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

** This special offer expires May 30, 1997.*

Matt Piserchio, HPLC Chemist and Product Line Manager

Randy Romesberg HPLC Chemist

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.

Please call

your local distributor

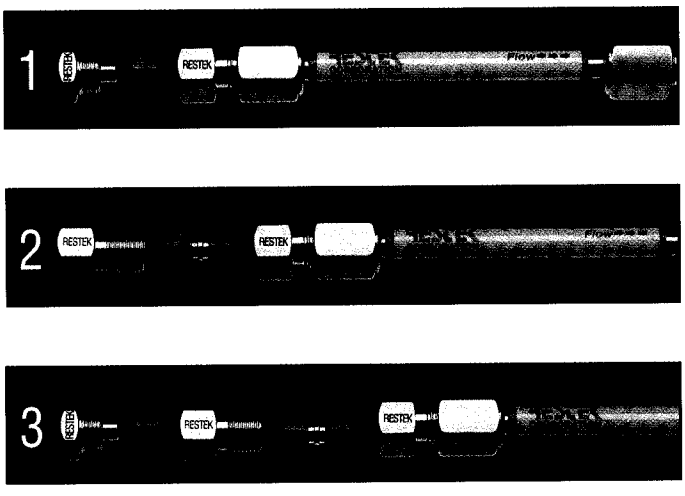
for more information or HPLC Technical Service

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

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.

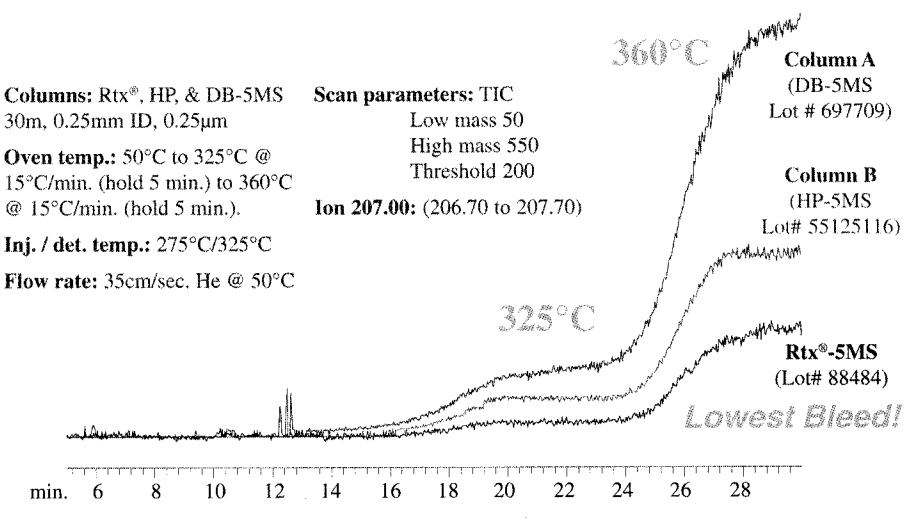
Bleed

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 parameters (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!



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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 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.

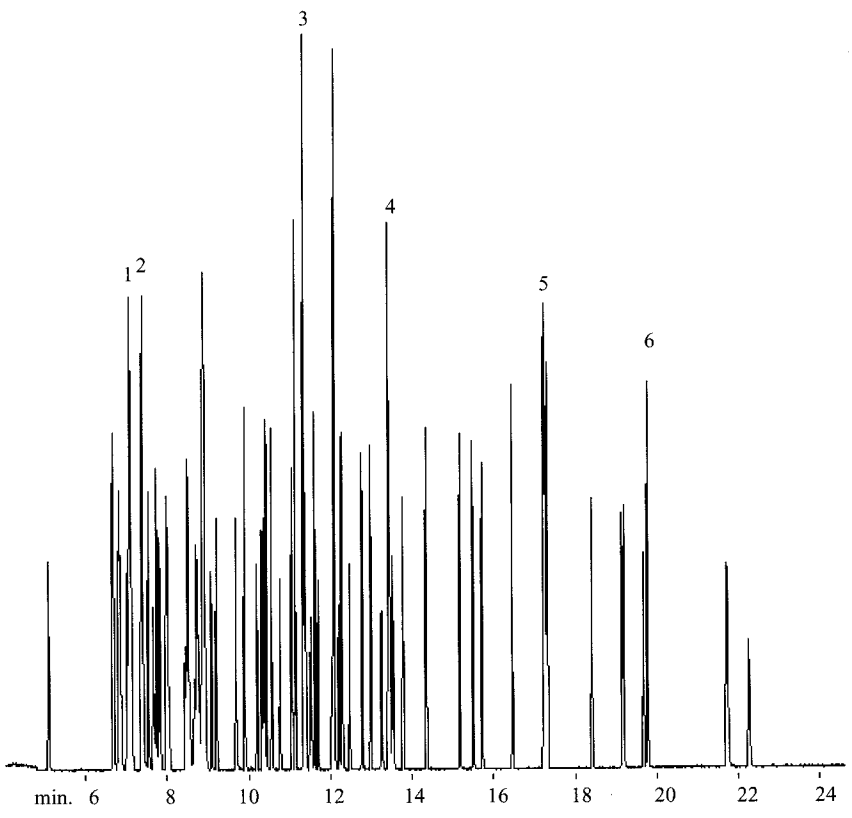
TABLE I: Rtx®-5MS demonstrates better response of active environmental compounds.

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

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. 1,4-dichlorobenzene-d4
2. naphthalene-d8
3. acenaphthene-d10
4. phenanthrene-d10
5. chrysene-d12
6. perylene-d12

Analysis courtesy of Inchcape 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.

PRODUCT LISTING

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
	1.00	cat.# 12651	cat.# 12654
0.53mm	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

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.

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.

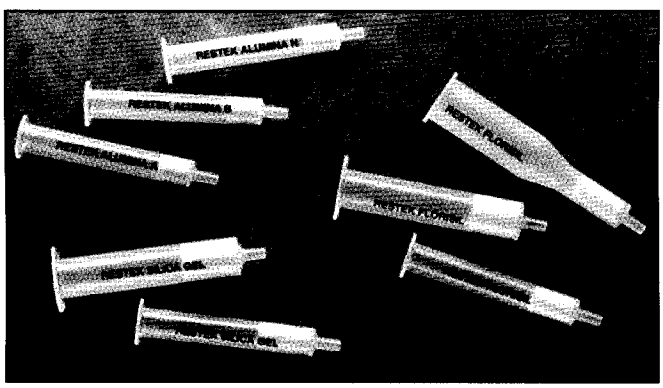
Are low bleed, excellent inertness, and long column lifetime too much to ask for in one capillary column? No! The Rtx®-5MS offers you the most column for your money.

The Restek 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

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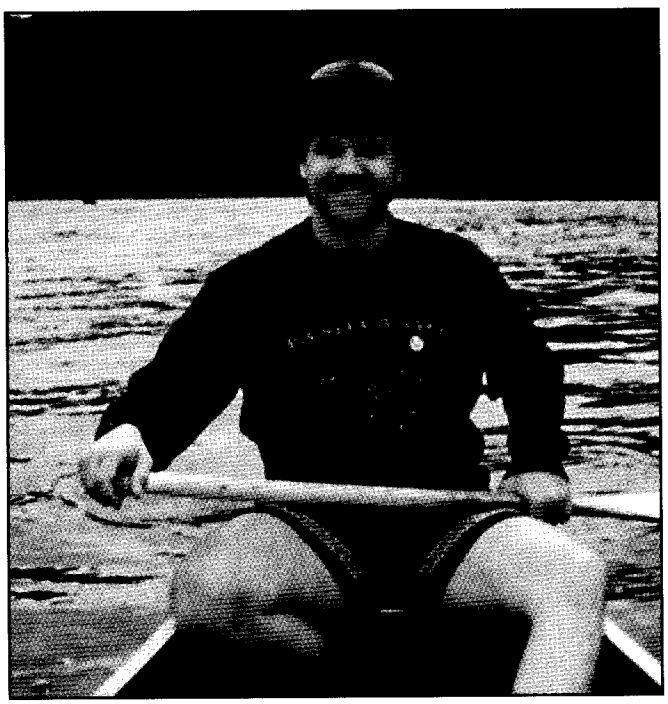
SILICA GEL
(silicon hydroxide, SiOH) is also a polar sorbent. The binding mechanism can be either hydrogen or dipole-dipole interaction. It is primarily used to adsorb species from non-polar solvents like hydrocarbons or substituted hydrocarbons and low polarity esters. Elution solvents are usually more 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.

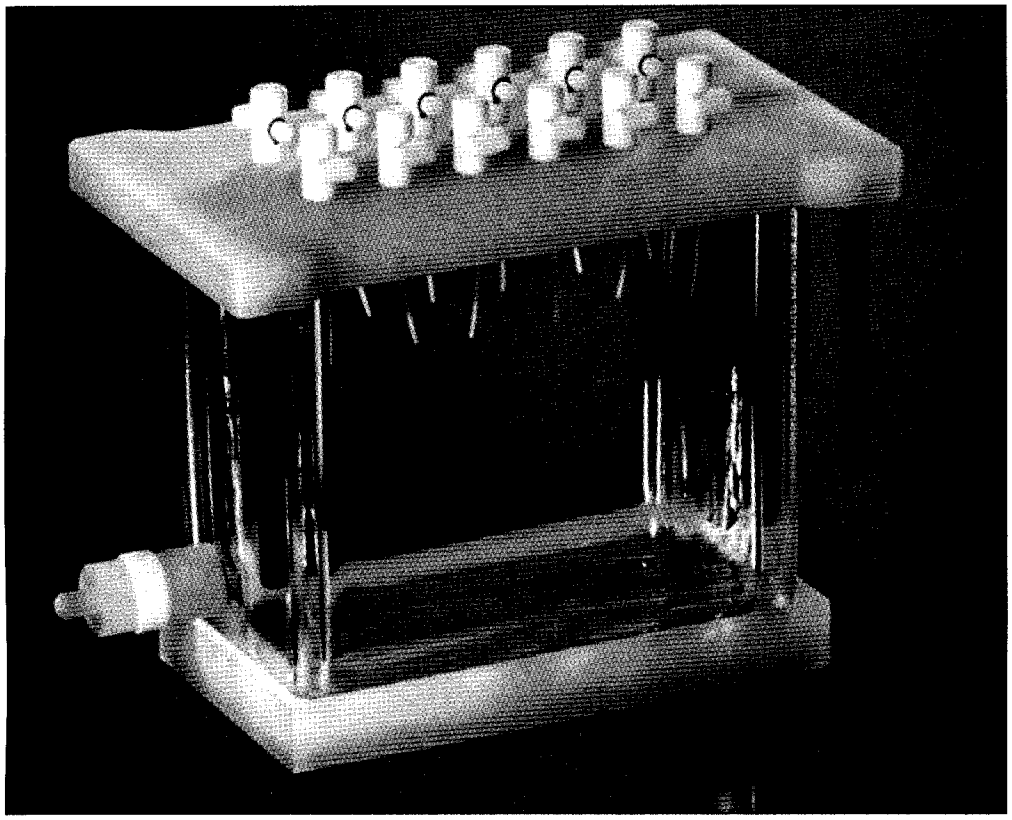
Florisil®, Alumina, and Silica have been used for many years in adsorption or extraction chemistry. These sorbents have stood the test of time and have proven to be some of the most useful products available to the laboratory chemist. Whether used as a clean-up device or for analyte concentration, these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

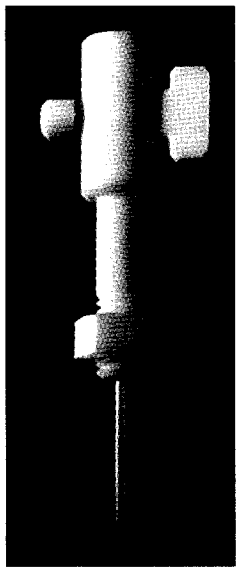


Sean's environmental insight keeps Restek on the leading edge of Sample Prep innovations.

Sean Randall: Environmental Applications Chemist & Sample Prep Product Manager

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 $^1/_{16}$" OD Teflon® tubing to press-fit inside the valve base. Not only does this create a completely inert sample pathway, but it allows inexpensive $^1/_{16}$" 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.





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

Innovators of High Resolution Chromatography Products

FRIT™ Inlet Liner Inserts

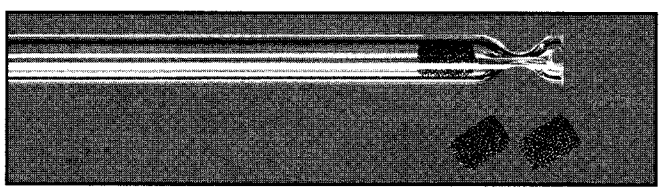
An alternative to glass wool packings for split & splitless injection liners.

- 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.*

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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**

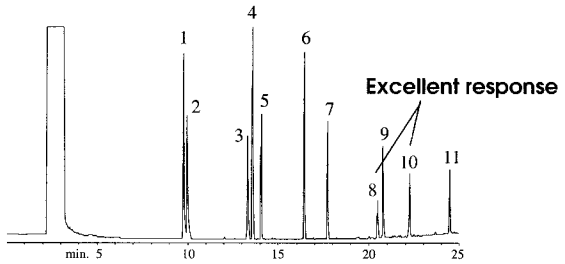
The inertness of CarboFrit™ inserts and fused silica wool was compared by analyzing a mixture of EPA Method 604 phenols. Figure 1 shows the analysis of these phenols. The response of 2,4-dinitrophenol (peak 8) and 2-methyl-4,6-dinitrophenol (peak 10) is significantly higher with the CarboFrit™-packed 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.

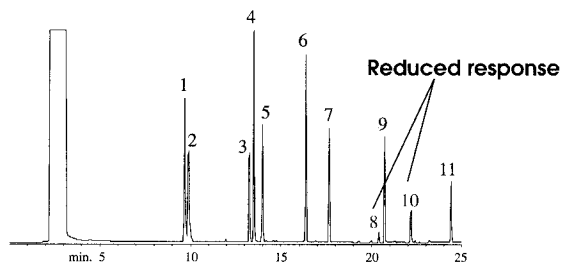
Figure 1: CarboFrit™ inserts show excellent response to active phenols including 2,4-dinitrophenol and pentachlorophenol compared to liners packed with deactivated glass wool.

Liner packed with a CarboFrit™ insert



1. phenol
2. 2-chlorophenol
3. 2-nitrophenol
4. 2,4-dimethylphenol
5. 2,4-dichlorophenol
6. 4-chloro-3-methylphenol
7. 2,4,6-trichlorophenol
8. 2,4-dinitrophenol
9. 4-nitrophenol
10. 2-methyl-4,6-dinitrophenol
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: H_2; **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.

CarboFrit™ Inlet Liners

Prepacked Sleeves:

4mm Splitless for HP	4mm Splitless 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