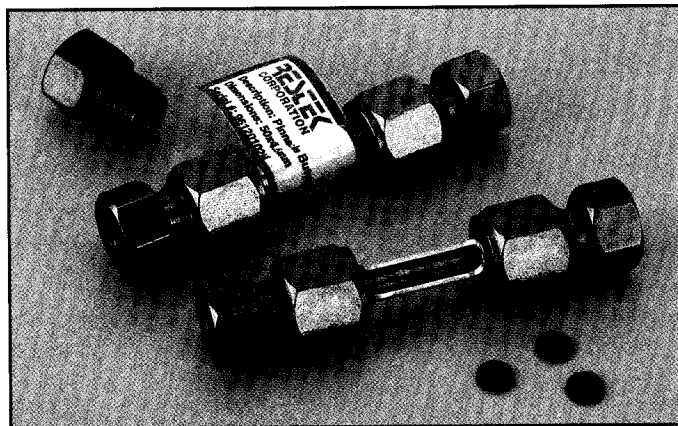
Silcosteel® GC columns and accessories have been proven effective by a significant number of satisfied customers. We have coated FID jets, FPD jets, entire injection ports, inlet seals, metal injection port liners, head space needles, nickel reaction tubes, and air sampling canisters. MXT® columns, rugged and inert metal GC capillary columns treated with Silcosteel®, are the column of choice in the process analyzer and portable GC markets. Now Silcosteel® technology is available for a full line of normal phase, reverse phase, and ion exchange HPLC columns.

Metal-free pathways are desired to reduce irreversible adsorption during HPLC analyses of proteins, peptides, and any other compounds that can undergo metal complexation.^{1,2} Currently, columns constructed of PEEK (polyetheretherketone) or GLT™ (glass-lined stainless steel tubing) are utilized for such analyses. Although both materials are effective, each of these materials has definite limitations.

Innovators of
High Resolution
Chromatography
Products

in this issue

Restek's Silcosteel® HPLC Columns	...pg. 1
n-Octane on Res-Sil™ C	...pg. 4
Silcosteel®	...pg. 6
Integra-Guard™ Columns	...pg. 8
Rt-βDEXcst Capillary Columns	...pg. 10
Koni's Korner	...pg. 11
Peak Performers	...pg. 14
SilcoSleeve™ Inlet Liners	...pg. 15



Spring
07
INTERNATIONAL

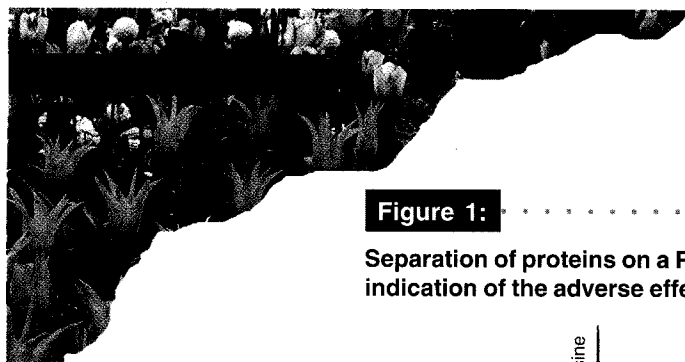
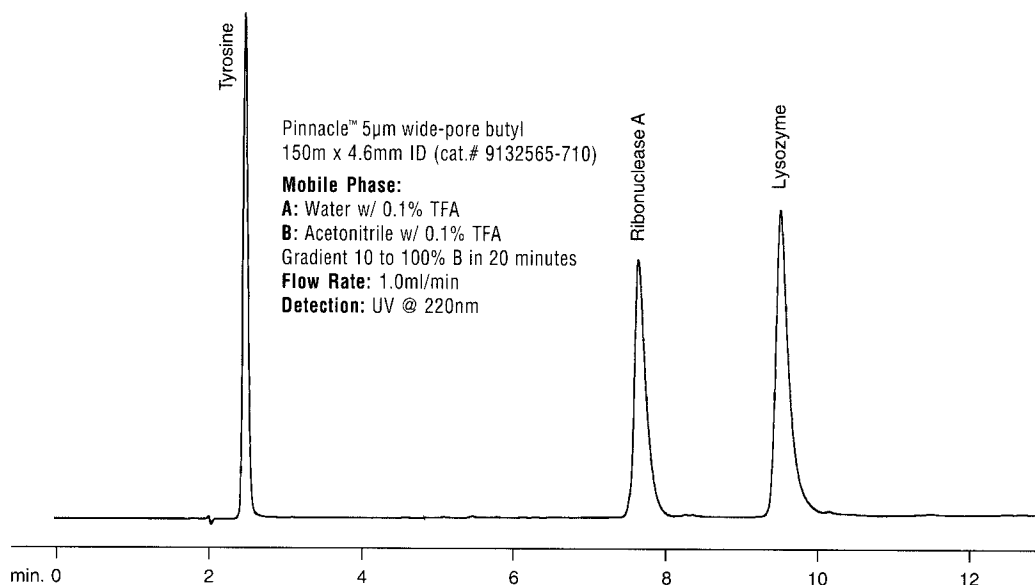


Figure 1:

Separation of proteins on a Pinnacle™ wide-pore butyl in Silcosteel® hardware shows no indication of the adverse effects of metal on protein adsorption.



PEEK has a limited pressure threshold and can contain chemical impurities. GLT™ can crack, especially at the column inlet or outlet, causing a disturbance in the flow pattern resulting in peak distortion and adsorption. Finally, both PEEK and GLT™ are available in limited configurations. Silcosteel® combines the ruggedness, thermal stability, and pressure limits of stainless steel with the inertness of GLT™ or PEEK. Silcosteel® can also be applied to any size HPLC column and virtually any other metal surface in the HPLC sample pathway.

The separation of proteins in **Figure 1** illustrates the inertness of the Silcosteel® layer. Ribonuclease A and Lysozyme elute with excellent symmetry using a standard gradient profile. Sharp, symmetrical peaks indicate the absence of the catalytic effect of metal surfaces that may cause protein adsorption. This will result in improved resolution and recovery when analyzing active species.

If your analyses demand a metal-free pathway, Restek has the Silcosteel®-treated HPLC column that is right for you. Our complete line of Pinnacle™ HPLC columns is now available in Silcosteel® hardware. *Simply add a "-710" to your part number to add the benefits of Silcosteel® to your HPLC analysis.*

For part numbers and prices, please call
your local distributor
or call to request a copy of our new 1997
Chromatography Products Guide

Add "-710" to your
part number and
get the benefits of
Silcosteel®
with your HPLC
analysis!

A complete line of HPLC columns is available from Restek in either Silcosteel® or Stainless Steel hardware:

Reverse Phase

Pinnacle ODS
Pinnacle Octyl
Pinnacle Phenyl
Pinnacle Butyl
Pinnacle Methyl
Pinnacle Ultra C18
Kromasil C18
Kromasil C8
Kromasil C4
Nucleosil C18
Nucleosil C8

Base Deactivated

Pinnacle ODS Amine
Pinnacle Octyl Amine
Pinnacle Phenyl Amine
Pinnacle Cyano Amine

Normal Phase

Pinnacle Silica
Pinnacle Amino
Pinnacle Cyano
Nucleosil Cyano

Ion Exchange

Pinnacle SAX
Nucleosil SCX

Specialty

Pinnacle TO-11
Pinnacle PAH
Pinnacle EcoSep
Pinnacle Wide-Pore Butyl

The Restek

Innovators of
High Resolution
Chromatography
Products

Fast GC Using Microbore Capillary Columns

by Kristi Sellers

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

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

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

Speed and Resolution

Table I compares the characteristics of microbore columns to conventional columns. This data holds the key to whether microbore columns are right for your analysis. The most striking difference of microbore columns is their high efficiency (plates/meter) compared to other diameters.

Table I indicates that a 0.10mm ID column is 160% more efficient than a 0.25mm ID column. This high effi-

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

Flow Rates

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

in this issue

**Fast GC Using Microbore
Capillary Columns**
...pg. 1

**Rtx®-CL Pesticides
Column**
...pg. 4

**Optimizing Capillary
Chiral Analyses**
...pg. 6

**Benefits of Restek HPLC
Columns**
...pg. 8

Koni's Korner
...pg. 10

Heated Purifiers
...pg. 12

**FIA Fuel Testing with the
Rtx®-5 Column**
...pg. 13

Peak Performers
...pg. 14

Pro ezGC™ for Windows®
...pg. 15

Table I:

Column Characteristics

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

Summer
INTERNATIONAL

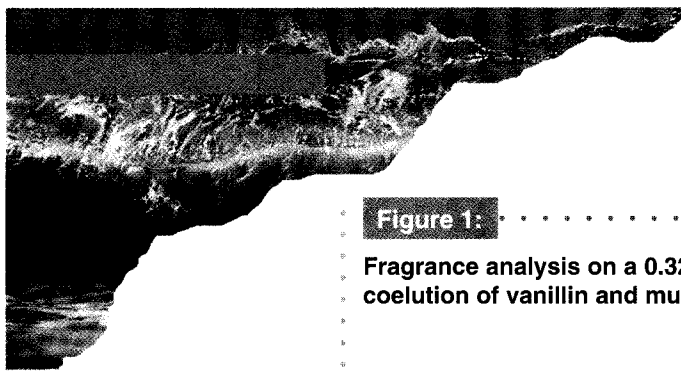
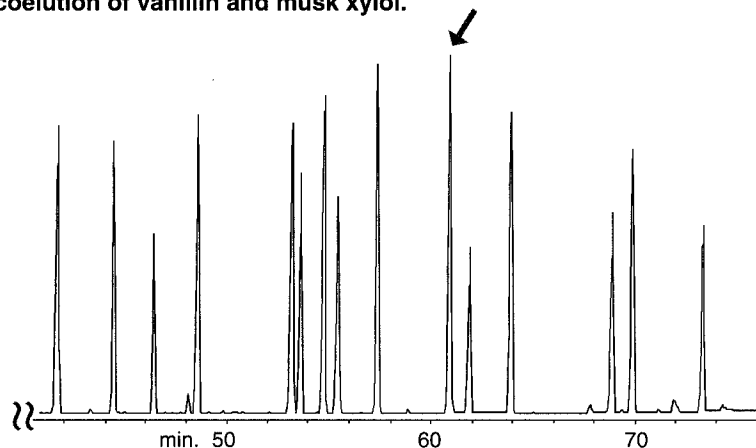


Figure 1:

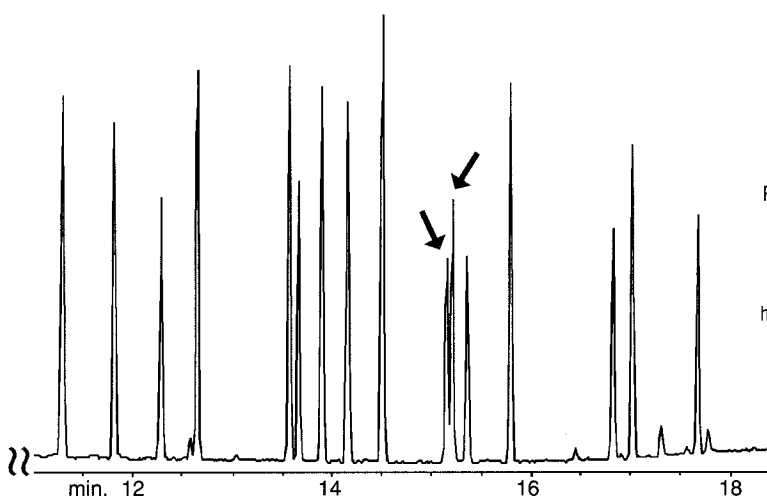
Fragrance analysis on a 0.32mm ID Rtx®-Wax column takes 75 minutes with complete coelution of vanillin and musk xylol.



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

Figure 2:

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



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

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

Operating Pressures

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

Sample Capacity

A limiting factor of a microbore column is the amount of sample that can be injected onto the column. Table I indicates that the sample capacity of a microbore column is ten times less than a 0.25mm ID column. Therefore, the on-column injection should be at least ten times lower for a microbore column.

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

contamination will occur more rapidly when dirty samples are injected. This means that 0.25 or 0.32 mm ID columns will be more rugged and require less maintenance for dirty samples than microbore columns.

Whenever possible, samples containing non-volatile residue should be avoided. If dirty samples are a must, extensive column and injection port maintenance is required. Otherwise, loss of resolution,

ghost peaks, and a high background signal will result.

Injector Considerations

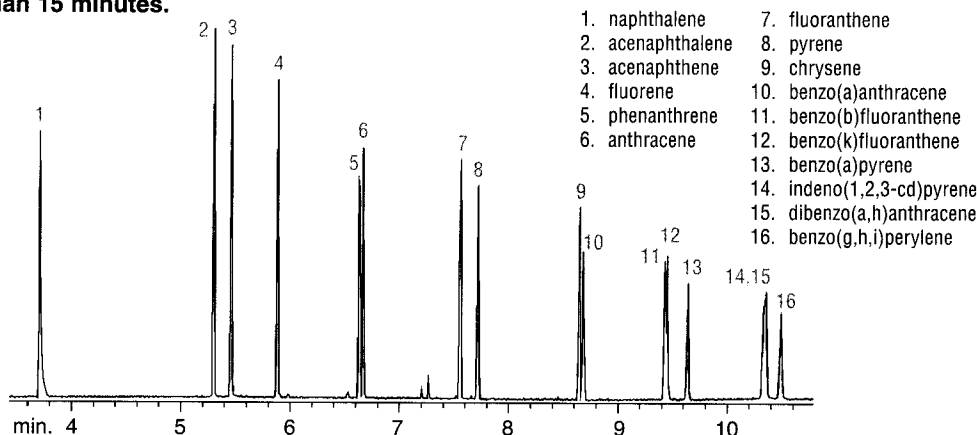
Direct and on-column injection modes are not recommended due to the required low flow rates and small bore size of these columns. Therefore, trace analyses are difficult to perform with microbore columns. Split and splitless injections are the best alternatives. However, since

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



Figure 3:

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



10m, 0.10mm ID, 0.10µm Rtx-5 (cat.# 41201). 0.5µL splitless injection. 41psi initial pressure, hold 2 min. 8 psi/min. to 99psi (hold 1.87 min.). 275°C, vent open @ 1 min. **Oven temp.:** 40°C (hold 0.5 min.) to 90°C @ 70°C/min. then to 100°C @ 5°C/min. then to 310°C @ 30°C/min. (hold 2 min.).

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

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

eluting compounds.

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

Detector Considerations

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

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

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

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

Product Listing:

Microbore Capillary Columns

0.10mm ID, 0.10µm

Column	temp. limits	10-meter	20-meter
Rtx®-1	-60 to 330/350°C	41101	41102
Rtx®-5	-60 to 330/350°C	41201	41202
Rtx®-Wax	20 to 250°C	41601	41602

0.10mm ID, 0.20µm

Column	temp. limits	10-meter	20-meter
Rtx®-Wax	20 to 240/250°C	41603	41604

0.10mm ID, 0.40µm

Column	temp. limits	10-meter	20-meter
Rtx®-1	-60 to 320/340°C	41103	41104
Rtx®-5	-60 to 320/340°C	41203	41204

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

The Advantage

Restek's Silcosteel® HPLC Columns

the strength of steel and inertness of glass

by Matt Piserchio

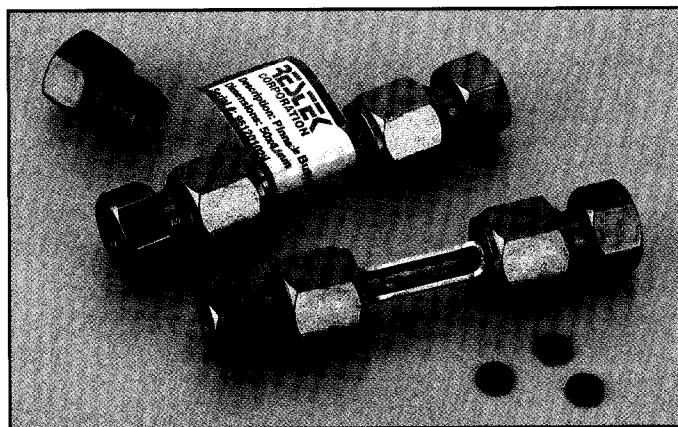
Combine the ruggedness and pressure limits of stainless steel with the inertness of PEEK or GLT™.

- Provide a shield between the metal surface and active species.
- Applicable to all column dimensions.

When analyzing active components, GC and HPLC sample pathways must be inert to prevent adsorption and poor chromatography. Unfortunately, GC sample pathways are constructed from metal to achieve and maintain the high temperatures needed for sample vaporization. In HPLC, columns and sample pathways require materials that can withstand high pressures. Contact of an active analyte

with metal surfaces can result in adsorption, sample degradation, and inaccurate quantitation.

Silcosteel® is a proprietary process developed by Restek that applies a thin layer of material to the surface of metals. This coating acts as a passivation layer by providing a shield between the metal surface and the active species, eliminating detrimental interactions.



Silcosteel® GC columns and accessories have been proven effective by a significant number of satisfied customers. We have coated FID jets, FPD jets, entire injection ports, inlet seals, metal injection port liners, head space needles, nickel reaction tubes, and air sampling canisters. MXT® columns, rugged and inert metal GC capillary columns treated with Silcosteel®, are the column of choice in the process analyzer and portable GC markets. Now Silcosteel® technology is available for a full line of normal phase, reverse phase, and ion exchange HPLC columns.

Metal-free pathways are desired to reduce irreversible adsorption during HPLC analyses of proteins, peptides, and any other compounds that can undergo metal complexation.^{1,2} Currently, columns constructed of PEEK (polyetheretherketone) or GLT™ (glass-lined stainless steel tubing) are utilized for such analyses. Although both materials are effective, each of these materials has definite limitations.

Innovators of
High Resolution
Chromatography
Products

in this issue

Restek's Silcosteel® HPLC Columns	...pg. 1
n-Octane on Res-Sil™ C	...pg. 4
Silcosteel®	...pg. 6
Integra-Guard™ Columns	...pg. 8
Rt-βDEXcst Capillary Columns	...pg. 10
Koni's Korner	...pg. 11
Peak Performers	...pg. 14
SilcoSleeve™ Inlet Liners	...pg. 15

Spring
INTERNATIONAL

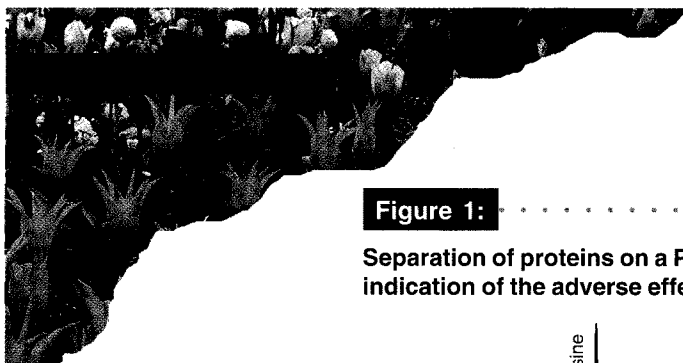
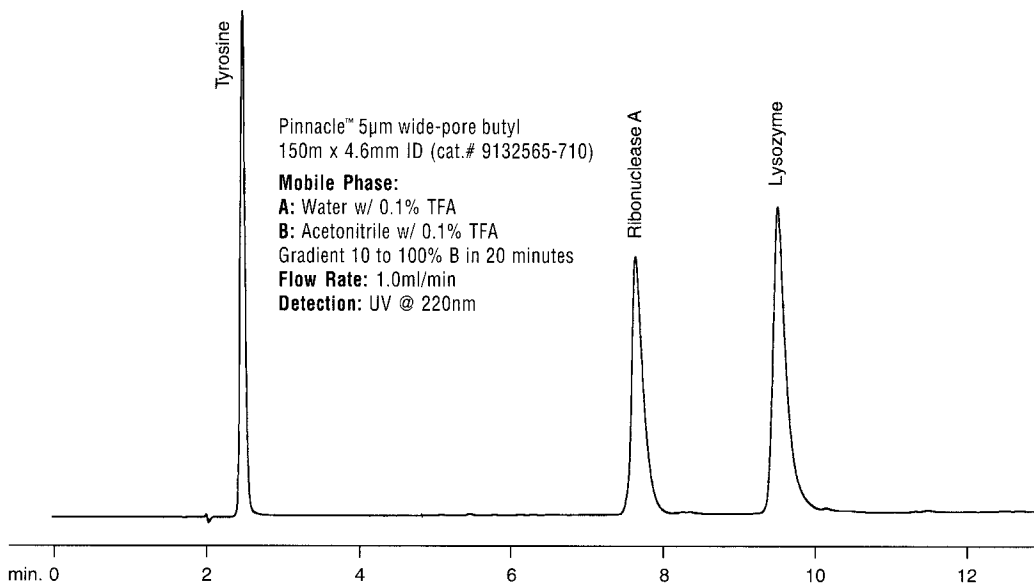


Figure 1:

Separation of proteins on a Pinnacle™ wide-pore butyl in Silcosteel® hardware shows no indication of the adverse effects of metal on protein adsorption.



PEEK has a limited pressure threshold and can contain chemical impurities. GLT™ can crack, especially at the column inlet or outlet, causing a disturbance in the flow pattern resulting in peak distortion and adsorption. Finally, both PEEK and GLT™ are available in limited configurations. Silcosteel® combines the ruggedness, thermal stability, and pressure limits of stainless steel with the inertness of GLT™ or PEEK. Silcosteel® can also be applied to any size HPLC column and virtually any other metal surface in the HPLC sample pathway.

The separation of proteins in **Figure 1** illustrates the inertness of the Silcosteel® layer. Ribonuclease A and Lysozyme elute with excellent symmetry using a standard gradient profile. Sharp, symmetrical peaks indicate the absence of the catalytic effect of metal surfaces that may cause protein adsorption. This will result in improved resolution and recovery when analyzing active species.

If your analyses demand a metal-free pathway, Restek has the Silcosteel®-treated HPLC column that is right for you. Our complete line of Pinnacle™ HPLC columns is now available in Silcosteel® hardware. *Simply add a "-710" to your part number to add the benefits of Silcosteel® to your HPLC analysis.*

For part numbers and prices, please call
your local distributor
or call to request a copy of our new 1997
Chromatography Products Guide

Add "-710" to your
part number and
get the benefits of
Silcosteel®
with your HPLC
analysis!

A complete line of HPLC columns is available from Restek in either Silcosteel® or Stainless Steel hardware:

Reverse Phase

Pinnacle ODS
Pinnacle Octyl
Pinnacle Phenyl
Pinnacle Butyl
Pinnacle Methyl
Pinnacle Ultra C18
Kromasil C18
Kromasil C8
Kromasil C4
Nucleosil C18
Nucleosil C8

Base Deactivated

Pinnacle ODS Amine
Pinnacle Octyl Amine
Pinnacle Phenyl Amine
Pinnacle Cyano Amine

Normal Phase

Pinnacle Silica
Pinnacle Amino
Pinnacle Cyano
Nucleosil Cyano

Ion Exchange

Pinnacle SAX
Nucleosil SCX

Specialty

Pinnacle TO-11
Pinnacle PAH
Pinnacle EcoSep
Pinnacle Wide-Pore Butyl

Act now to receive
the added benefits
of Silcosteel® for
your HPLC column
at no extra cost!*

* This special offer expires
May 30, 1997.

References

1. P.C. Sadek, P.W. Carr, L.D. Bowers and L.C. Haddad, *Anal. Biochem.*, **144**(1985)128.
2. C.N. Trumbore, R.D. Tremblay, J.T. Penrose, M. Mercer and F. Kelleher, *J. Chromatogr.*, **280**(1983)43.



Matt Piserchio, HPLC Chemist
and Product Line Manager



Randy Romesberg
HPLC Chemist

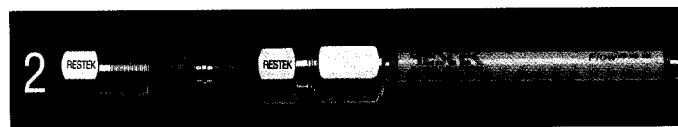
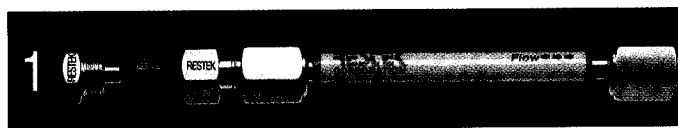
Please call
your local distributor
for more information or HPLC Technical Service

HPLC Trident™ Guard Column System

by Matt Piserchio

Call for ordering information!

- Our convenient and economical leak-free system is as easy as 1-2-3.
- The versatile configuration protects against all levels of contamination.
- The Trident™ system's integral design eliminates troublesome tubing connections.



1. The system's foundation consists of the analytical column configured with our exclusive Trident™ end fitting and XF filter fitting. This configuration contains the standard internal frit as well as a replaceable external frit, which can be easily changed without disturbing the packed bed. Changing the external frit can reverse the effects of accumulated particles, such as high back pressure or peak distortion. To order this basic configuration, add a "-700" to any Restek HPLC column catalog number.

2. The system can also be configured to accept an integral guard cartridge for greater protection against sample contaminants. The integral design eliminates

the need for a separate holder and connecting tubing, which can cause additional band broadening. To obtain this configuration, order any Restek HPLC column (include the "-700" suffix), the XG male fitting, and the appropriate pack of guard cartridges.

3. For maximum protection against contaminants and particulate matter, the system can be configured with both an integral guard cartridge and a replaceable external frit. To obtain this configuration, order any Restek HPLC column (include the "-700" suffix), the XG-XF male fitting, and the appropriate pack of guard cartridges.

Restek Corporation

• 3 •

INTERNATIONAL

RESTEK

Innovators of
High Resolution
Chromatography
Products

Rtx®-5MS The True **LOW BLEED LEADER!**

Every column manufacturer claims to have the lowest bleed capillary column for use with GC/MS.

Restek decided to conduct a side-by-side test of several commercially available "MS" columns for bleed, response and performance. Our testing indicates that the Rtx®-5MS is the ideal column for GC/MS applications requiring high sensitivity.

Bleed

The Rtx®-5MS was compared to two other "MS" columns in an HP 5890 Series II GC with an HP 5971 Mass Selective Detector. Each column was tested under identical conditions with respect to both GC and MSD param-

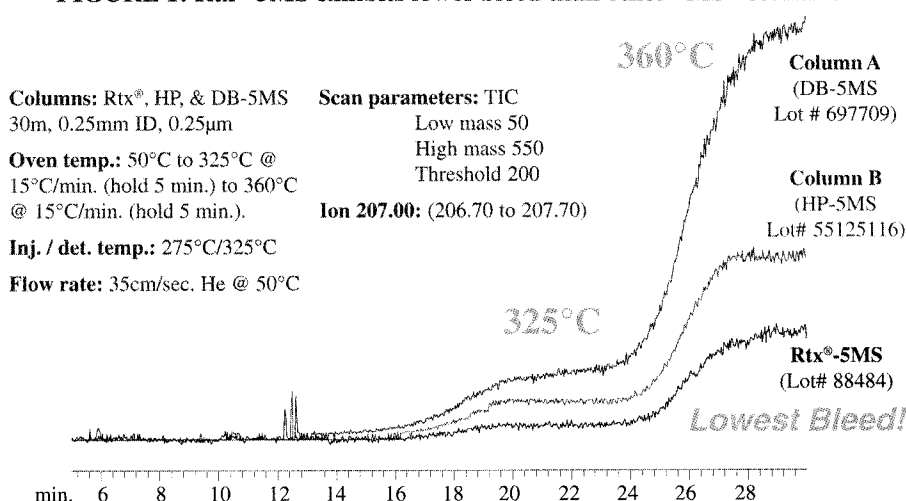
eters (e.g. linear velocity, temperatures, tuning, etc.). Figure 1 shows the plot of mass 207, the most characteristic bleed ion of a polysiloxane stationary phase. The Rtx®-5MS column exhibits lower bleed at both 325°C

and 360°C compared to the other two "MS" columns.

How important is having a column with low bleed?

Column bleed can ultimately effect sensitivity, spectral quality, and source contamination. When a column exhibits high bleed, the signal-to-noise (s/n) ratio is reduced. A low s/n ratio results in poor sensitivity and can decrease the quality of analyte spectra. A decrease in spectral quality complicates the interpretation of mass spectra that makes accurate compound identification difficult or impossible. Reduced column bleed is critical for ion trap mass

FIGURE 1: Rtx®-5MS exhibits lower bleed than other "MS" columns!



In this Issue

Rtx®-5MS Column
...pg. 1

Silcosteel® Packed Columns
...pg. 4

Stabilwax® Bonded Packed Column
...pg. 5

Rtx®-5/Rtx®-50 Columns for Pesticide Analysis
...pg. 6

How Old is YOUR Capillary Column
...pg. 8

Koni's Korner
...pg. 10

Integra-Guard™ Column
...pg. 12

SilcoCan™ Canister with Pressure/Vacuum Gauge
...pg. 13

Peak Performers
...pg. 14

Summer
96
INTERNATIONAL

Rtx®-5MS

The True **LOW BLEED LEADER!**

spectrometers. The automatic gain control feature of these instruments will significantly reduce sensitivity as column bleed increases during temperature programming. Using low bleed Rtx®-5MS columns will result in increased sensitivity of ion trap GC/MS systems. If a column continues to contribute high bleed, it may result in source contamination. A contaminated source should be cleaned, which may take up to a full day, resulting in lost manpower and valuable

Components	Rtx®-5MS	DB-5MS	HP-5MS
n-nitroso-di-n-propylamine	0.30	0.28	0.25
2,4-dinitrophenol	0.62	0.53	0.52
4-nitrophenol	0.83	0.82	0.76
4-nitroaniline	0.93	0.69	0.80
pentachlorophenol	1.38	1.30	1.34

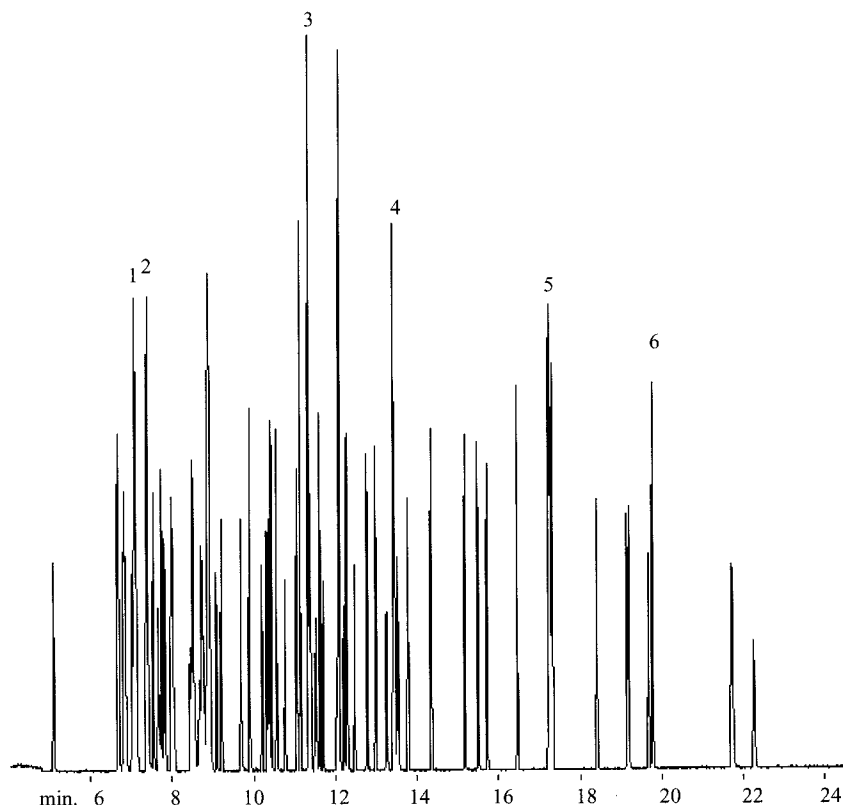
instrument time. Because each Rtx®-5MS column is thoroughly tested for low bleed, it is the column of choice for the prevention of these problems.

Inertness

Low bleed levels are important, but inertness is also a critical factor when choosing a capillary column for GC/MS analysis. How do active environmental compounds

respond on the Rtx®-5MS compared to other "MS" columns? A 14 component test mixture containing five highly active compounds was injected onto each of the three "MS" columns. The results of

FIGURE 2: The Rtx®-5MS GUARD column shows excellent inertness and low bleed for the analysis of semi-volatile pollutants without increasing analysis time.



30m, 0.25mm ID, 0.25µm Rtx®-5MS
Integra-Guard (cat.# 12623-124)
2.0µl injection of Semi-Volatile
Calibration mix. Concentration: 20ng/µl.

Oven temp.: 45°C (hold 3.5 min.) to 95°C
@ 40°C/min., to 295°C @ 17°C/min.
(hold 2 min.), to 320°C @ 40°C/min.
(hold 5.9 min.).

Inj./det. temp.: 250°C/310°C

Linear velocity: 32cm/sec. @ 40°C

Scan rate: 0.8 sec./scan

Scan range: 35-500amu

Flow rate: 1.03ml/min. after EPC
pressure pulse

Ionization: EI

Electron range: 70eV

Splitless hold time: 0.95 min.

Internal Standards:

- 1,4-dichlorobenzene-d4
- naphthalene-d8
- acenaphthene-d10
- phenanthrene-d10
- chrysene-d12
- perylene-d12

Analysis courtesy of Incheape Testing Services -
Aquatec Laboratories, Burlington, Vermont. Image
file courtesy of Thru-Put Systems, Inc.

five replicate analyses on each column is shown in Table I. The average response for each of these difficult compounds is higher on the Rtx®-5MS than on either of the competitive columns.

When performing EPA Semi-volatile analyses, the Rtx®-5MS column will exceed the QA performance criteria for inertness and offer considerably low bleed. An example chromatogram is shown in Figure 2.

Column Lifetime

The "MS" column you choose not only must have low bleed and excellent inertness, but it also needs to last. Only Restek offers Integra-Guard™ technology for your mass spec columns. Integra-Guard™ columns have built-in protection without any connectors that can leak and cause loss in sensitivity and possible damage to the mass spec. The built-in guard column prevents sample contaminants from reaching the coated portion of the column. For more information on Restek's Integra-Guard™ columns, please call your local distributor.

Get the Facts

Are low bleed, excellent inertness, and long column lifetime too much to ask for in one capillary column? No! The Rtx®-5MS offers you the most column for your money.

Rtx®-5MS (Crossbond® 5% diphenyl - 95% dimethyl polysiloxane)

ID	µm	15-Meter	30-Meter
0.25mm	0.10	cat.# 12605	cat.# 12608
	0.25	cat.# 12620	cat.# 12623
	0.50	cat.# 12635	cat.# 12638
	1.00	cat.# 12650	cat.# 12653
0.32mm	0.10	cat.# 12606	cat.# 12609
	0.25	cat.# 12621	cat.# 12624
	0.50	cat.# 12636	cat.# 12639
0.53mm	1.00	cat.# 12651	cat.# 12654
	0.50	cat.# 12637	cat.# 12640
	1.00	cat.# 12652	cat.# 12655
	1.50	cat.# 12667	cat.# 12670

Rtx®-5MS INTEGRA-GUARD™ (30-meter column with a built-in 5-meter guard column)

µm	0.25mm ID	0.32mm ID	0.53mm ID
0.25	12623-124	12624-125	—
0.50	12638-124	12639-125	12640-126
1.00	12653-124	12654-125	12655-126
1.50	—	—	12670-126

Restek has offered low bleed GC/MS columns since 1991. The Rtx®-5MS continues this tradition and gives the best overall performance for bleed, response, and resolution when compared to competitive offerings. Rtx®-5MS bleed and response factor specifications have been established to ensure that every column exceeds the requirements of the EPA Semi-volatile Pollutants Methods 625 and 8270.

PRODUCT LISTING

SEMI-VOLATILE ORGANICS KIT

(3/90 SOW)

contains 1ml ea. of these mixes:

SV Screening Mix (#31000)
SV Tuning Compound (#31001)
B/N Surrogate Std. Mix
(3/90 SOW) (#31002)
Acid Surrogate Std. Mix
(3/90 SOW) (#31003)
B/N Matrix Spike Mix (#31004)
Acid Matrix Spike Mix (#31005)
SV Internal Standard Mix (#31006)
SV Calibration Mix #1 (#31007)
SV Calibration Mix #2 (#31008)
SV Calibration Mix #3 (#31009)
SV Calibration Mix #4 (#31010)
SV Calibration Mix #5 (#31011)
SV Calibration Mix #6 (#31012)
SV Calibration Mix #7 (#31013)
3,3'-dichlorobenzidine (#31026)

Cat.# 31051 each

Cat.# 31151 w/ data pk.

Are low bleed, excellent inertness,
and long column lifetime too much
to ask for in one capillary column?

No! The Rtx®-5MS offers you the
most column for your money.

RESTEK

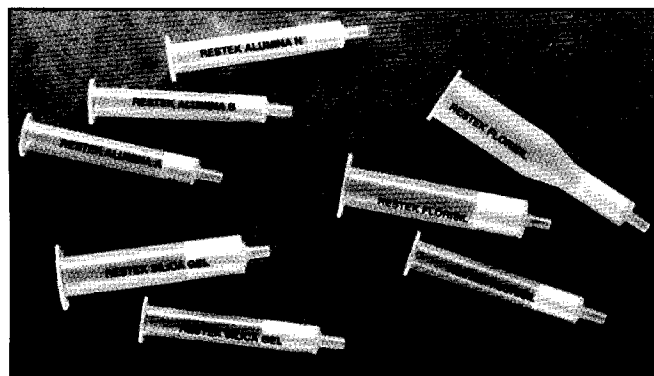
The Advantage

SUPERIOR SPE CARTRIDGES

- Activated Florisil®, Silica Gel and Alumina cartridges.
- Non-contaminating medical grade polypropylene tubes and polyethylene frits.
- Ultra Pure Stainless Steel frits also available.
- Large volume (10ml) cartridges available for large samples.
- Attach easily to the Resprep™ -12T extraction system.

Restek now offers Florisil®, Silica Gel, and Alumina solid phase extraction cartridges for use in EPA Methods 3620A, 3630B, 3610A, and 3611A. Restek's extraction cartridges are made of ultra-clean medical grade polypropylene tubes and polyethylene frits that are quality control tested for purity and cleanliness prior to packing with sorbent. Stainless steel frits are also available for those analyses that require ultra purity. **Large volume tubes (10ml) are ideal** when sample or rinse volumes are greater than the volume that can be held by standard SPE cartridges. This eliminates the need for multiple transfers of the sample. All sorbents are thoroughly quality control tested to ensure reproducible

extraction selectivity from lot-to-lot. All cartridges are flow-tested to ensure a rapid, even flow of solvent through the cartridge for fast, efficient extraction every time. The use of these cartridges with our unique all Teflon® Resprep™ -12T vacuum manifold system will provide the most reliable,



efficient and contamination-free extractions available.

BACKGROUND

Silica Gel, Alumina and Florisil® have been used for many years in chemical laboratories because of their ability to adsorb a variety of compounds. They were popular long before the advent of convenient pre-packed disposable cartridges such as those used for solid phase extraction (SPE).

However, with the introduction of these cartridges, the job of sample clean up is much simpler. This fact is evidenced by the growing list of procedures using these materials, especially in official methods recognized by the United States Environmental Protection Agency (USEPA) and Association of Official Analytical Chemists (AOAC).

FLORISIL® (magnesium silicon oxide, Mg_2SiO_3) is a highly active, polar sorbent. Because of its slightly basic surface, it is good for the adsorption of low to moderately polar species in either an aqueous or non-aqueous matrix. Florisil® has been used to determine the amount of insecticides in grain, aflatoxins in animal feed, fungicides in waste water and flavor compounds in milk.

Innovators of
High Resolution
Chromatography
Products

In this Issue

Superior SPE Cartridges
...pg. 1

Rtx® -1 Bonded Packed
Columns
...pg. 4

Integral HPLC Guard
Column System
...pg. 6

SilcoCan™ Canisters
...pg. 7

Restek Capillary
Columns
...pg. 8

Pro ezGC™ for Windows®
...pg. 10

Sleeves for HP 6890/
5890
...pg. 11

Koni's Korner
...pg. 12

Peak Performers
...pg. 14

Winter
96
INTERNATIONAL



SILICA GEL

(silicon hydroxide, SiOH) is also a polar sorbent. The binding mechanism can be either hydrogen or dipole-dipole interaction. It is primarily used to adsorb species from non-polar solvents like hydrocarbons or substituted hydrocarbons and low polarity esters. Elution solvents are usually more polar and include polar esters, ethers, alcohols, acetonitrile or water. Silica can also be used as a medium strength cation exchanger in aqueous solutions. One important use of Silica is to separate polychlorinated biphenyls from oil samples (PCBs were commonly used in transformer oils to improve their electrical breakdown characteristics).

Another "official" application for silica was recently suggested by the USEPA in their proposed method to determine the oil and grease content of aqueous samples. After the sample is treated by traditional extraction techniques (either SPE or liquid/liquid), the hexane eluent is exposed to Silica in order to fractionate the petroleum from non-petroleum based species.

ALUMINA

(aluminum oxide, Al_2O_3) is available in acidic, basic and neutral grades, and is used in a manner similar to Silica since it has a highly active polar surface. The binding mechanisms also include specific interaction with the pi electrons of aromatic hydrocarbons. This characteristic has been used for applications like crude oil fractionation. Ionic grades

can also be used as low strength ion exchangers. Alumina has often been used for cleaning up homogenates of vegetable samples. Non-polar species are allowed to pass through the Alumina bed while polar extractables from the vegetables are commonly retained on the Alumina. Applications where Alumina has been used include the determination of cosmetic contents, as well as the extraction of basic drugs from blood plasma, organochlorine pesticides from vegetables, and polycyclic aromatic hydrocarbons from animal feeds.

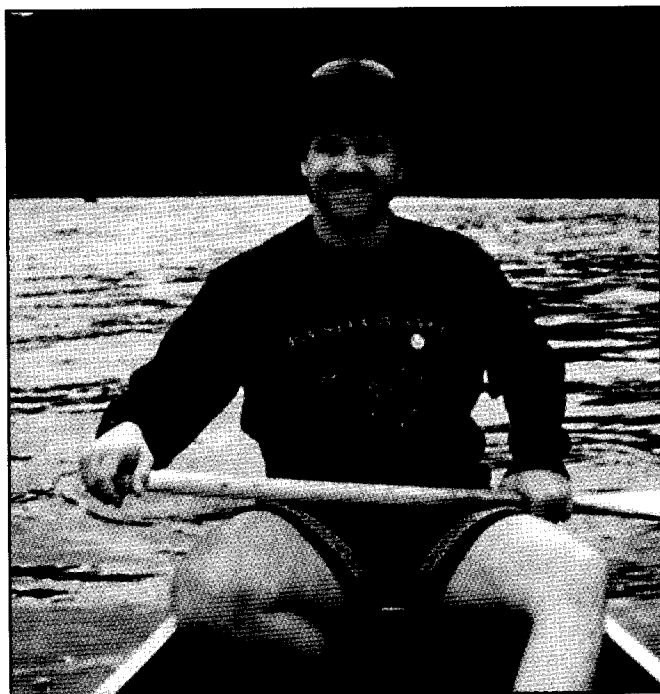
Florasil®, Alumina, and Silica have been used for many years in adsorption or extraction chemistry. These sorbents have stood the test of time and have proven to be some of the most useful products available to the laboratory chemist. Whether used as a clean-up device or for analyte concentration, these materials will continue to demonstrate their versatility and utility in laboratories throughout the world.

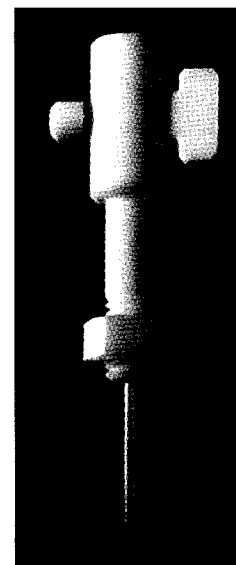
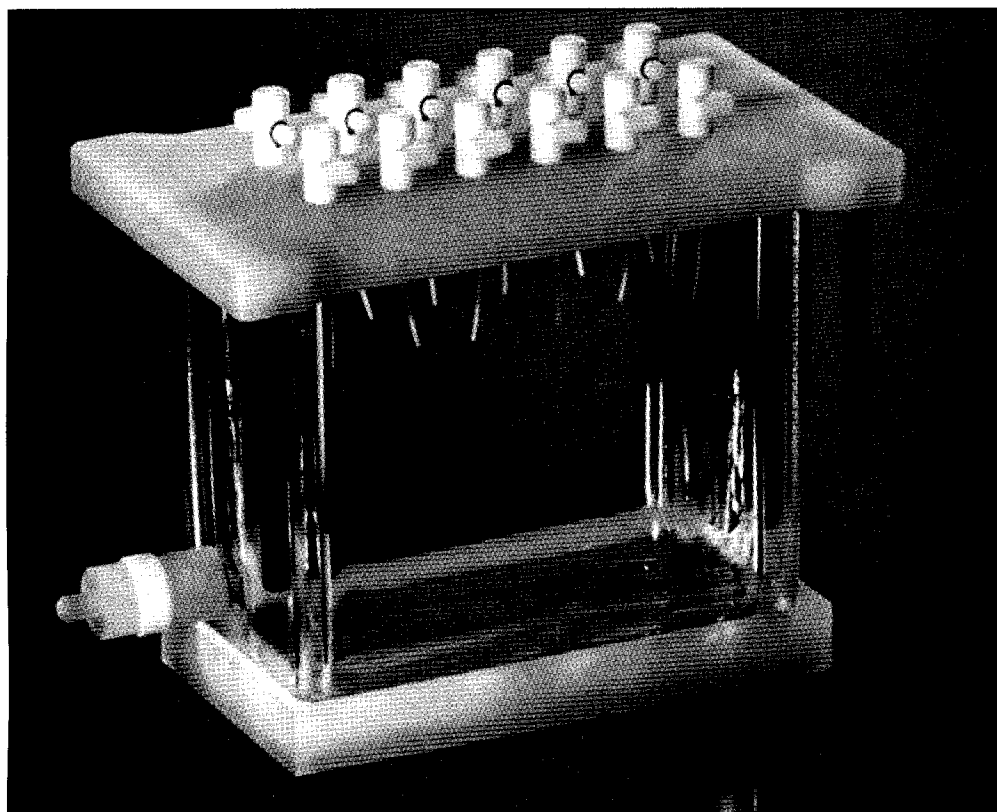
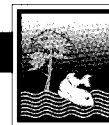
RESPREP™-12T SPE MANIFOLD SYSTEM

Designed specifically for the use of SPE cartridges, such as Florisil®, Silica Gel or Alumina, the 12 position manifold has many new improvements over the traditional manifold system. One major weakness in the traditional manifold system is the control valves. We redesigned the valves for inertness and durability. Our valves, constructed of Teflon®, mount securely to the manifold top via a durable threaded nut that won't break if side torque is applied against the valve. We made precise flow regulation convenient by providing a valve turning tool that screws into the top plate for storage. The base of the valve was designed to allow $\frac{1}{16}$ " OD Teflon® tubing to press-fit inside the valve base. Not only does this create a completely inert sample pathway, but it allows inexpensive $\frac{1}{16}$ " Teflon® tubing to be cut to the appropriate length for different size collection vessels. The complete Teflon® flow path ensures that your sample will not be contaminated from polypropylene or metal valve parts. Other improvements include a polypropylene base plate with rubber feet so the manifold will not slide and scratch the bench, a multipurpose interior sample rack, and built in legs to support the manifold top.

Sean's environmental insight keeps Restek on the leading edge of Sample Prep innovations.

Sean Randall: Environmental Applications Chemist & Sample Prep Product Manager





Sturdy Teflon® valve offers inertness and longevity.

RESPREP™-12T Extraction System for SPE Cartridges: cat.# 24001

Complete Kit includes: propylene top with twelve sturdy Teflon® flow regulation valves, 1/16" Teflon® tubing, glass block with built in vacuum regulator and scratch resistant polypropylene base, multipurpose sample holding rack, and convenient valve turning tool.

Name	Sorbent Mass (mg)	Cartridge Volume (ml)	Frit Style*	Pkg. Size	Catalog Number
Florisil® Cartridges	500	3	PE	50	24031
Florisil® Cartridges	500	3	SS	50	24032
Florisil® Cartridges	500	10	PE	50	24033
Florisil® Cartridges	1000	6	PE	30	24034
Silica Gel Cartridges	500	3	PE	50	24035
Silica Gel Cartridges	500	3	SS	50	24036
Silica Gel Cartridges	500	10	PE	50	24037
Silica Gel Cartridges	1000	6	PE	30	24038
Alumina-N Cartridges	500	3	PE	50	24039
Alumina-N Cartridges	500	3	SS	50	24040
Alumina-N Cartridges	500	10	PE	50	24041
Alumina-A Cartridges	500	3	PE	50	24042
Alumina-A Cartridges	500	3	SS	50	24043
Alumina-A Cartridges	500	10	PE	50	24044
Alumina-B Cartridges	500	3	PE	50	24045
Alumina-B Cartridges	500	3	SS	50	24046
Alumina-B Cartridges	500	10	PE	50	24047

*PE: Polyethylene frits; SS: Stainless steel frits

An Application Note is available detailing the Florisil® cleanup procedure for Organochlorine Pesticides and PCBs via the USEPA CLP methodology. Call your local distributor to receive a copy.

The Advantage

CarboFrit™ Inlet Liner Inserts

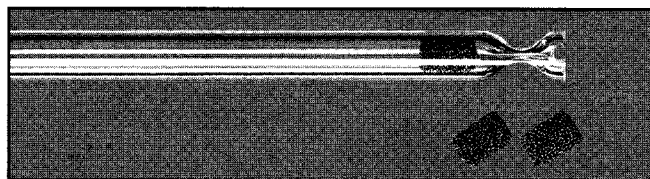
An alternative to glass wool packings for split
& splitless injection liners.

- Exhibits excellent inertness for highly active compounds.
- Allows unimpeded inlet flows.
- Provides low inlet liner pressure drop.
- Improves trapping of high molecular weight contaminants.
- Eliminates off gassing or bleed from deactivation.

Packing split/splitless inlet liners with glass or fused silica wool improves sample vaporization, traps non-volatile sample residue, and is necessary for proper operation of fast autosamplers.¹ However, the benefits to analytical systems are sometimes overshadowed by problems such as adsorption of active compounds, variable packing densities, and off gassing or bleed from deactivation agents. Chromatographers have learned to live with these problems because there were no alternatives – until now.

Researchers at Restek developed an alternative material for packing inlet liners that overcomes many of the limitations of glass or fused silica wool. CarboFrit™ inlet liner inserts provide chromatographers with the same advantages as glass wool: improved vaporization and low pressure drop with superior inertness, higher temperature stability, and better trapping of high molecular weight contaminants. The uniform pore size of these frits guarantees consistent flow through the liner.

The CarboFrit™ inserts are available prepacked in 4mm ID split and splitless liners for HP and Varian GCs or individually as replacement packing. They are easy to install into any inlet liner with a 3.5mm or greater ID* and can be easily replaced if contaminated by dirty sample residue or septum particles. Analysts no longer have to fumble with brittle wool or worry if active sites have been exposed.



CarboFrit™ packing offers the advantages of glass wool but with superior inertness, higher temperature stability, improved retention of sample contaminants, and more consistent packing densities.

The inertness of the CarboFrit™ inserts was evaluated with several active classes of compounds including pesticides and phenols. Endrin, a chlorinated pesticide, is a very good indicator of sleeve inertness. It will readily break down to endrin aldehyde and endrin ketone in an active injection system. A 50pg standard of endrin was injected into five different liners packed with CarboFrit™ inserts. Table I (on page 2) shows the endrin breakdown results for these five liners. The average breakdown was less than 3%, which is well within the 20% breakdown guidelines required in most EPA protocols.

*Liners with IDs less than 3.5mm can be packed on a custom basis.

Innovators of
High Resolution
Chromatography
Products

In this Issue

CarboFrit™ Inlet Liner
Inserts
...pg. 1

Integra-Guard™
Columns
...pg. 3

Septa Comparisons
...pg. 4

Resprep™-C8 Disks
...pg. 6

MXT® vs. Fused Silica
Columns
...pg. 8

Redefining
Environmental HPLC
...pg. 10

Koni's Korner
...pg. 12

Peak Performers
...pg. 14

Spring
96
INTERNATIONAL



Table 1: Endrin Breakdown Results with CarboFrit™ Inserts

Sleeve #	% Endrin Breakdown
1	3.4
2	7.8
3	0.8
4	0.4
5	2.3
Average	2.9
Standard Dev.	2.7

The inertness of CarboFrit™ inserts and fused silica wool was compared by analyzing a mixture of EPA Method 604 phenols. Figure 1 shows the analysis of these phenols. The response of 2,4-dinitrophenol (peak 8) and 2-methyl-4,6-dinitrophenol (peak 10) is significantly higher with the CarboFrit™-packed liner. Even though the glass wool packed into the first liner was deactivated, some active sites were exposed from placing it into the sleeve. These exposed sites can adsorb low levels of active compounds such as 2,4-dinitrophenol and 2-methyl-4,6-dinitrophenol.

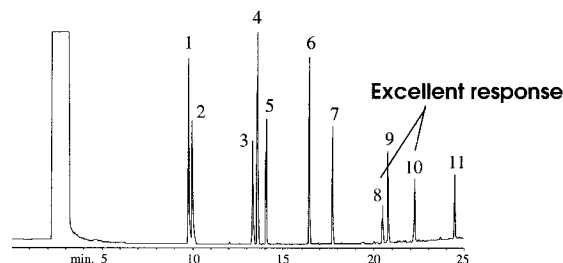
One limitation we discovered was that the CarboFrit™ inserts can retain low concentrations of hydrocarbons above C30 or 4- and 5-ring aromatic hydrocarbons at levels less than 20ng/μl in the splitless injection mode. However, at concentrations commonly used for split injections (>20ng/μl), no retention was observed. For all other classes of compounds and lower molecular weight hydrocarbons, no retention was observed. This is true even at levels below 50pg. Increased injection port temperatures will reduce retention of high molecular weight aromatic compounds at trace levels when using

CarboFrit™ inserts. Elevating injection port temperatures to as high as 400°C will ensure that these components completely elute from the injector. Unlike deactivated glass wool that can release siloxane deactivants at high injection port temperatures, CarboFrit™ inserts show no background contamination peaks even at injection port temperatures of 350°C. CarboFrit™-packed liners can be oxidized at high temperatures in the presence of room air. Therefore, the injector should be cooled before installing or replacing the CarboFrit™ insert. In addition, high-purity carrier gas and oxygen-removal traps should be used on carrier gas lines.

Restek has developed an alternative liner packing that offers all the positive features of wool without the adsorption problems. The CarboFrit™ insert is easy to install and replace and can be used for a wide range of applications including alcohols, amines, pesticides, esters, dioxins, triglycerides, fatty acid methyl esters, and hydrocarbons. Call Restek to discuss how these inserts can help with your analyses.

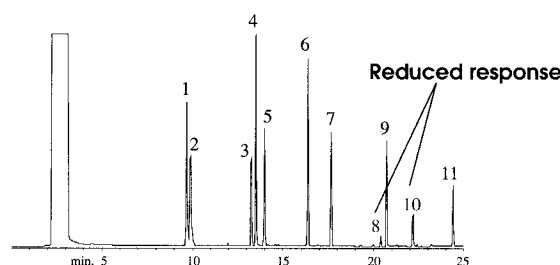
Figure 1: CarboFrit™ inserts show excellent response to active phenols including 2,4-dinitrophenol and pentachlorophenol compared to liners packed with deactivated glass wool.

Liner packed with a CarboFrit™ insert



- | | | |
|-------------------|----------------------------|--------------------------------|
| 1. phenol | 4. 2,4-dimethylphenol | 8. 2,4-dinitrophenol |
| 2. 2-chlorophenol | 5. 2,4-dichlorophenol | 9. 4-nitrophenol |
| 3. 2-nitrophenol | 6. 4-chloro-3-methylphenol | 10. 2-methyl-4,6-dinitrophenol |
| | 7. 2,4,6-trichlorophenol | 11. pentachlorophenol |

Liner packed with deactivated wool



30m, 0.25mm ID, 0.25μm XTI®-5 (cat.# 12223). 1.0μl splitless injection of method 604 phenols. **Oven temp.:** 50°C (hold 4 min.) to 250°C @ 8°C/min.; **Inj. & det. temp.:** 275°C; Carrier gas: H₂; **Linear velocity:** 24cm/sec. set @ 50°C; **Splitless hold time:** 1 min.; **Split vent flow:** 50ml/min.

¹ Grob, K., "Sample Evaporation in Hot GC Injectors". *The Restek Advantage*, Winter 1996, pp. 12-13.



Prepacked Sleeves:

4mm Splitless for HP	for Varian	4mm Gooseneck for HP
20772-209.1	20904-209.1	20798-209.1
20773-209.5	20905-209.5	20799-209.5
20774-209.25	20906-209.25	20800-209.25

The catalog numbers above ending in ".1" are single packs, ".5" are 5-packs, and ".25" are 25-packs.

To order other sleeves >3.5mm ID prepacked with CarboFrit™ inserts, add the appropriate suffix to the inlet sleeve catalog number.

Each	-209.1
5-pack	-209.5
25-pack	-209.25

Replacement Frits & Accessories:	Cat.#
CarboFrit™ (10-Pack)	20295
Puller/Packing tool	21642