the RESTEK ADVANTAGE

2007.03

Analytical Alternatives

Get selectivity! 5 stationary phases on <2µm UHPLC columns.

More choices for biodiesel: new metal columns with optional built-in retention gaps.

Fast or faster? Options for chlorinated pesticides.

and much more inside…

RESTEK

Chromatography Products

the Restek Advantage
2007.03

IN THIS ISSUE

Retention Cross-over Phenomenon in Gas Chromatography–Can the Mystery be Revealed? Part 2

By Werner Engewald, Ph.D., Professor Emeritus, University of Leipzig, Institute of Analytical Chemistry, Leipzig, Germany; engewald@uni-leipzig.de

In the last issue of the Restek Advantage (2007.02), I showed some examples of the cross-over phenomenon on polar (polyethyleneglycol) columns. Here in Part 2, we will examine the cross-over phenomenon on nonpolar columns.

It is known to a lesser extent that changes in peak elution order also occur on nonpolar or weakly polar stationary phases for hydrocarbons that differ only in their carbon skeleton, e.g. aliphatic versus cyclic compounds or cyclic compounds differing in their ring number. The terpenes sabinene, β-pinene and myrcene are given as an example in Figure 1. The cross-over effect was observed on a polydimethylsiloxane phase with 5% phenyl (60m, 0.25mm ID, 1µm film thickness) as well as on a 100% polydimethylsiloxane phase (60m, 0.32mm ID, 0.5µm film thickness). The column temperature was increased from 90°C to 160°C using isothermal mode. The elution order changed from sabinene, β-pinene, myrcene at 90°C to myrcene, sabinene, β-pinene at 160°C. What could be the reason for this effect? A closer look at the molecular structure shows that sabinene and β-pinene are double ring systems whereas myrcene is an aliphatic hydrocarbon.

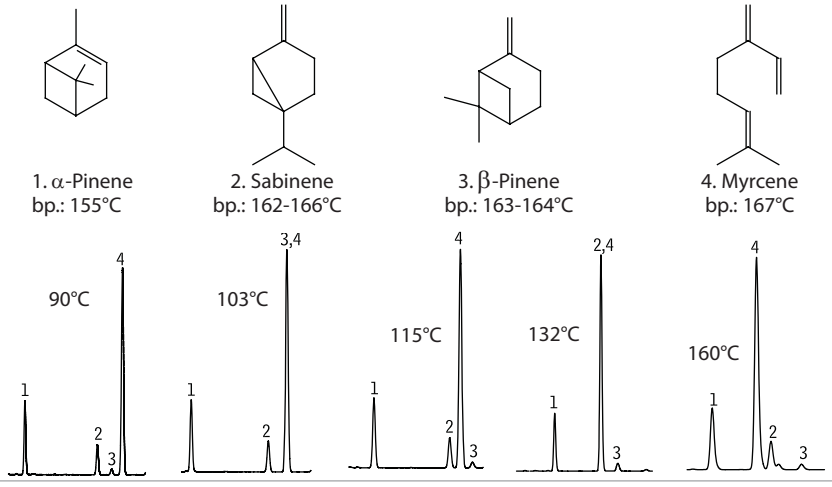
Other interesting analyte pairs prone to cross-over on methylsiloxane phases at different column temperatures are *o*-xylene/*n*-nonane, naphthalene/dodecane, as well as 1,2,3-trimethylbenzene/*n*-decane. In the latter case we also observe coelution and cross-over at different temperature programming rates. At a heating rate of 2°C/min., *n*-decane elutes before 1,2,3-trimethylbenzene, at 5°C/min. coelution occurs, and at 20°C the aromatic hydrocarbon is the first peak (100% PDMS column, 12m, 0.2mm ID, 0.33µm film thickness, starting temperature 35°C). It seems obvious that the geometry of the molecule, e.g. cyclic versus open chain, contributes to the cross-over phenomenon.

Nevertheless, I have this long-standing friendly discussion with a former student of mine, who persistently points out that the examples we have been looking at so far are always pairs of conjugated versus nonconjugated compounds and that π interactions, specifically with phenyl modified phases, should be taken into account.

Let's, therefore, go back to the structure of substances presented in Figure 2: they are exclusively saturated aliphatic and alicyclic hydrocarbons. The data in Figure 2 are from Hively and Hinton (1968) and in that paper the relative retention and retention indices of approxi-

Continued on page 23.

Figure 1 Elution order on a 100% PDMS column at various temperatures (isothermal GC)



New MXT®-Biodiesel TG Column Line

Stable to 430°C, for high temperature analyses.

By Barry L. Burger, Petroleum Innovations Chemist

- Sharp glyceride peaks give more accurate quantitation.
- Stable at 430°C; more robust than fused silica at high temperatures.
- Integra-Gap™ built-in retention gap eliminates manual connection.

Restek has raised the bar with a new high-temperature MXT®-Biodiesel TG column line to complement our fused silica column line for biodiesel analysis. These new MXT®-Biodiesel TG columns are stable to 430°C and offer unique retention gap options that minimize dead volume and leaks. Choose either a 0.32mm column factory-coupled to a 0.53mm retention gap, or select a single unit 0.53mm column featuring Integra-Gap™, a built-in retention gap that eliminates the need for a connector. Both designs are extremely stable at high temperatures and produce fast elution times and sharp peaks for high molecular weight glycerides.

Unsurpassed Stability

The high temperature programs required for analysis of biodiesel oils (B100) by either ASTM D-6584 or EN-14105 methodology present a significant challenge to the analytical column. High-temperature fused silica tubing breaks down under these extreme conditions, but the metal MXT® tubing does not degrade, even at temperatures up to 430°C (Figure 1). This allows analysts to bake out any residue eluting after the triglycerides, preventing carryover without damaging the column.

So how well do the MXT®-Biodiesel TG columns perform? We conducted a benchmarking experiment comparing an MXT®-Biodiesel TG column with Integra-Gap™ to a high-temperature fused silica column coupled to a conventional 0.53mm retention gap. Methodology followed ASTM method D-6584, except the final temperature was modified to 430°C. Both columns were subjected to 100 temperature cycles up to 430°C and derivatized B100 was injected.

This evaluation was performed using a Shimadzu 2010 gas chromatograph equipped with a flame ionization detector, a model AOC 20i + S autosampler with a 10µL SGE syringe and 42mm 26-gauge needle, and a cold on-column programmable injector with a stainless steel injector insert. A Parker hydrogen generator supplied the carrier gas. Peak symmetry and retention time were evaluated as indicators of thermal stability.

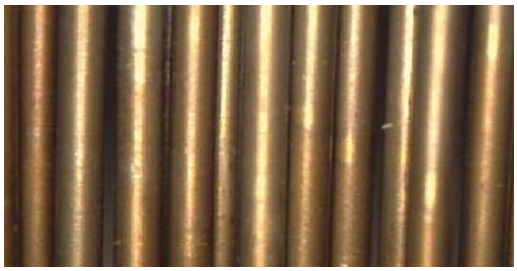
Peak symmetry of butanetriol on a commercial high-temperature fused silica column deteriorates after just 20 injections, compared to the excellent symmetry that is maintained on the MXT®-Biodiesel TG column (Figure 2). In addition to peak shape, retention time stability was used to evaluate column performance. The decrease in retention time seen on the high-temperature fused silica column indicates the liquid phase is being lost (Figure 3). In contrast, the consistent retention times obtained on the MXT®-Biodiesel TG column demonstrate its stability. Practically, this translates into reliable performance and longer column lifetimes.

Analytical Alternatives

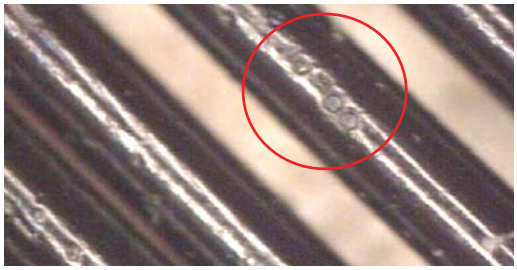
Factory connected 0.32mm MXT®-Biodiesel TG columns & 0.53mm retention gaps

For accurate analysis of heavy triglycerides, on-column injection is required. ASTM D-6584 describes the use of a 0.32mm analytical column coupled with a 0.53mm retention gap. The 0.53mm ID retention gap allows the cool on-column technique to be used, but care must be taken to minimize dead volume and to establish a leak-tight connection. Restek's 0.32mm MXT®-Biodiesel TG columns are factory-coupled to a 0.53mm MXT® retention gap with an Alumaseal™ connector, ensuring a leak-

Figure 1 MXT®-Biodiesel TG columns are undamaged by high thermal cycles compared to high-temperature fused silica columns which breakdown under the same conditions.



MXT®-Biodiesel TG columns are undamaged by high thermal cycles.



HT fused silica columns, labeled as stable to 430°C, show pitting and breakdown.

100 temperature cycles to 430°C totaling 500 minutes at maximum temperature.

Figure 2 Stable and consistent peak shape for the internal standard butanetriol gives you more accurate quantitation.

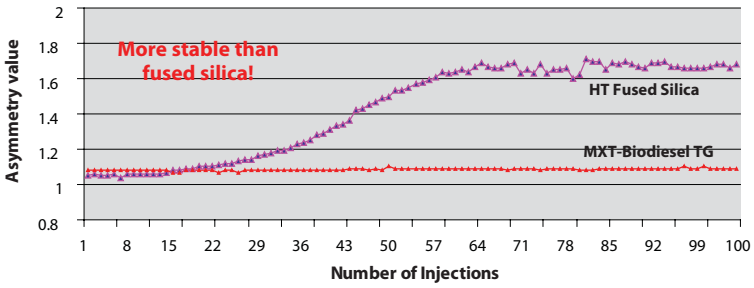


Figure 3 Retention time is stable on a metal MXT®-Biodiesel TG column, even after 100 cycles up to 430°C.

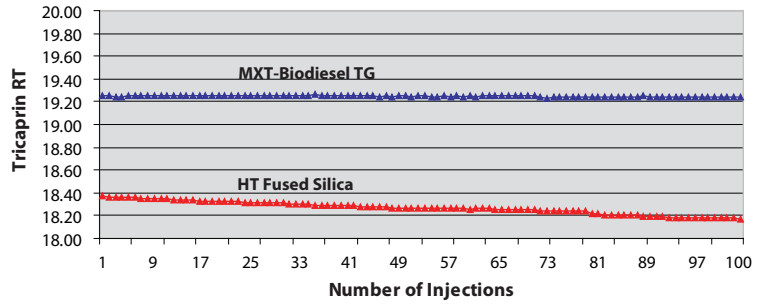
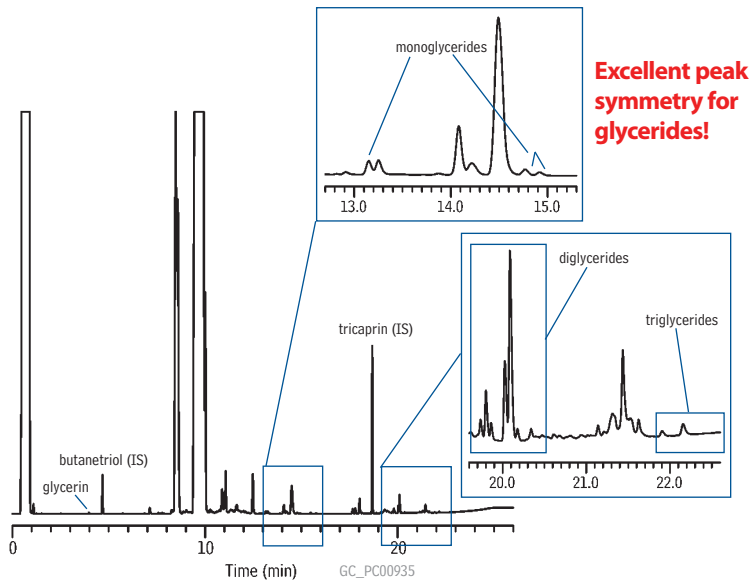
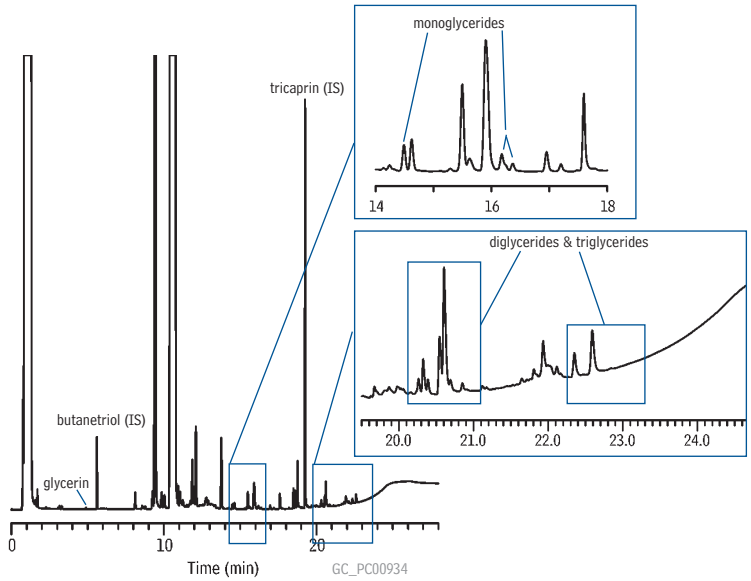


Figure 4 Derivatized B100 samples resolve well on the 0.32mm MXT®-Biodiesel TG column, which is factory-coupled to a 0.53mm MXT® retention gap.



Column: MXT®-Biodiesel TG 10m, 0.32mm ID, 0.1µm with 2m x 0.53mm retention gap
Sample: B100 + IS Butanetriol & Tricaprin derivatized with MSTFA as per ASTM D-6584
Instrument: Shimadzu 2010
Inj.: 1.0µL cool on-column; Inj. temp.: oven track
Carrier gas: hydrogen, constant flow
Flow rate: 4mL/min.
Oven temp.: 50°C (hold 1 min.) to 180°C @ 15°C/min., to 230°C @ 7°C/min., to 430°C @ 30°C/min. (hold 5 min.)
Det.: FID @ 430°C

Figure 5 Equivalent chromatographic quality on the 0.53mm MXT®-Biodiesel TG analytical column with Integra-Gap™



Column: MXT®-Biodiesel TG 13m, 0.53mm ID, 0.16µm with built-in 2m Integra-Gap™ (total column length 15m)
Sample: B100 + IS Butanetriol & Tricaprin derivatized with MSTFA as per ASTM D-6584
Instrument: Shimadzu 2010
Inj.: 1.0µL cool on-column; Inj. temp.: oven track
Carrier gas: hydrogen, constant flow
Flow rate: 4mL/min.
Oven temp.: 50°C (hold 1 min.) to 180°C @ 15°C/min., to 230°C @ 7°C/min., to 430°C @ 30°C/min. (hold 5 min.)
Det.: FID @ 430°C
(Data acquired on prototype column)

tight connection. Target analytes resolve well and the solvent and triglyceride peaks show excellent symmetry (Figure 4).

0.53mm MXT®-Biodiesel TG columns

The 0.53mm MXT®-Biodiesel TG columns are a simpler alternative to using a 0.32mm column coupled to a 0.53mm retention gap. Restek applied Integra-Gap™ technology to the 0.53mm MXT®-Biodiesel TG columns, eliminating the column coupling. These single unit leak-proof columns feature a built-in retention gap, reducing the risk of peak broadening and tailing. Chromatography from the 0.53mm MXT®-Biodiesel TG with Integra-Gap™ technology (Figure 5) is excellent and comparable to that obtained on the 0.32mm ID column in Figure 4.

Conclusion

As demonstrated, for high temperature GC analysis, the metal MXT®-Biodiesel TG column is a rugged column that withstands the harsh temperatures required for total residual glycerin analysis. The column has the resolution needed for accurate, reliable results and is more stable at high temperatures than competitive fused silica columns, leading to longer column lifetimes. To improve the reliability and robustness of your biodiesel analyses, try one of our MXT®-Biodiesel TG columns.

MXT®-Biodiesel TG Column

ID	df (µm)	temp. limits	14-Meter w/2m Integra-Gap™
0.53mm	0.16	-60 to 380/430°C	70289

thank **you**

Instrument provided courtesy of Shimadzu

www.shimadzu.com

Optimize Selectivity & Efficiency in UHPLC Separations

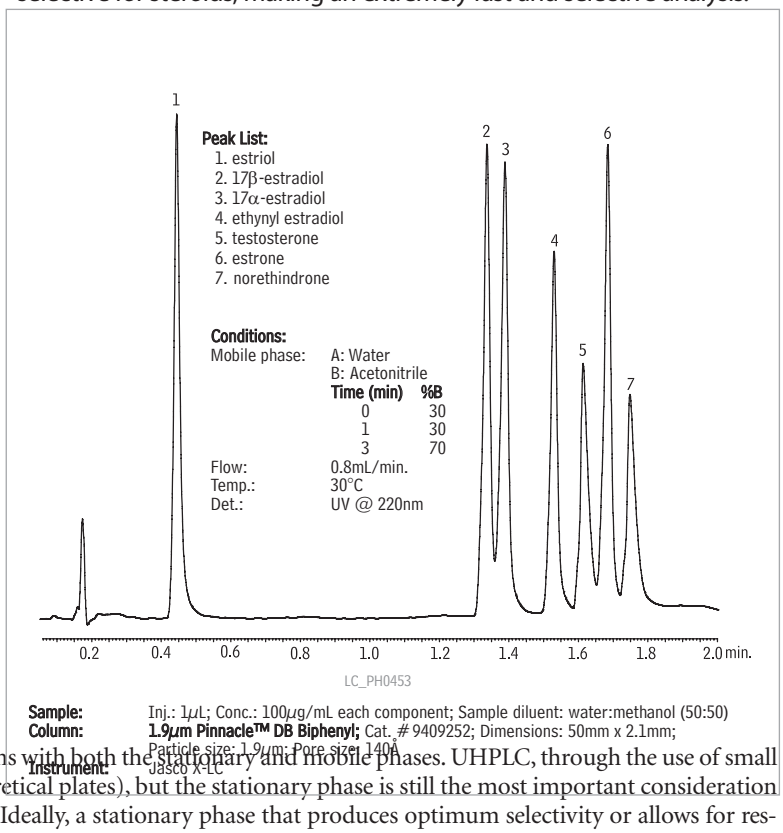
With More Stationary Phase Choices on 1.9µm Pinnacle™ DB HPLC Columns

By Rick Lake, Pharmaceutical Innovations Chemist

- Largest variety of stationary phases for UHPLC.
- Faster analyses, uncompromised chromatography.
- 100% Restek manufactured—from base silica to final packed column.

Since the late 1960s continual advancements have been made in HPLC column technology, and over time the trend has been toward smaller particle sizes. This trend has led us to where we are today—Ultra-High Performance Liquid Chromatography (UHPLC). UHPLC is a milestone in the evolution of LC in that columns packed with <2µm particles, used with instrumentation capable of handling the resulting high back pressures, make possible extremely fast and efficient separations. UHPLC is a very powerful tool for today's practicing chromatographer, as it can significantly increase the efficiency of a chromatographic separation. In addition, the wider range of usable flow rates makes high speed separations possible. However, in light of this new technology, it is important that we do not forget the importance of selectivity. In this article, we will review the significance of selectivity in obtaining acceptable resolution and demonstrate how having choices in stationary phase allows you to maximize the benefits of UHPLC.

Figure 1 Restek's 1.9 µm Pinnacle™ DB Biphenyl columns are highly selective for steroids, making an extremely fast and selective analysis.



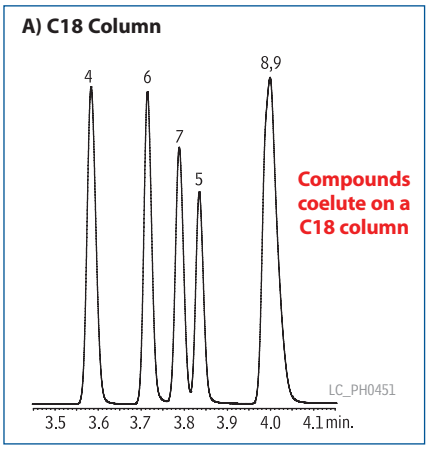
In past articles we have discussed the physical advantages that are driving interest in small particles, mainly the influence of particle size on usable flow rates and peak efficiency. Although small particles have made faster separations possible, selectivity has the greatest effect on resolution. Selectivity, in turn, is governed predominantly by analyte interactions with both the stationary and mobile phases. UHPLC, through the use of small particle columns, does maximize efficiency (e.g. theoretical plates), but the stationary phase is still the most important consideration when attempting to resolve mixtures of compounds. Ideally, a stationary phase that produces optimum selectivity or allows for resolution of compounds in a timely manner should be selected.

Previously, some advantages of selectivity in specific separations have been noted. For example, the use of a unique Biphenyl stationary phase has shown excellent selectivity for aromatic or fused ring compounds. When using the Biphenyl stationary phase and combining it with the heightened efficiencies of the 1.9µm Pinnacle™ DB column, we can produce highly selective and fast separations of steroids (Figure 1). A Pinnacle™ DB 1.9µm Biphenyl column can separate a test mix of seven hormones in under 2 minutes, a feat not possible through C18 selectivity.

Another example of unique selectivity available on a 1.9µm particle size column is the PFP Propyl (pentafluorphenyl propyl) stationary phase for halogenated drug compounds. This phase is very selective and retentive for organohalogens or other compounds containing basic or electronegative functionalities. To demonstrate heightened selectivity for halogenated drug compounds, we assayed a test mix of eight benzodiazepines and two metabolites, a mix commonly assayed on a C18 colum, in just over 4 minutes with complete resolution (Figure 2). To get the same level of selectivity from a C18 column, a shallower gradient would be needed, prolonging the analysis time. Since the selectivity of the Pinnacle™ DB 1.9µm PFP Propyl column elutes the benzodiazepines in quick succession, a simple gradient still allows for the earlier elution of the more polar metabolites, while maintaining a fast overall run time.

Restek is committed to giving the practicing chromatographer choices, and has therefore sought to deliver the widest selection of stationary phases available with <2µm particle sizes. The goal of chromatography is always to resolve compounds of interest in the fastest time possible. By combining the benefits of UHPLC with Restek's complement of unique stationary phase choices, faster separations become a reality.

Figure 2 Fast, selective analysis of benzodiazepines is made possible by combining the speed of UHPLC with the enhanced selectivity of the 1.9µm Pinnacle™ DB PFP Propyl column.



Peak List:	Conc (µg/ml)
1. 7-amino clonazepam*	20
2. 7-amino flunitrazepam*	20
3. bromazepam	100
4. oxazepam	100
5. lorazepam	100
6. clonazepam	100
7. nitrazepam	100
8. nordiazepam	100
9. flunitrazepam	100
10. diazepam	100

* metabolite

Sample:
Inj.: 1µL
Conc.: as listed
Sample diluent: starting mobile phase (80:20 A:B)

Column: **A. C18 Column**
B. 1.9µm Pinnacle™ DB PFP Propyl
Cat. #: A. 9414212
B. 9419212
Dimensions: 100mm x 2.1mm
Particle size: 1.9µm
Pore size: 140Å

Instrument: Jasco X-LC

Conditions:
Mobile phase: A: 0.1% formic acid in water
B: 0.1% formic acid in acetonitrile

Time (min.)	%B
0	20
1	20
6	80

Flow: 0.6 mL/min.
Temp.: 40°C
Det.: UV @254 nm

1.9µm Pinnacle™ DB HPLC Columns

Physical Characteristics:

particle size: 1.9µm
pore size: 140Å
endcap: yes
pH range: 2.5 - 7.5
temperature limit: 80°C

1.9µm Pinnacle™ DB C18 column, 2.1mm	cat. #
30mm	9414232
50mm	9414252
100mm	9414212
1.9µm Pinnacle™ DB Silica column, 2.1mm	**cat. #**
30mm	9410232
50mm	9410252
100mm	9410212
1.9µm Pinnacle™ DB PFP Propyl column, 2.1mm	**cat. #**
30mm	9419232
50mm	9419252
100mm	9419212
1.9µm Pinnacle™ DB Biphenyl column, 2.1mm	**cat. #**
30mm	9409232
50mm	9409252
100mm	9409212
1.9µm Pinnacle™ Aqueous C18 column, 2.1mm	**cat. #**
30mm	9418232
50mm	9418252
100mm	9418212

More phases coming soon!

Faster Organochlorine Pesticide Sample Throughput

On New Rtx®-CLPesticides & Rtx®-CLPesticides2 Columns

By Jason Thomas, Environmental Innovations Chemist

- Dramatically improve sample throughput.
- Results in <7min. by conventional analysis, or <5min. using the Gerstel MACH system.
- Outstanding resolution on all columns.

As the environmental testing market continues to be very competitive, laboratory operating costs are a critical concern. Increasing sample throughput is one way to reduce costs, and shortening analytical run time is an effective way to do this. Here we offer methods for reducing run time for the organochlorine pesticides analyzed under US EPA Method 8081. The significant reduction in both analysis time and more significantly, cycle time, offered here is a major benefit for environmental laboratories.

Restek developed the Rtx®-CLPesticides and Rtx®-CLPesticides2 column pair specifically for chlorinated pesticides. These phases were designed to separate the isomers and the structurally similar pairs on the list of target analytes. Here we introduce new film thicknesses with optimized phase ratios for some of the columns in this line. Using these new stationary phase film thicknesses and the optimized run conditions shown, the 20 compounds in US EPA Method 8081 can be separated to baseline in less than 7 minutes (Figure 1). This allows rapid analysis without sacrificing column capacity, which translates, of course, into much improved sample throughput for your laboratory.

An Even Faster Alternative

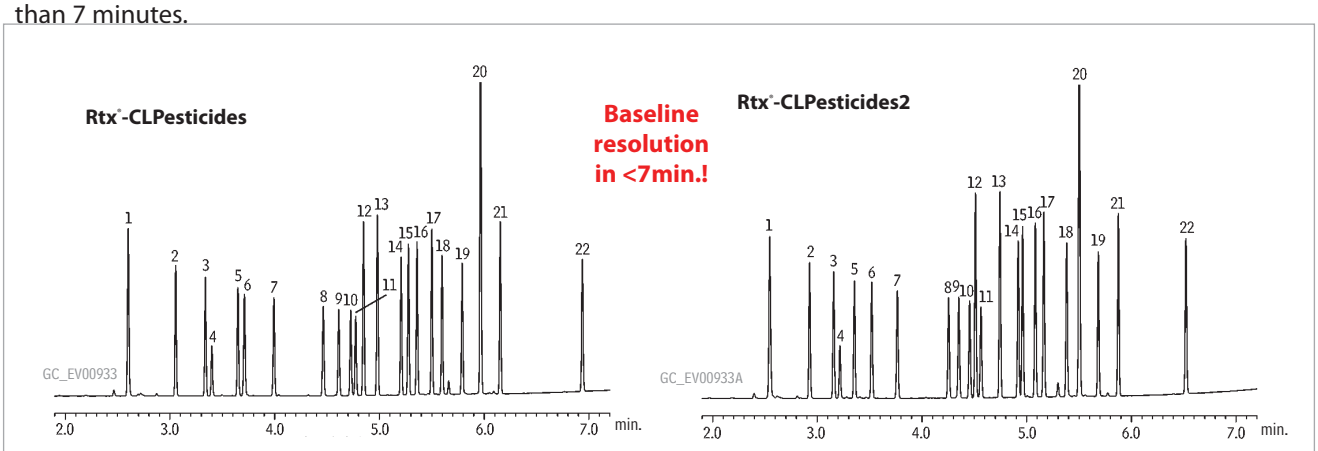
In the attempt to obtain faster analytical run times, several different concepts have been introduced to improve the stock performance of standard GCs. One of the most recent and versatile ideas is the low thermal mass method by Gerstel using an apparatus called the MACH, (Modular Accelerated Column Heater) (Figure 2). This system operates by heating the capillary column outside of the GC oven in a small column module mounted on the oven door.

This apparatus provides several important advantages. First, due to the low thermal mass of the unit, very rapid heating and cooling times can be realized, which significantly shortens cycle times. Second, because of the way the column is wrapped, very uniform heating occurs, which eliminates the eddies and hot spots produced in a conventional GC oven. Finally, since the column modules are independently controlled, two different temperature programs can be run simultaneously, which allows each column to be optimized individually.

Restek applied this novel MACH technology to EPA Method 8081 using an Rtx®-CLPesticides and Rtx®-CLPesticides2 column pair. Almost 100% baseline resolution was obtained for all 22 pesticides and surrogates, on both columns, in under five minutes (Figure 3). This combination of ultra-fast analysis time and outstanding resolution is a result of the unique selectivity and high efficiency of the phases combined with the narrow peaks associated with ultra-rapid ramp rates.

Regardless of whether you choose to embrace the new fast-GC technology, or continue to adhere to more conventional GC, Restek Rtx®-CLPesticides and Rtx®-CLPesticides2 columns can provide exceptional performance and very rapid run times when analyzing chlorinated pesticides.

Figure 1 Baseline resolution of organochlorine pesticides on the 0.18mm ID Rtx®-CLPesticides column pair in less than 7 minutes.



1. 2,4,5,6-tetrachloro-*m*-xylene (SS)
2. α-BHC
3. γ-BHC
4. β-BHC
5. δ-BHC
6. heptachlor
7. aldrin
8. heptachlor epoxide (isomer B)
9. γ-chlordane
10. α-chlordane
11. endosulfan I
12. 4,4'-DDE
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. endosulfan sulfate
20. methoxychlor
21. endrin ketone
22. decachlorobiphenyl (SS)

Column: Rtx®-CLPesticides 30m, 0.32mm ID, 0.32µm (cat.# 11141) and Rtx®-CLPesticides2 30m, 0.32mm ID, 0.25µm (cat.# 11324) with 5m x 0.32mm ID Rxi™ deactivated guard tubing (cat.# 10039), connected using Universal "Y" Press-Tight® connector (cat.# 20405-261)
Sample: Organochlorine Pesticide Mix AB #2, 8-80µg/mL each component in hexane/toluene (cat.# 32292), Pesticide Surrogate Mix, 200µg/mL each component in acetone (cat.# 32000)
Inj.: 1.0µL splitless (hold 0.3 min.), 4mm single gooseneck inlet liner (cat.# 20799); Inj. temp.: 250°C; Carrier gas: helium, constant flow; Linear velocity: 60cm/sec. @ 120°C; Oven temp.: 120°C to 200°C @45°C/min. to 230°C @15°C/min to 330°C (hold 2 min.) @ 30°C/min.; Det.: ECD @330°C

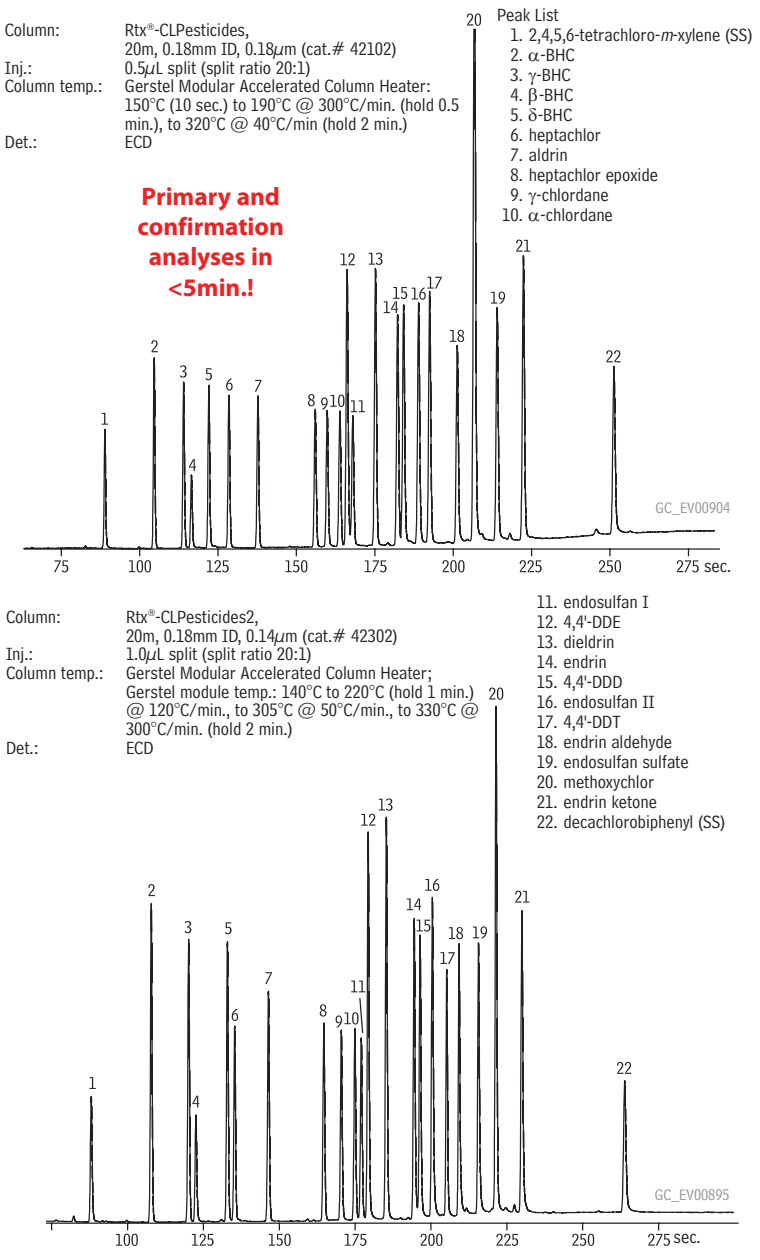
Figure 2 Two column modules in a Gerstel MACH column heating system.

The MACH system allows independent temperature programming of up to two columns, simultaneously.

thank **you**

Instrument provided courtesy of Gerstel USA.
www.gerstelus.com

Figure 3 Resolve organochlorine pesticides in less than 5 minutes using the Rtx®-CLPesticides columns and the MACH system.



Sample: Organochlorine Pesticide Mix AB #2 (8-80µg/mL each component in hexane/toluene 1:1, (cat.# 32292), Pesticide Surrogate Mix (200µg/mL each component in acetone, cat.# 32000)

Rtx®-CLPesticides Columns (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	0.18	-60 to 310/330°C	20-Meter	42102
0.53mm	0.50	-60 to 300/320°C	30-Meter	11140

Rtx®-CLPesticides2 Columns (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	0.14	-60 to 310/330°C	20-Meter	42302
0.53mm	0.42	-60 to 300/320°C	30-Meter	11340

Pesticide Surrogate Mix

decachlorobiphenyl 2,4,5,6-tetrachloro-*m*-xylene
200µg/mL each in acetone, 1mL/ampul
cat. # 32000

Organochlorine Pesticide Mix AB #2

(20 components)

aldrin	8µg/mL	dieldrin	16
α-BHC	8	endosulfan I	8
β-BHC	8	endosulfan II	16
δ-BHC	8	endosulfan sulfate	16
γ-BHC (lindane)	8	endrin	16
α-chlordane	8	endrin aldehyde	16
γ-chlordane	8	endrin ketone	16
4,4'-DDD	16	heptachlor	8
4,4'-DDE	16	heptachlor epoxide (B)	8
4,4'-DDT	16	methoxychlor	80

In hexane:toluene (1:1), 1mL/ampul
cat. # 32292

Resprep™ Florisil® SPE Cartridges: Normal Phase

	3mL/500mg (50-pk.)	6mL/500mg (30-pk.)	6mL/1000mg (30-pk.)
Florisil®	24031	—	24034
(EPA SW 846 methods and CLP protocols)	24032*	26086**	26085**

*Teflon® frits **Glass tubes with Teflon® frits

CarboPrep™ SPE Cartridges

	Tube Volume, Bed Weight	qty.	cat#
CarboPrep™ 90	3mL, 250mg	50-pk.	26091

get **connected**

See page 20-21 for our list of connectors and connector kits.

Resolving the Benzo(j)fluoranthene Challenge

Separate New PAHs Quickly Using the Rxi™-17 GC Column

By Robert Freeman, Environmental Innovations Chemist

- Fully resolve benzo(j)fluoranthene from benzo(b) & (k).
- Excellent resolution of 16 priority pollutant PAHs.
- Separate difficult dibenzo pyrene isomers.

New Compounds, New Challenges

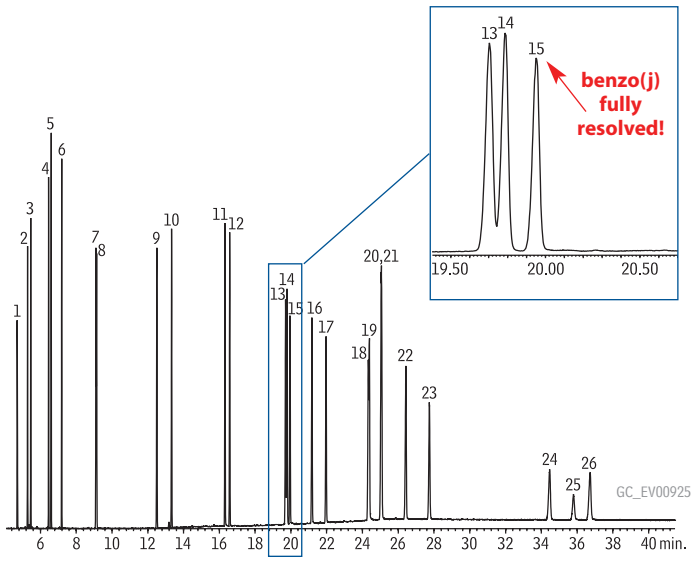
Polynuclear aromatic hydrocarbons (PAHs) are widespread organic pollutants that significantly affect environmental quality and raise human health concerns. The US EPA mandates testing of 16 priority PAH pollutants, while analyte lists in other countries are expanding to include compounds such as benzo(j)fluoranthene, dibenzo(a,h)-acridine, and dibenzo(a,e)pyrene, that are difficult to analyze under conventional test conditions. Benzo(j)fluoranthene and benzo(b)fluoranthene, for example, co-elute on a 5%diphenyl/95%dimethyl polysiloxane stationary phase. When reporting of individual concentrations for each isomer is required, conventional methods are not viable and new solutions must be found.

The Rxi™ Alternative

The Rxi™-17 column contains a 50% diphenyl/50% dimethyl polysiloxane stationary phase. The higher concentration of phenyl groups in this stationary phase increases retention of phenyl-containing compounds, such as PAHs, thus facilitating separation. We also used a Drilled Uniliner® inlet liner since it eliminates sample exposure to cold spots and potentially active metal components in the injection port. Using a pulsed splitless injection, we maximize sample transfer to the column while minimizing high molecular weight discrimination.

The data in Figure 1 demonstrate the excellent resolution of benzo(j)fluoranthene achievable on the Rxi™-17 column. Phenanthrene and anthracene also resolve well on this column under slower run conditions (data not shown). Using the Rxi™-17 column with an optimized temperature program is a practical solution to the challenges posed by expanding PAH analyte lists. If you are struggling to quantify PAHs on conventional columns, try the Rxi™-17 column and the optimized temperature program shown here.

Figure 1 Fast, effective separation of target PAHs using an Rxi™-17 column and an optimized temperature program.



Peak List	Ret. Time (min.)		Ret. Time (min.)
1. naphthalene	4.70	14. benzo(k)fluoranthene	19.78
2. 1-methylnaphthalene	5.28	15. benzo(j)fluoranthene	19.95
3. 2-methylnaphthalene	5.46	16. benzo(a)pyrene	21.17
4. acenaphthylene	6.45	17. 3-methylcholanthrene	21.97
5. acenaphthene	6.60	18. dibenzo(a,h)acridine	24.33
6. fluorene	7.18	19. dibenzo(a,j)acridine	24.39
7. phenanthrene	9.10	20. indeno(1,2,3-cd)pyrene	25.04
8. anthracene	9.14	21. dibenzo(a,h)anthracene	25.07
9. fluoranthene	12.50	22. benzo(ghi)perylene	26.43
10. pyrene	13.33	23. 7H-dibenzo(c,g)carbazole	27.75
11. benzo(a)anthracene	16.32	24. dibenzo(a,e)pyrene	34.46
12. chrysene	16.58	25. dibenzo(a,i)pyrene	35.80
13. benzo(b)fluoranthene	19.70	26. dibenzo(a,h)pyrene	36.73

Column: Rxi™-17, 30m, 0.25mm ID, 0.25µm (cat.# 13523)
Sample: PAH mix, 20µg/mL each component: EPA Method 610 Mix (cat.# 31011), PAH Supplement Mix (cat.# 31857), 1-methylnaphthalene (cat#31283), 2-methylnaphthalene (cat#31285); Inj.: 1.0µL pulsed splitless injection (20ng each component on column), 4mm Drilled Uniliner® inlet liner with hole at top (cat # 21055); pulse: 20psi @ 0.3 min., 40mL/min. @ 0.2 min. Inj. temp.: 300°C; Carrier gas: helium, constant flow; Flow rate: 1.2mL/min.; Oven temp.: 90°C (hold 1.0 min.) to 215°C @ 25°C/min. (hold 0.5 min.) to 235°C @ 4°C/min., to 280°C @ 15°C/min., to 320°C @ 4°C/min. (hold 20 min.); Det.: Agilent 5973 GC/MS; Scan range: 50-550 amu; Solvent delay: 4.0 min.; Tune: DFTPP; Ionization: EI

Rxi™-17 Columns (fused silica)

(Crossbond® 50% diphenyl / 50% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.25mm	0.25	40 to 280/300°C	30-Meter	13523

Direct Injection Liners for Agilent GCs

ID* x OD & Length (mm)	qty.	cat.#
Drilled Uniliner® (hole on top)		
4.0 ID x 6.3 OD x 78.5	5-pk.	21055

SV Calibration Mix #5 / 610 PAH Mix (16 components)

acenaphthene	benzo(k)fluoranthene	indeno(1,2,3-cd)pyrene
acenaphthylene	benzo(ghi)perylene	naphthalene
anthracene	chrysene	phenanthrene
benzo(a)anthracene	dibenzo(a,h)anthracene	pyrene
benzo(a)pyrene	fluoranthene	
benzo(b)fluoranthene	fluorene	

2,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31011

PAH Supplement Mix for Method 8100 (8 components)

benzo(j)fluoranthene	7H-dibenzo(c,g)carbazole	dibenzo(a,i)pyrene
dibenzo(a,h)acridine	dibenzo(a,e)pyrene	3-methylcholanthrene
dibenzo(a,j)acridine	dibenzo(a,h)pyrene	

1000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31857

Analysis of Nitrofurans in Honey

Using LC/MS/MS and an Ultra C18 Column

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

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

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

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

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

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

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

Acknowledgement

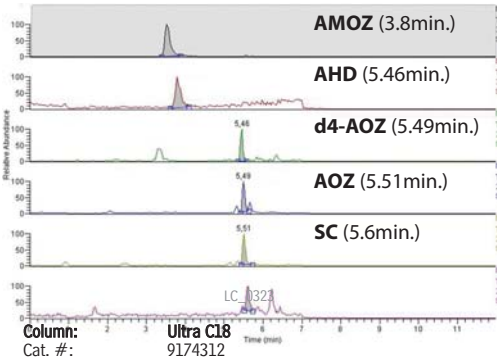
We are grateful to EIDOMET SRL, Restek distributor in Argentina, and application chemist Dr. Alejandro Albornoz, for the analytical work discussed in this article.

Ultra C18 HPLC Column

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

3µm Column, 2.1mm	cat. #
100mm	9174312

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



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

Conditions:
Mobile phase: A: 0.05% formic acid in methanol
B: 0.05% formic acid – 5 mM NH_4 formate in water

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

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

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

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

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

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

Why Derivatize?

Improve GC Separations with Derivatization

By Kristi Sellers, Innovations Chemist

- Get better separations with increased resolution and response.
- Learn how to choose proper reagents for desired reactions.

Many laboratories include derivatization as part of their sample preparation for gas chromatography (GC) analysis. So, what is derivatization? Why is it important and how do you choose a derivatizing reagent? The discussion below answers these questions. By choosing the right derivatization reagent and procedure you can increase resolution and analyte response, significantly improving your separations.

What is derivatization?

Derivatization is the process by which a compound is chemically changed, producing a new compound that has properties more amenable to a particular analytical method. Some samples analyzed by GC require derivatization in order to make them suitable for analysis. Compounds that have poor volatility, poor thermal stability, or that can be adsorbed in the injector will exhibit nonreproducible peak areas, heights, and shapes. Other compounds that respond poorly on a specific detector may need to be "tagged" with a different functional group to improve detection. For example, tagging with chlorine can improve response on an ECD (electron capture detector). In addition to improving suitability and response, derivatization can improve resolution between coeluting compounds and overlapping peaks.[1]

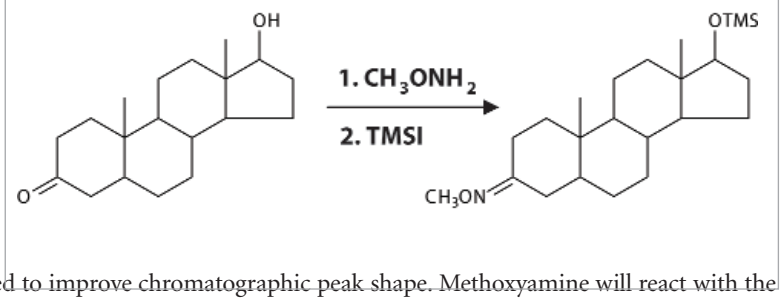
How do I choose a derivatizing reagent?

A good derivatizing reagent and procedure should produce the desired chemical modification of the compound(s) of interest, and be reproducible, efficient, and nonhazardous.[2] For GC, there are three basic types of derivatization reactions: silylation, acylation, and alkylation. Silylating reagents react with compounds containing active hydrogens; these reagents are the most common type used in GC. Acylating reagents react with highly polar functional groups such as amino acids or carbohydrates. Alkylating reagents target active hydrogens on amines and acidic hydroxyl groups.[3] Multiple derivatizing reagents may be necessary for compounds containing several different functional groups such as androsterone (Figure 1). In these multi-step derivatization procedures the use of other types of reagents, such as oxime, hydrazone, methylation, and cyclic derivatives, may be necessary.

A multi-step example

Derivatization can substantially improve chromatographic results, as seen in this example derivatization of androsterone (Figure 1). Androsterone contains a hydroxyl group and a carbonyl group and exhibits poor peak shape and poor separation if analyzed underivatized by GC (Figure 2b). Using silylation, active hydrogens on OH, SH, and NH groups can be replaced.[3] Since *n*-trimethylsilylimidazole (TMSI) is a strong silyl donor, it will react readily with the hydroxyl group on the androsterone molecule creating a trimethylsilyl (TMS) derivative. Because androsterone also contains a carbonyl group, another derivatizing reagent is needed to improve chromatographic peak shape. Methoxyamine will react with the carbonyl group forming an oxime derivative (CH_3ON). Oxime derivatives not only improve chromatographic performance, but also alter GC separations. Figure 2a shows the chromatographic result of derivatizing sex hormones using TMSI and methoxyamine; retention times are increased, separation is increased, and peak shapes and responses are improved.

Figure 1 Derivatization reaction of androsterone using TMSI/methoxyamine.



Conclusion

Derivatizing compounds for GC often is necessary to obtain reproducible chromatographic results. Eliminating this step to save time can be costly and produce inaccurate and unreliable results. A well-chosen derivatization procedure, based on the chemical composition of the target compounds, can significantly improve your chemical separations.

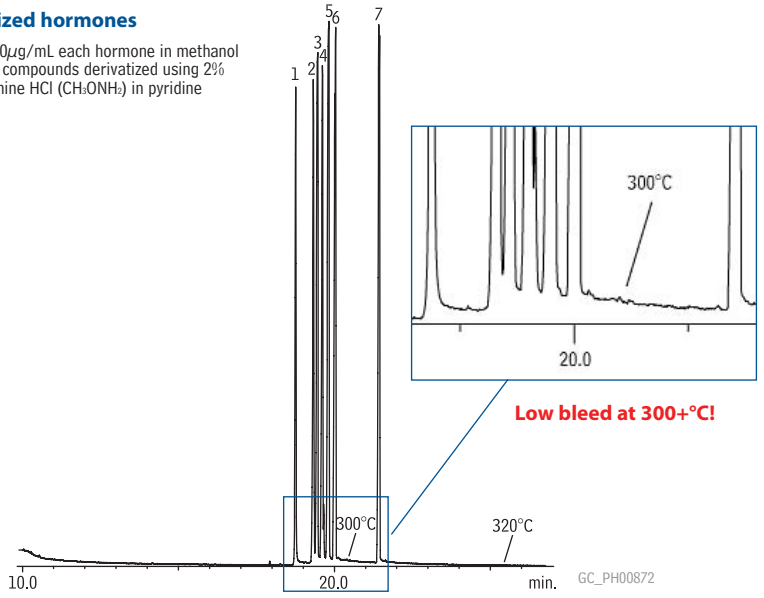
References

1 Knapp D., Handbook of Analytical Derivatization Reactions, Wiley-Interscience, 1979, pp.2-24, 449-453, 482.

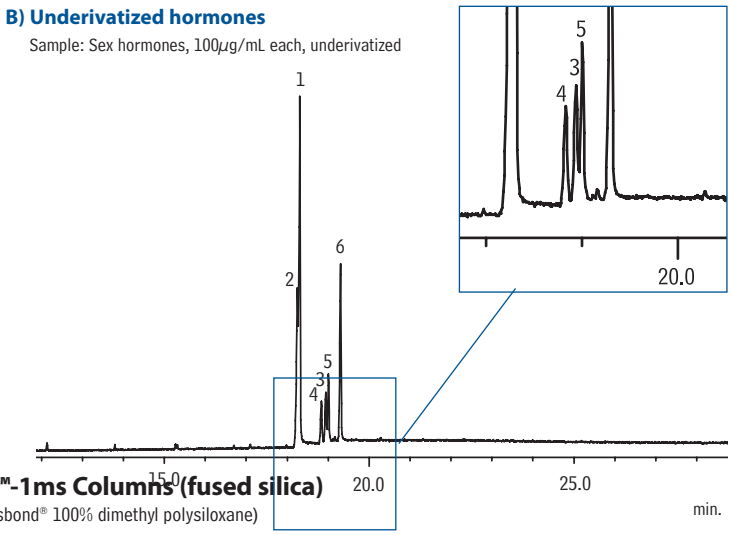
2 www.piercenet.com

3 Grob R., Barry E., Modern Practice of Gas Chromatography, Wiley-Interscience, 2004, pp. 817-818.

Figure 2 Derivatized hormones show excellent resolution and more symmetrical peak shapes than underivatized hormones.



For the derivatization procedure used in this analysis, see Knapp's *Handbook of Analytical Derivatization Reactions*, page 482.



Column:	Rxi™-1ms 30m, 0.25mm ID, 0.25µm (cat. # 13323)
Inj.:	1.0µL splitless (hold 0.5min.), 3.5mm single gooseneck inlet liner (cat.# 20961)
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1mL/min.
Oven temp.:	100°C to 320°C @ 10°C/min. (hold for 10 min.)
Det.:	MS: Shimadzu 17A with QP5000
Transfer line temp.:	280°C
Scan range:	40-700amu
Ionization:	EI
Mode:	Scan

Rxi™-1ms Columns (fused silica)
(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.25mm	0.25	-60 to 330/350°C	30-Meter	13323

Splitless Liners for Shimadzu GCs

**Nominal ID at syringe needle expulsion point.

Superior Fractionation of Extractable Petroleum Hydrocarbons

Get More Accurate Results Using Restek SPE Tubes

By Lydia Nolan, Innovations Chemist

- Easier quantitation; lower background & less interference.
- Reliable, reproducible results.
- Unique packaging designed for convenience and storage stability.

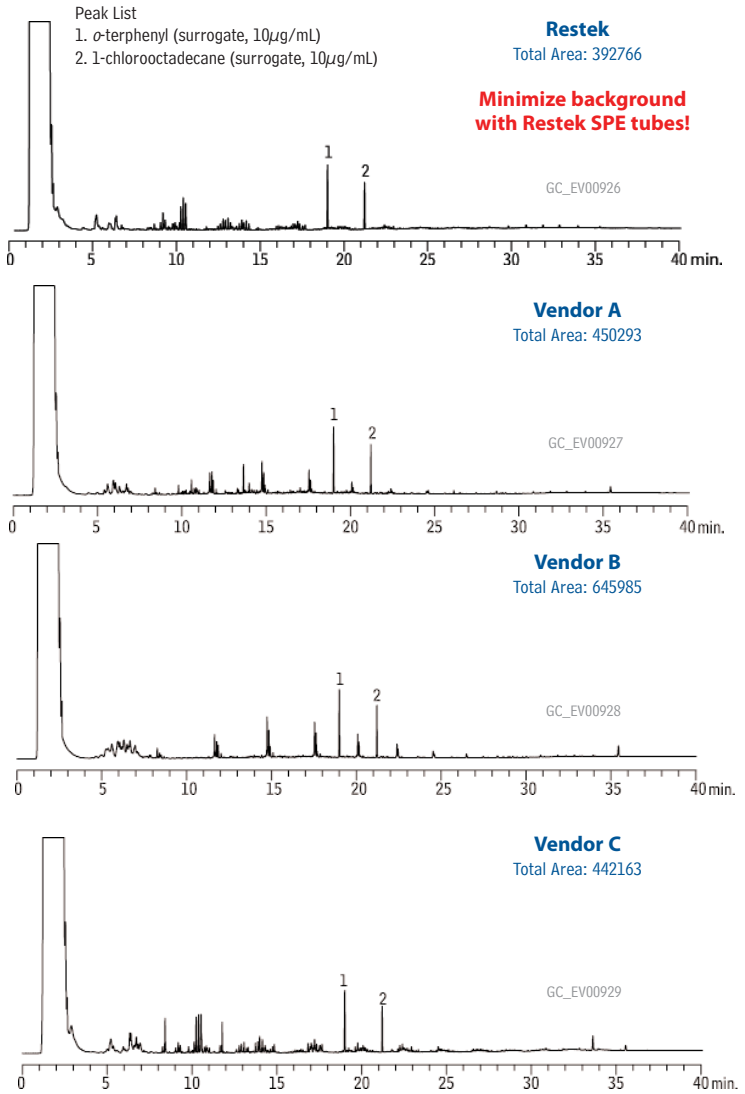
There is an increasing public awareness of the threat to public health from leaking underground storage tanks. Both federal and state agencies have developed methods to address the testing of potential problem sites. The Massachusetts Department of Environmental Protection's "Method for the Determination of Extractable Petroleum Hydrocarbons (EPH)" has recently been updated and is based on solvent extraction of water and soil/sediment matrices, followed by silica gel SPE fractionation of aliphatics and aromatics from C9 through C36 hydrocarbon ranges.

The quality and conformation of the silica SPE clean-up column is essential to acceptable fractionation and recovery results. Commercial silica SPE products streamline this process, but it is important to understand the quality and performance differences among the available products, and the impact they have on your results. The activity level and capacity of the silica, the compression of the bed, and the quality of the constituents and packaging are all critical to getting accurate and reliable results. The data in Table I show how even very minor amounts of excess moisture (known amounts added for experimental purposes during the first conditioning step) or long-term storage without desiccation can produce early breakthrough of the sensitive analytes from the aromatic fraction into the aliphatic fraction.

To ensure maximum shelf-life and minimum environmental exposure after opening these cartridges, Restek packages them into 5 smaller packs of 4 SPE tubes each—the fewest cartridges per pack available. We also provide an additional outer, resealable barrier bag, making successful short- and long-term product storage easier for the user.

Activity level of the silica and consistency of the cartridge packing are essential for reliable fractionation recovery and reproducibility. The recovery and reproducibility of results for the fractionation surrogates (2-fluorobiphenyl, 2-bromonaphthalene and naphthalene) are critical to determining if breakthrough is occurring. Again, in comparing several commercial sources, using optimized conditions for each vendor, results show that the Restek Massachusetts EPH cartridges are capable of quantitative (greater than 97%) and reliable (RSDs less than 7.3) recoveries for these critical markers (Table II).

Figure 1 Restek Massachusetts EPH SPE tubes show the lowest overall level of background response.



Column: Rtx®-5, 30m, 0.32mm ID, 0.25µm (cat.# 10224)
Sample: Massachusetts EPH Surrogate Spike Mix (4000µg/mL each component) diluted to 400µg/mL in hexane.
Spiking standard: 50µL Inj.: 0.5µL, splitless (hold 0.5 min.), PSS split/splitless inlet liner (cat.#21717)
Inj. temp.: 280°C; Carrier gas: helium, constant velocity; Pressure pulse program: 50cm/sec @ -0.71 min., split ratio = 0 @ -0.70 min., split ratio = 20:1 @ 0.75 min., 35cm/sec @ 0.8 min.; Linear velocity: 35cm/sec
Oven temp.: 60°C (hold 1 min.) to 310°C @ 8°C/min. (hold 12 min.), Det.: FID @ 330°C

* Total area counts exclude response for solvent front and surrogate peaks
1. o-terphenyl (surrogate) 10ng on-column
2. 1-chlorooctadecane (surrogate) 10ng on-column
All cartridges were extracted with 15mL hexane, without prior conditioning. Extract blanks were then spiked with the 50µL of MA EPH Surrogate Spike mix, cat#31479 (diluted to 400µg/mL with hexane), and dried down to 1mL with gentle nitrogen purge.

Table I Excess moisture and improper storage compromise results by causing breakthrough into the aromatic fraction.

	% Breakthrough into Hexane (Aliphatic) Fraction			
Analyte	Package intact, no added moisture	200µL water added	Package opened, resealed, stored on shelf, 1 year	Package intact, stored on shelf, 1 year
Naphthalene	0.0	0.0	----	----
2-fluorobiphenyl (surrogate)	0.0	0.0	----	----
2-bromonaphthalene (surrogate)	0.0	4.4	33.3	28.5

Table II Restek Massachusetts EPH SPE tubes provide more accurate and reproducible results for critical marker compounds.

	Restek			Vendor A			Vendor B			Vendor C		
Analyte	percent recovery	STD	%RSD	percent recovery	STD	%RSD	percent recovery	STD	%RSD	percent recovery	STD	%RSD
naphthalene	103.1	7.5	7.2	101.2	10.1	10.0	88.8	2.8	3.1	66.5	2.6	3.9
2-fluorobiphenyl	97.8	6.6	6.7	100.4	13.7	13.6	99.3	5.0	5.0	104.2	6.6	6.4
2-bromonaphthalene	98.6	5.3	5.4	71	7.1	10.0	50.0	8.1	16.1	29.2	1.9	6.6

All tubes were 20 or 25mL with approximately 5g silica packing. Conditioning: 15mL hexane. Sample: 0.5mL of each fractionation check standard and surrogate standard. Elution for fraction #1 (aliphatics): 17-20mL hexane (volume was optimized for each supplier and lot of tubes). Elution for fraction #2 (aromatics): 20mL of CH_2Cl_2. Each fraction was dried to a total volume of 1mL and analyzed by GC.[1]

MA Fractionation Check Mix (31 components)

PAHs:	Hydrocarbons:
acenaphthene	*n*-nonane (C9)
acenaphthylene	*n*-decane (C10)
anthracene	*n*-dodecane (C12)
benzo(a)anthracene	*n*-tetradecane (C14)
benzo(a)pyrene	*n*-hexadecane (C16)
benzo(b)fluoranthene	*n*-octadecane (C18)
benzo(k)fluoranthene	*n*-nonadecane (C19)
benzo(ghi)perylene	*n*-eicosane (C20)
chrysene	*n*-docosane (C22)
dibenzo(a,h)anthracene	*n*-tetracosane (C24)
fluoranthene	*n*-hexacosane (C26)
fluorene	*n*-octacosane (C28)
indeno(1,2,3-cd)pyrene	*n*-triacontane (C30)
2-methylnaphthalene	*n*-hexatriacontane (C36)
naphthalene	
phenanthrene	
pyrene	

25µg/mL each in hexane, 1mL/ampul
cat. # 31481

MA Fractionation Surrogate Spike Mix

2-bromonaphthalene 2-fluorobiphenyl
4,000µg/mL each in hexane, 1mL/ampul
cat. # 31480

MA EPH Surrogate Spike Mix

1-chlorooctadecane *o*-terphenyl
4,000µg/mL each in acetone, 1mL/ampul
cat. # 31479

Method Specific SPE Cartridges: Massachusetts EPH

Tube Volume, Bed Weight	qty.	cat.#
20mL, 5g	20-pk.	26065

Rtx®-5 Columns (fused silica)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	0.25	-60 to 330/350°C	30-Meter	10224

Splitless Liners for PerkinElmer GCs

ID* x OD & Length (mm)	qty.	cat.#

Coextractables are another major concern with commercial cartridges. The contaminants may be found in the packaging, cartridge materials such as the SPE tube and frits, and the silica itself. The solvent blank extractions shown in Figure 1 were collected from cartridges that were not pre-conditioned. Restek cartridges show the lowest level of background peak area counts, indicating the lowest level of background extractables.

When cartridges start out with low levels of extractables, it may not be necessary to use the methylene chloride pre-treatment allowed in the method. This pre-treatment can easily compromise the fractionation performance of the cartridge beds and should be avoided whenever possible. In addition, fewer product-related contaminants will provide clearer quantitation and require fewer manual reviews of the data generated from the final chromatograms.

In all of the key performance areas, the Restek Massachusetts EPH SPE tubes outperformed other commercially available products. Our cartridges are designed to deliver accurate, reliable, and reproducible results. For high quality separation products developed to prevent breakthrough and minimize background, reach for Restek sample preparation products.

References

1 *Method for the Determination of Extractable Petroleum Hydrocarbons (EPH).* Massachusetts Department of Environmental Protection, Division of Environmental Analysis, Office of Research and Standards, Bureau of Waste Site Cleanup, Revision 1.1, May 2004.

for **more** info

For more information on our selection of SPE tubes, visit us online at **www.restek.com**

Prevent Mercury Loss During Transport and Storage

Use Siltek® Surface Treatment on Steel Components

By Gary Barone, Restek Performance Coatings Division

- Rugged—withstands temperatures up to 400°C.
- Meets system inertness requirements.
- Eliminates costly retests.

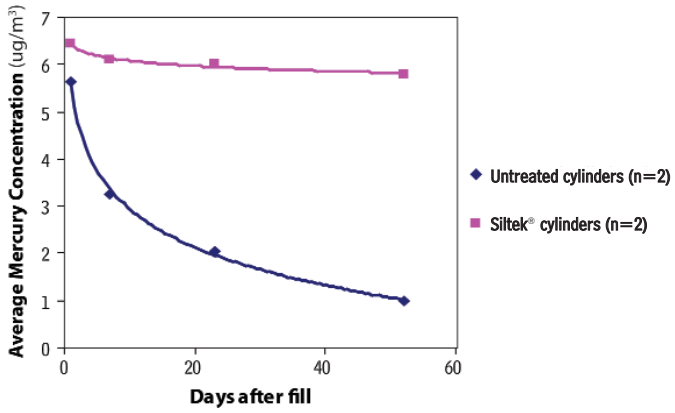
As concerns grow over mercury in the environment, new regulations have been developed to measure, and eventually reduce, mercury emissions from coal-fired electric utilities. For example, the US EPA will require all electric utilities to measure mercury emissions starting on January 1, 2009. The most popular methods of sampling will be based on continuous mercury monitoring systems (CMMS) and sorbent tube samplers. To ensure quantitative storage and transfer, and accurate analysis, of the low levels of mercury in streams sampled from flue stacks, these sampling systems must be inert.

Siltek® surface treatment has been used in a wide variety of applications in which an inert surface is of paramount importance. To measure the impact of Siltek® treatment on adsorption of mercury during storage, we compared the performances of 304 grade stainless steel gas sampling cylinders (Swagelok®, Solon OH) with and without Siltek® treatment.

We filled each cylinder with 8µg/m³ of elemental mercury (approximately 1 part per billion) (Spectra Gases, Alpha NJ) and assessed the mercury concentration in each cylinder over time to determine changes in mercury concentration. Detection was achieved by direct interface gas sampling to an atomic adsorption detector. Sample pathway regulator and tubing were Siltek® treated to ensure accurate transfer.

The data in Figure 1 demonstrate that Siltek® treatment provides a stable surface for elemental mercury, and untreated stainless steel does not. Based on these results, we conclude that Siltek® surface treatment for steel or stainless steel components and tubing in CMMS and sorbent tube mercury sampling systems will improve analytical reliability. For more information about Siltek® surface treatment, visit us at: **www.restekcoatings.com**

Figure 1 Siltek® treated gas sampling cylinders show very good inertness toward mercury.



Siltek®/Sulfinert® Treated Coiled Electropolished 316L Grade Stainless Steel Tubing

ID	OD	cat.#	5-24 ft.	25-99 ft.	100-299 ft.	>300 ft.
0.085" (2.16mm)	1/8" (3.18mm)*	22538				
0.180" (4.57mm)	1/4" (6.35mm)**	22539				

1/8" OD: 5 ft. to 100 ft. in one continuous coil; 1/4" OD: 5 ft. to 300 ft. in one continuous coil. Longer lengths will be more than one coil. Note: required length in meters x 3.2808 = length in feet.

Siltek®/Sulfinert® Treated Coiled 316L Grade Stainless Steel Tubing

ID	OD	cat.#	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
0.055" (1.40mm)	1/8" (3.18mm)**	22508				
0.180" (4.57mm)	1/4" (6.35mm)**	22509				
0.277" (7.04mm)	3/8" (9.52mm)***	22914				

Siltek®/Sulfinert® Treated Straight Seamless 316L Grade Stainless Steel Tubing

6 foot Length

ID	OD	qty.	cat.#
0.055" (1.40mm)	1/8" (3.18mm)**	ea.	22901
0.180" (4.57mm)	1/4" (6.35mm)**	ea.	22902
0.277" (7.04mm)	3/8" (9.52mm)***	ea.	22903

*0.020" wall thickness **0.035" wall thickness ***0.049" wall thickness

Sulfinert® Treated Alta-Robbins Sample Cylinder Valves

Description	qty.	cat.#
1/4" NPT Exit	ea.	21400
1/4" Compression Exit	ea.	21401
1/4" NPT with Dip Tube*	ea.	21402
1/4" NPT with 2850psi Rupture Disc	ea.	21403
1/4" NPT Male Inlet x 1/4" Female Outlet with 2850psi Rupture Disc	ea.	21404

Specify dip tube length or % outage when ordering (maximum length = 5.25"/ 13.3cm)

United States patent 6,444,326 (Siltek®/Sulfinert®)

thank **you**

Ted Neeme and Steve Mandel from Spectra Gases for their contributions to this work.

Sulfinert® Treated Swagelok® Sample Cylinders

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

Protect Sample Integrity and Prolong Sampling System Lifetime

Using Hydroguard™ Deactivated/Silcosteel® Treated Tubing

By Gary Barone, Restek Performance Coatings Division

- Prevents adsorption of sample components to an active surface.
- Long-lasting water resistance, increases instrument up-time.
- Specifically designed and tested for deactivating purge and trap or headspace systems.

Current regulations for drinking water and waste water require quantifying contaminant component concentrations at parts-per-trillion levels. As the demands of analytical methods and the sensitivity of analytical instruments advance, so has the need for improved inertness of the components of the sample pathway. In analyses at parts-per-trillion concentrations, any surface activity in the transfer system can adsorb significant amounts of active analytes and greatly impact the reliability of the data. Furthermore, components of purge and trap or headspace systems often are in contact with steam, which can create activity very quickly—even in coated system components. To address this need, we have created a superior surface for the tubing in purge and trap or headspace systems: Hydroguard™ deactivated/Silcosteel® treated stainless steel tubing.

For more than a decade, Restek's proprietary Silcosteel® and Siltek®/Sulfinert® treatments† have been ideal solutions for creating inert stainless steel pathways. Now, we have developed and rigorously tested Hydroguard™ deactivated/Silcosteel® treated stainless steel tubing specifically to meet the demanding requirements and environments of purge and trap and headspace systems.

Hydroguard™ deactivated/Silcosteel® treated tubing is preferred for situations in which water vaporization is encountered, as in purge and trap systems. Unique deactivation chemistry creates a high-density surface that is not readily attacked by hydrolysis. High-density Hydroguard™ deactivation at the outer surface effectively prevents water vapor from contacting the Silcosteel® treated stainless steel surface below. Thus, an inert surface is maintained in the face of highly aggressive conditions, and active analytes pass through the tubing without adsorbing to the surface.

Regardless of your application, we highly recommend Hydroguard™ deactivated/Silcosteel® treated tubing to improve analytical reliability from your purge and trap or headspace system.

† United States patents 6,511,760 (Silcosteel®) and 6,444,326 (Siltek®/Sulfinert®).

Silcosteel® Treated Hydroguard™ Deactivated Electropolished 316L Grade Stainless Steel Tubing

ID	OD	cat.#	5-24 ft.	25-99 ft.	100-299 ft.	>300 ft.
0.085" (2.16mm)	1/8" (3.18mm)*	22489				
0.180" (4.57mm)	1/4" (6.35mm)**	22488				

Silcosteel® Treated Hydroguard™ Deactivated Seamless 316L Grade Stainless Steel Tubing

ID	OD	cat.#	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
0.055" (1.40mm)	1/8" (3.18mm)**	22491				
0.180" (4.57mm)	1/4" (6.35mm)**	22490				

Silcosteel® Treated Hydroguard™ Deactivated 304 Grade Stainless Steel Tubing

ID	OD	cat.#	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
0.010" (0.25mm)	1/16" (1.59mm)	22497				
0.020" (0.51mm)	1/16" (1.59mm)	22496				
0.030" (0.76mm)	1/16" (1.59mm)	22495				
0.040" (1.02mm)	1/16" (1.59mm)	22494				
0.085" (2.16mm)	1/8" (3.18mm)*	22493				
0.210" (5.33mm)	1/4" (6.35mm)*	22492				

*0.020" wall thickness
**0.035" wall thickness

Hub-Cap Mobile Phase Accessories

Simplify Mobile Phase Delivery with the Hub-Cap Filter Kit

Introducing our new Hub Cap filter kit! The Hub-Cap filter allows you to simultaneously transfer and filter your mobile phases. The bottle tops and adaptors are designed to fit securely on 4-liter solvent bottles and eliminate messy, loose-fitting parafilm or foil wraps. Tidy up your mobile phase delivery—try a Hub-Cap today!

Keep your mobile phase lines under control—use Hub-Cap bottle tops instead of parafilm, aluminum foil, or tape on your mobile phase reservoirs.

Hub-Cap Filter Kit

new!

cat. #26395

Transfer and filter mobile phases in one easy step.
Kit includes: bottle adapter, bottle adapter nut, filter inlet cap, grid support, vacuum hose barb, tube compression fitting, 47mm grid, 47mm .22µm filter membrane, 47mm .45µm filter membrane, 1/4" OD x 1/8" ID ultra chemical resistant, Teflon® FEP lined Tygon® tubing (3'), 6" x 6" box with shrink wrap insert

Description	qty.	cat.#
Hub-Cap Filter Kit	kit	26395
Replacement Membrane Filters	**qty.**	**cat.#**
Polyproylene Membrane Filters, 47mm, 0.45µm	100-pk.	26396
Polyproylene Membrane Filters, 47mm, 0.22µm	100-pk.	26397
Nylon Membrane Filters, 47mm, 0.45µm	100-pk.	26398
Nylon Membrane Filters, 47mm, 0.22µm	100-pk.	26399

Hub-Cap 4 Liter Bottle Tops

cat. #26541

Most bottles use a GL45 cap. New Hub-Cap bottle tops are a great way to neatly keep your mobile phase lines where they belong. Use instead of parafilm, aluminum foil, or tape on your mobile phase reservoirs.

Description	qty.	cat.#
Hub-Cap (assembly of the bottle cap and plug)	kit	26541
Hub-Cap Multi-pack	3-pk.	26542

Hub-Cap Adapters

cat. #26538

cat. #26540

Allow the use of the Opti-Cap™ with 4-liter solvent bottles.

Description	qty.	cat.#
Hub-Cap Adapter	ea.	26538
Hub-Cap Adapter Multi-pack	3-pk.	26539
Hub-Cap Adapter and Opti-Cap™	kit	26540

Cool Tools!

Restek Innovations Save You Time and Money

A Clean Square Cut...

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

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

Scoring Wafer with Handle

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

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

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

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

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

Ceramic Scoring Wafers

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

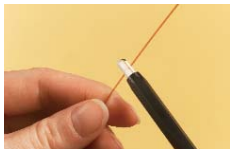
Exert just enough pressure to put a slight arc in the tubing. The tubing should fall off or break with a slight tap of the wafer.

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

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

Sapphire Scribe

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



One quick stroke...

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

Description	qty.	cat.#
Sapphire Scribe	ea.	20182

Capillary Column Caps

- Attach to the column in seconds to form an airtight seal.
- Increase column lifetime—prevent moisture and air from entering the column during storage.
- Color-coded for identifying detector and injector ends.
- Not recommended for reuse.

Description	qty.	cat.#
Capillary Column Caps	10-pk.	21044

Peak Performers

Routine Connections Made Simple

By Donna Lidgett, GC Accessories Product Marketing Manager

restek
innovation!

SeCure™ "Y" Connector Kits

- Connect two analytical columns to a transfer line or guard column.
- Use standard "Y" Press-Tight® connectors and 1/16" graphite ferrules.
- Reliable seal integrity, will not unexpectedly disconnect during temperature-programmed analyses.
- Open design allows visual confirmation of the seal for added confidence in the connection.

Combine the simplicity of a "Y" Press-Tight® connector with the strength of a metal union. The ferrules and knurled nuts hold the fused silica tubing in place, which prevents the tubing from unexpectedly disconnecting, even at temperatures as high as 400°C.

Kits include: SeCure™ "Y" connector body, 3 knurled nuts, "Y" Universal Press-Tight® union, 3 ferrules.

Description	Ferrules Fit Column ID	qty.	cat.#
SeCure™ "Y" Connector Kit	0.18/0.25/0.28mm	kit	20276
SeCure™ "Y" Connector Kit	0.32mm	kit	20277
SeCure™ "Y" Connector Kit	0.45/0.53mm	kit	20278
Knurled nut		3-pk.	20279

Make secure, reliable column-to-column connections with SeCure™ "Y" connectors.

Graphite Ferrules for SeCure™ "Y" Connectors

- Preconditioned to minimize out-gassing.
- High-purity, high-density graphite.
- Stable to 450°C.
- No binders that can off-gas or adsorb analytes.
- Smooth surface and clean edges.

Ferrule ID	Fits Column ID	Graphite 10-pk.	Graphite 50-pk.
0.4mm	0.18/0.25/0.28mm	20200	20227
0.5mm	0.32mm	20201	20228
0.8mm	0.45/0.53mm	20202	20224

- Connect a guard column to an analytical column.
- Connect a column to a transfer line.
- Connect two columns in series.
- Repair a broken column.

Kits include: Vu2 Union™ body, 2 knurled nuts, 2 Press-Tight® unions, and 4 ferrules

Description	Ferrules Fit Column ID	qty.	cat.#
Vu2 Union™ Connector Kit	0.10/0.15mm	kit	22220
Vu2 Union™ Connector Kit	0.18/0.28mm	kit	21105
Vu2 Union™ Connector Kit	0.32mm	kit	21106
Vu2 Union™ Connector Kit	0.45/0.53mm	kit	21107
Knurled nut		2-pk.	21108

NOTE: Not recommended for GC column-to-MS connections—use the Vacuum Vu-Union® available at www.restek.com.

The Vu2 Union™ connector open design allows visual confirmation of the seal; secondary seals ensure a leak-tight connection.

Graphite Ferrules for Vu2 Union™ Connectors

- High-purity, high-density graphite.
- Stable to 450°C.
- No binders that can off-gas or adsorb analytes.
- Smooth surface and clean edges.

Ferrule ID	Fits Column ID	Graphite 2-pk.	Graphite 10-pk.
0.3mm	0.10/0.15mm	22221	22222
0.4mm	0.18/0.28mm	20280	20281
0.5mm	0.32mm	20282	20283
0.8mm	0.45/0.53mm	20284	20285

Universal Press-Tight® Connectors

- Connect a guard column to an analytical column.
- Repair a broken column.
- Connect a column outlet to a transfer line.
- Deactivated Press-Tight® connectors assure better recovery of polar and non-polar compounds.
- Siltek® treated connectors are ideal for organochlorine pesticides analysis.
- Fit column ODs from 0.33–0.74mm (Restek 0.1mm–0.53mm ID).

Description	5-pk.	25-pk.	100-pk.
Universal Press-Tight® Connectors	20400	20401	20402
Deactivated, Universal Press-Tight® Connectors	20429	20430	20431
Siltek® Treated Universal Press-Tight® Connectors	20480	20449	20481

Universal Angled Press-Tight® Connectors

- Angle approximates the curvature of a capillary column, reduces strain on column-end connections.

Description	5-pk.	25-pk.	100-pk.
Universal Angled Press-Tight® Connectors	20446	20447	20448
Deactivated Universal Angled Press-Tight® Connectors	20446-261	20447-261	20448-261
Siltek® Treated Universal Angled Press-Tight® Connectors	20482	20483	20484

Universal "Y" Press-Tight® Connectors

- Split sample flow onto two columns.
- Split a single column flow to two detectors—perform confirmation analysis with a single injection.
- Deactivated Press-Tight® connectors assure better recovery of polar and non-polar compounds.
- Siltek® treated connectors are ideal for organochlorine pesticides analysis.
- Fit column ODs from 0.33–0.74mm (Restek 0.1mm–0.53mm ID).

An alternative method of performing dual-column confirmational analyses!

Description	ea.	3-pk.
Universal "Y" Press-Tight® Connector	20405	20406
Deactivated Universal "Y" Press-Tight® Connector	20405-261	20406-261
Siltek® Treated Universal "Y" Press-Tight® Connector	20485	20486

Universal Angled "Y" Press-Tight® Connectors

- Inlet and outlet ends conform to the column curvature—alleviates column-end connection strain.

Description	ea.	3-pk.
Universal Angled "Y" Press-Tight® Connector	20403	20404
Deactivated Universal Angled "Y" Press-Tight® Connector	20403-261	20404-261
Siltek® Treated Universal Angled "Y" Press-Tight® Connector	20487	20469

MXT™-Union Connector Kits for Fused Silica Columns

- Low-dead-volume, leak-tight connection.
- Reusable.
- Siltek® treatment ensures maximum inertness.
- Ideal for connecting a guard column or transfer line to an analytical column.
- Use to oven temperatures of 350°C.
- Available in union and "Y" configurations.

SILTEK® treated

These MXT™ connectors can be used with fused silica tubing, because of a Valcon polyimide 1/32-inch one-piece fused silica adaptor. This unique graphite-reinforced composite allows a capillary column to slide into the adaptor and be locked in place simply by loosening and tightening the fitting. Each kit contains the MXT™ union, two 1/32-inch nuts and two one-piece fused silica adaptors.

MXT™-Union Connector Kits for Fused Silica Columns

Description	qty.	cat.#
For 0.25mm ID Fused Silica Columns	kit	21386
For 0.32mm ID Fused Silica Columns	kit	21385
For 0.53mm ID Fused Silica Columns	kit	21384

MXT™ "Y"-Union Connector Kits for Fused Silica Columns

Description	qty.	cat.#
For 0.25mm ID Fused Silica Columns	kit	21389
For 0.32mm ID Fused Silica Columns	kit	21388
For 0.53mm ID Fused Silica Columns	kit	21387

Get Connected!

By Al Carusone, Technical Service

Have you ever had to connect a GC analytical column to a guard column or transfer line? Or repair a broken column? How about connecting two columns in series or performing confirmation analysis with a single injection? All of these connections are possible with Restek's extensive selection of GC connectors. In most situations, connector choice is a personal preference and Restek offers several options. Here we review differences among our connectors and answer some frequently asked questions about our popular Press-Tight® connectors.

The **Press-Tight® connector**, a glass connector with a tapered internal diameter at each end, is the quickest and least expensive option. Straight or angled Press-Tight® connectors are effective for fused silica-to-fused silica connections for standard applications at temperatures below 325°C. The resulting connections are inert and have low dead volume.

The **MXT™-Union connectors** are unbreakable metal connectors that are reusable and ensure a low dead volume. They are designed for metal-to-metal connections, but also can make metal-to-fused silica unions using a Valcon polyimide adaptor. This unique graphite-reinforced composite allows a capillary column to slide into the adaptor and be locked in place simply by loosening and tightening the nuts.

If you require a fused silica-to-fused silica connector for high temperature applications, try Restek's **Vu2 Union™ connector** or **SeCure™ "Y" connector**. They combine the simplicity of a glass connector with the strength of a metal connector. Both connectors feature an open design that allows visual confirmation of the seal, and also have secondary seals to help maintain a leak-tight connection. These ultra-strong connections will not disconnect unexpectedly under temperature changes, vibrations, or other stresses normally encountered in GC analysis.

Restek also offers a **Vacuum Vu-Union® connector** for connecting a fused silica column to a mass spec transfer line. The Vacuum Vu-Union® connector utilizes Vespel® ferrules for nonpermeable vacuum connections. A specifically designed Vu-Union® glass insert permits more torque to be applied to the ferrules without fear of cracking the insert. As with the Vu2 Union™, you can confirm the seal through the window of the connector.

get the connection

see page 20-21 for a sampling of our connectors, or visit us online at www.restek.com

What is the difference between angled and regular Press-Tight® connectors?

The only difference between these connectors is their shape. A Press-Tight® connector is a straight tube; an angled Press-Tight® connector has a slight angle in the middle which reduces the strain on the fused silica tubing. This is of particular use in making a connection in a broken column, when you must make the connection within the column coils.

How can I obtain a leak-tight seal using a Press-Tight® connector?

Press-Tight® connectors are easy to use, but if they are not properly sealed, they can loosen due to thermal expansion during temperature-programmed runs. The keys to successful sealing are: 1) making a clean, square cut on the column and 2) moistening the end of the column with methanol before seating it into the connector. A small amount of polyimide resin also helps prevent the seal from separating during temperature cycling.

A brown ring indicates a proper seal.

Can Press-Tight® connectors be used with MXT® columns?

No. To achieve a leak-tight metal-to-metal connection, we recommend the **MXT™ Low Dead Volume connector** for metal columns. These low dead volume connectors are Siltek® treated to make them inert to active compounds, and they can be used up to 400°C without degrading the deactivation layer. MXT™ tubing can even be connected to fused silica tubing using an MXT™ connector with a Valcon Polyimide ferrule instead of a stainless steel ferrule.

Retention Cross-over Phenomenon in Gas Chromatography—Can the Mystery be Revealed? Part 2

Continued from page 2

mately 250 compounds were measured on a squalane stationary phase at four temperatures.[1] From these data one can identify numerous reversals in elution order of aliphatic and cyclic hydrocarbons. The solute interactions with a squalane stationary phase, the most nonpolar stationary phase one can use, are largely a result of dispersion interactions. The authors stated that the magnitude of temperature variation is a function of the size of the molecule expressed by the cross-sectional area of the molecules, which should also prove my point in my next discussion over coffee with my former student.

Finally, coming back to our first example in Part 1, both components not only show different functional groups, they also differ in their carbon skeleton (Figure 3). Linalool is an aliphatic alcohol and camphor is a bi-cyclic ketone, which means that not only the functional groups but also the difference in molecular geometry will contribute to the cross-over phenomenon.

What can we learn from this discussion? Peak overlapping and cross-over in peak elution order caused by variation of column temperature or temperature programming rate can occur not only on polar stationary phases for compounds with different functional groups but also on nonpolar or weak polar stationary phases for compounds that differ in their carbon skeleton. The analyst should, therefore, carefully examine the structure of the compounds to be separated if the information is available. Furthermore, it is recommended to study analyte retention carefully at various temperatures for difficult separations as an important aspect of method optimization.

References:
1 Hively, R.A. and R.E. Hinton, J. Gas Chromatogr. 6 (1968) 203 – 217.

Figure 2 Retention indices on squalane (IS) as a function of Tc for isothermal GC at 27°C, 49°C, 67°C, and 86°C.[1]

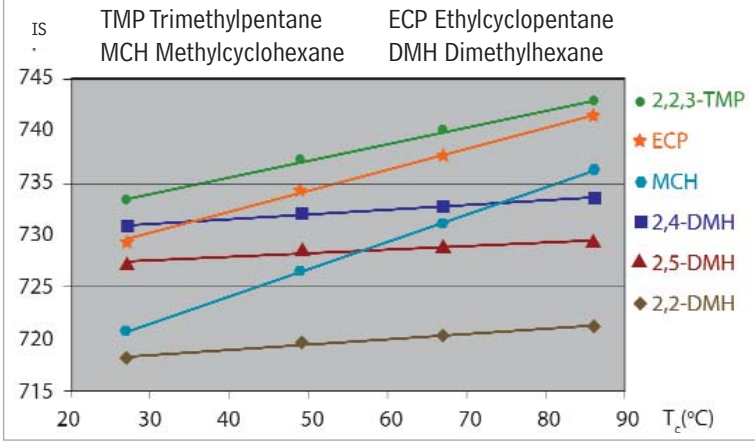
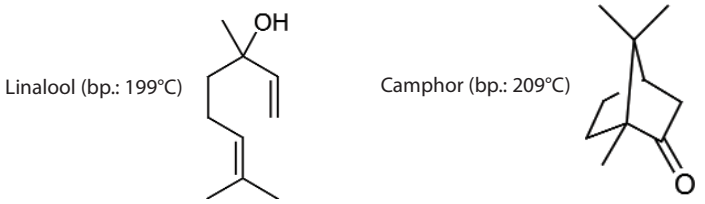


Figure 3 Functional groups influence elution order.



Tradeshow Schedule

We'd be happy to talk with you at any of the following meetings or shows. We'll post our booth numbers as they become available to us.

September, 2007

Date September 2-7
Show Dioxin 2007
Location Hotel Okura, Tokyo

Date September 13
Show New Jersey Mass Spectrometry Discussion Group Annual Vendor Show
Location DoubleTree Hotel, Somerset, NJ

Date September 16-20
Show AOAC International 121st Annual Meeting & Expo
Location Hyatt Regency Orange County, Anaheim, CA

Date September 25-28
Show Midwestern Association of Forensic Scientists (MAFS)
Location Park Place Hotel, Traverse City, MI

Date September 26-28
Show Vapor Intrusion Conference
Location Providence, RI

October, 2007

Date October 2-4
Show ISA Expo 2007
Location Reliant Center, Houston, TX

Date October 10-12
Show ACIL National Meeting
Location InterContinental Hotel Buckhead, Atlanta, GA

Date October 13-20
Show Society of Forensic Toxicology (SOFT)
Location Chapel Hill, NC

Date October 16-17
Show Gulf Coast Conference
Location Moody Garden Convention Center, Galveston, TX

Date October 18-21
Show Beijing Conference & Exhibition on Instrument Analysis
Location Beijing Exhibition Center, Beijing, China, Booth #00

Date October 30-November 1
Show Chem Show
Location Javits Convention Center, New York, NY

Date October 30-November 2
Show 2007 SEMA Show
Location Las Vegas Convention Center, Las Vegas, NV

Date October 31-November 3
Show 33rd Annual NEAFS Meeting
Location The Sagamore Resort, Bolton Landing, NY

November, 2007

Date November 1
Show 2007 ANACHEM Symposium
Location Burton Manor, 27777 Schoolcraft Road, Livonia, MI

Date November 7-9
Show 3rd International Symposium on Recent Advances in Food Analysis
Location Diplomat Hotel–Conference Center, Prague, Czech Republic

Date November 11-15
Show Eastern Analytical Symposium (EAS)
Location Garden State Convention & Exhibit Center, Somerset, NJ

Date November 11-15
Show 2007 AAPS Annual Meeting and Exposition
Location San Diego Convention Center, San Diego, CA

Date November 28-30
Show 31st Int'l Symposium on Capillary Chromatography & Electrophoresis
tel Albuquerque, Albuquerque, NM

es, see our Tradeshow Calendar
com/ontheroad.