the RESTEK ADVANTAGE

2006.01

introducing...

New Rxi™ GC Column Series

The Ultimate High Performance Fused Silica Capillary Column

While GC analytical methods have continued to evolve, capillary column technology has been largely unchanged over the last several years. Using new techniques for deactivation, stationary phase synthesis, and coating, and tight controls over column manufacturing, Restek has set the benchmark for column performance by introducing the Rxi series. Inside you will find more information about this revolutionary new family of GC columns which demonstrate...

- unsurpassed inertness
- ultra low bleed
- guaranteed column to column reproducibility

Exclusively from Restek!

See **pages 3 & 8**.

RESTEK 20 YEARS

Turning Visions into Reality™

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Erratum
The transfer line used in the methyl *tert*-butyl ether / *tert*-butyl alcohol analysis reported in Advantage 2005v4 (Figure 1, page 4) was the factory-installed Eclipse transfer line.

We thank Laura Chambers at O.I. Analytical, College Station, Texas, for reviewing the analysis with us, and we are very grateful to O.I. Analytical for their generous loan of the O.I. 4660 Eclipse purge and trap system.

By Professor Walter Jennings ("Walt")
Professor Emeritus, University of California, Davis; Co-Founder, J&W Scientific, Inc.; Co-Founder, AirToxics, Ltd.; waltj@pacbell.net

For the past few years, the aging process has been catching up with the Jennings family. While I still enjoy participating in seminars and lectures, I now find seven to ten events on a two week trip is more tiring than it was just a few years ago. In addition, my wife has been successfully battling Parkinson's disease for almost twenty years, but we realize that it is now inexorably advancing. Hence, she needs more of my time, and I must limit myself to shorter absences. Because of these developments, I permitted my Agilent contract to expire when it ran out on June 30, 2005. Agilent was sympathetic and understanding, our parting was amicable, and I still value my contacts with them. But after two months in my home office, I sometimes feel a need for the challenge of discussion and argument, and when friends at Restek asked if I would be interested in writing a short paper that was purely educational and pushing no particular product line, it sounded appealing. Here it is.

The "Replacement" Column, A Recurring Problem in Gas Chromatography

One of the problems that gas chromatographers frequently ask concerns the behavior of a "replacement" column. Even skilled practitioners have been known to protest when they install a replacement column, use the same operational parameters, and find that not only have solute retention times shifted, but peak 15 now elutes prior to peak 13. In most such cases, they blame the column manufacturer. There are programs available to correct this problem, but some of those solutions have been so simplified that the user still has no comprehension of the causative factors, a state of blissful ignorance which should be corrected.

Columns are produced, bought, and sold using nominal measurements, e.g., "30 meters x 0.25mm, film thickness 0.25µm". As a specification, this is not equivalent to "30.0 meters x 250µm". Depending on the manufacturer's specifications, the actual column dimensions may be "30 +/- 1 meter x 250 +/- 6µm". Some manufacturers now give better attention to the length tolerance, but the diametric variation will continue to be a problem. Fused silica draw towers are often computer controlled, with the feed rate of the silica blank, the draw rate of the capillary tube, and the temperature of the softening oven controlled by a computer whose input comes from a laser micrometer that monitors the tubing diameter during the drawing process. In our hands, a blank could be drawn to approximately 14 kilometers of 0.25mm capillary tubing. The two ends of that tube may show a significant variation in diameter, but those changes occur so slowly that over lengths of a few hundred meters the diameter is reasonably constant. It is rare to find a column where the diameters at the two ends are significantly different, but it is not unusual to find that columns from the two ends of that draw, or from different draws, do exhibit significant differences in diameter, e.g., 244µm vs. 256µm[i].

An analyst whose original 29.9m x 256µm column is replaced by one measuring 30.1m x 244µm will likely experience difficulties if he or she uses the same operational parameters, i.e., same temperature program, same carrier gas, same inlet and outlet pressures. Because of the geometric differences, the columns possess different pressure drops and under the same operational parameters, the carrier gas velocities would be different in the two columns. This will affect solute retention times, and this introduces the major problem.

Gas chromatography is a volatility phenomenon, and solutes elute in a sequence mandated by what I prefer to call their "net vapor pressures". The net vapor pressure is a function of the intrinsic vapor pressure of that solute, increased by the temperature at that point in the program, and *further decreased by the sum of all interactions between that solute and that stationary phase.*[ii] The strengths of these various interactive forces usually vary inversely with temperature in a non-linear manner, and for a given increase in temperature both the rate of change and the degree of change are unique functions of that solute in that stationary phase under these particular conditions. As a result, the molecules of a chromatographing solute experience a specific temperature-sensitive "selectivity profile" in their passage through the column. These interactions are rendered moot while those molecules are in the mobile phase, and endure only while they are in contact with the stationary phase. Hence we are interested in keeping retention times, and particularly t'_R (time in stationary phase) constant from column to column and run to run. From the two relationships of $K_c = ßk$ and $\mu = L/t_M$ we can establish that $t'_R = c_S/c_M \times d_f/d_c \times L/u$. The three terms of course are the distribution constant, the reciprocal of the phase ratio, ß, and column length divided by the average linear gas velocity. K_c is a function of the solute, the stationary phase, and the temperature. While, by definition, the temperature changes in program mode, the rate of change is constant, run to run and column to column, under the same program parameters, and one can usually ignore this term if the two stationary phases are indeed identical[iii]. The second term can also be ignored, provided the ratio of d_f/d_c is constant. Column diameter, d_c and column length are both nominal values and usually differ from column to column. We can compensate for either or both of these by varying the gas velocity, u. This is most easily accomplished in constant pressure mode. In constant flow mode it is more complicated and beyond the scope of this paper.

In constant pressure mode, the solution is quite simple, assuming that the replacement column has the same stationary phase and the same phase ratio as the original column. 1) Using the original operational parameters (initial temperature and program parameters, column inlet and outlet pressures, same carrier) install the new column and inject the same mixture. 2) Determine the retention time of an easily identifiable peak, and compare this to the retention time of that peak on the original column. 3) Adjust the column inlet pressure to make the retention time of the target peak the same as it was on the original column. Retention times on the replacement column should now agree closely with the values observed on the original column, each solute will now experience its original temperature-sensitive "selectivity profile", and chromatograms generated on the replacement column should essentially duplicate those from the original column.

i Fortunately, the column phase ratio (ß) is usually unaffected by these changes in diameter because almost all manufacturers currently employ static coating methods. Provided the concentration of the stationary phase in the coating solution remains constant, the ratio of the film thickness (d_f) to column diameter (d_c) will remain constant.

ii These interactions include (but are not limited to) dispersive interactions, hydrogen bonding and other proton forms of proton sharing, dipole interactions, and in some cases, molecular size and shape.

iii In some cases, surface preparation and deactivation treatments can also affect retentions. These treatments are generally proprietary and vary from manufacturer to manufacturer. With complex mixtures, the separations achieved on columns coated with the same stationary phase but from different suppliers may yield slightly different results.

New Rxi™ Fused Silica Columns

Ultimate High-Performance Fused Silica Capillary Columns

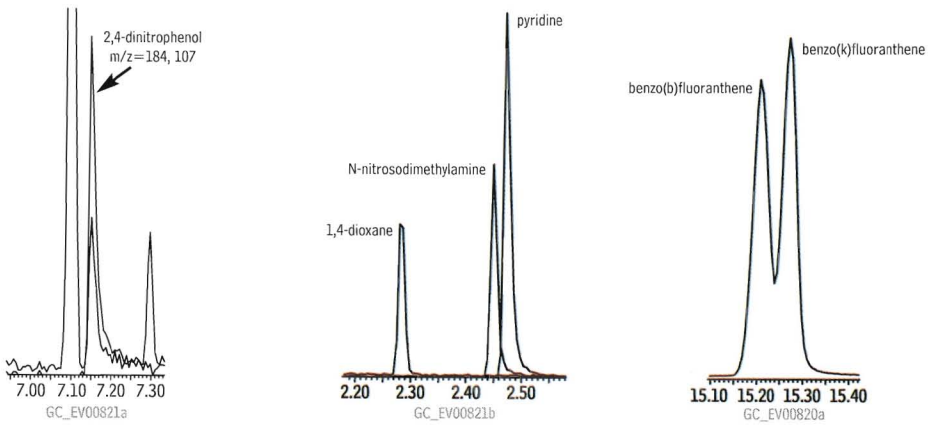
- Unsurpassed inertness for low-level acidic and basic compounds.
- Ultra-low bleed.
- Reliable performance, guaranteed column to column reproducibility.
- Guaranteed to work perfectly with retention time-locking software.

In recent years there have been few advances in capillary GC column technology. Through new, innovative technology, Restek has developed and optimized a column-making procedure that assures low bleed, unsurpassed inertness, and exceptionally reproducible columns, batch to batch.

Unsurpassed Inertness

Rxi™ columns improve chromatography for many acidic or basic compounds. Surface activity in a column is revealed by the sensitivity and peak shapes for analytes such as 2,4-dinitrophenol (acidic) and pyridine (basic). Sub-nanogram quantities of these compounds are a stringent test of inertness. Rxi™ columns' unsurpassed inertness allows analysis of acidic or basic compounds under the same conditions, as shown here.

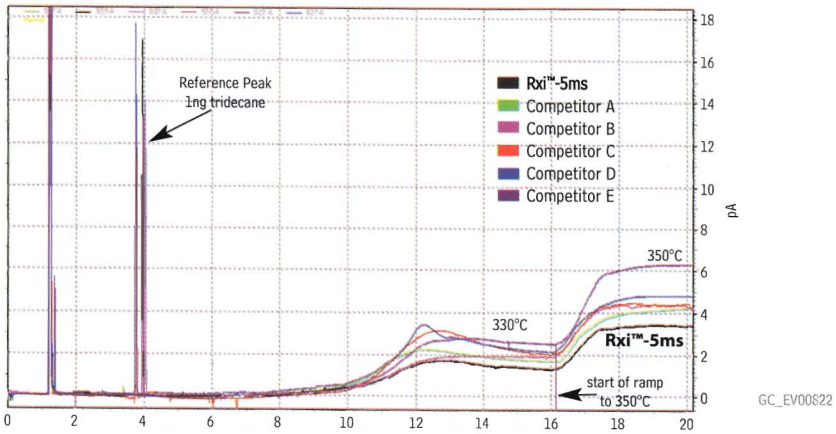
Figure 1 Rxi™-5ms columns have excellent sensitivity and symmetry for difficult compounds.



Ultra-Low Bleed

Bleed from Rxi™ columns is the lowest in the industry, simplifying trace-level analysis with mass spectrometric detectors (MSD, ion trap, etc.), electron capture detection (ECD), nitrogen-phosphorus detection (NPD), or other sensitive detection methods.

Figure 2 Profiles for widely used columns prove Rxi™-5ms has the lowest bleed!



Introducing...

Restek's exceptionally inert (Rxi) fused silica capillary columns:
In addition to bleed, column-to-column uniformity has been elusive - see editorial *The "Replacement" Column A Recurring Problem in Gas Chromatography*, by guest editor Professor Walter Jennings, on the facing page.

The processes we use to make new Rxi™ columns enable us to **guarantee** highly uniform performance, column to column and lot to lot, including perfect match-up with retention time-locking software. It is our promise and commitment to you that every Rxi™ column you receive will be **exactly** as good as the one it replaces.

Continued on the outside back cover.

Improved SPE Cartridges for Massachusetts EPH Analysis

Monitoring Environmental Petroleum Hydrocarbons by SPE/GC

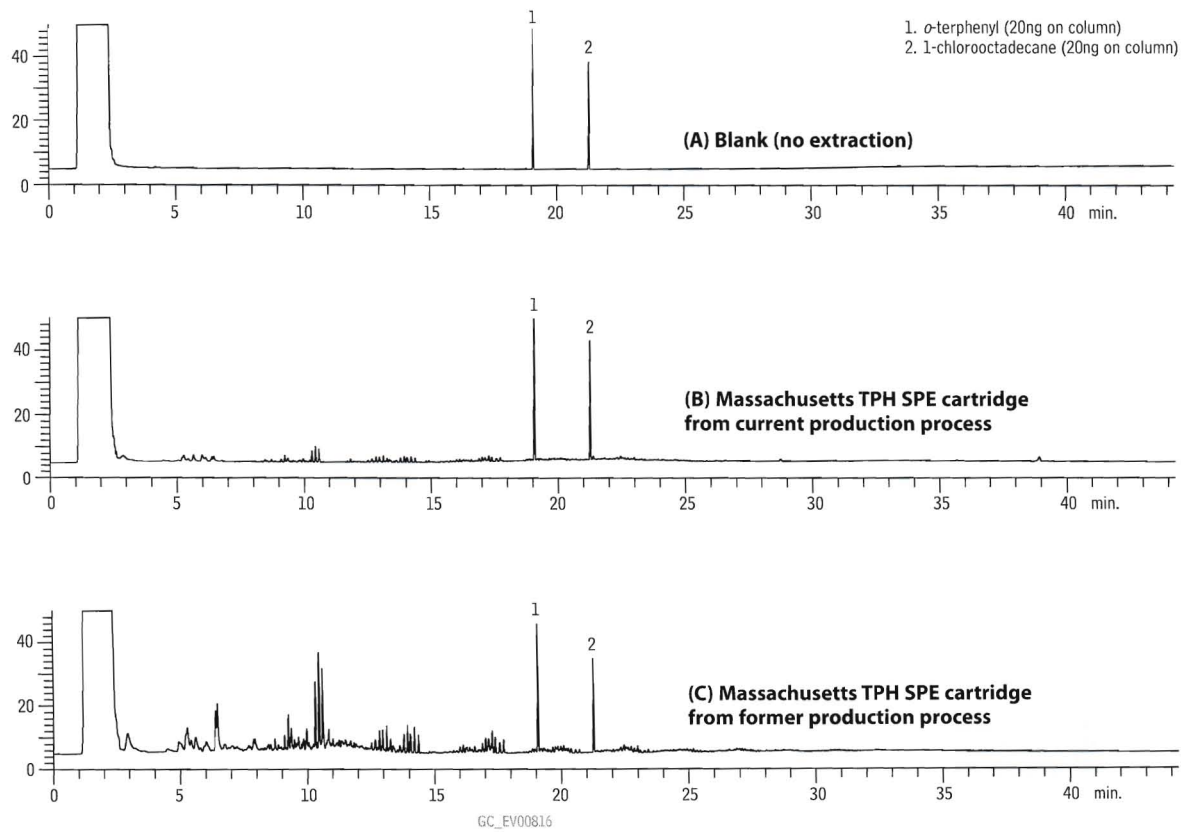
By Lydia Nolan, Innovations Chemist

- New Massachusetts TPH SPE cartridges reduce extractable contaminants and assure more reliable fractionation.
- Large uniform lots of silica reduce frequency of verifying fractionation results.
- New packaging reduces coextractables, provides better protection from humidity.

Concern about the effects of materials from leaking underground petroleum storage tanks has grown over the last ten years. In addition to the US Environmental Protection Agency and international groups, several US states, including Massachusetts[1] and Texas[2], have developed methods for analyzing samples from their geographical areas. Because of the broad and thorough nature of the quantitative information generated by the Massachusetts Department of Environmental Protection method[1], many site managers and engineering firms outside of Massachusetts request this method for their samples.

Most frustrating for labs using this method has been the uniformity of commercial silica gel-containing solid phase extraction (SPE) cartridges used to prepare the samples for analysis. The activity level and capacity of the silica, the uniformity of the bed, and the quality of the cartridge components and packaging are critical toward good results. Slight variations in the silica material, or in humidity levels during manufacture of the cartridges, can have a dramatic effect on the hexane fractionation results. The volume of hexane required to fractionate the aliphatic portion of the sample, without allowing any aromatic analytes to break through into the hexane fraction, can vary, typically from 17mL to 20mL ± 0.5mL, and must be determined for every lot of cartridges. Trace levels of phthalates and other contaminants from cartridges, frits, and packaging are easily extracted with the desired analytes, complicating

Figure 1 Low levels of background extractables in new Restek Massachusetts TPH SPE cartridges.



Column:	Rtx®-5 30m, 0.32mm ID, 0.25µm (cat. #10224)
Sample:	50µL diluted MA EPH Surrogate Spike Mix (cat.# 31479) (diluted to 400µg/mL)
Inj.:	0.5µL splitless (hold 0.75 min.), Precision™ split inlet liner with wool (cat.# 21027)
Inj. temp.:	290°C
Inj. press. prog.:	pressure pulse to 50cm/sec. @ -0.71min. pressure to 35cm/sec. @ 0.8 min.
Carrier gas:	helium
Linear velocity:	35cm/sec., constant velocity
Oven temp.:	40°C (hold 1 min.) to 310°C @ 15°C/min.
Det.:	PerkinElmer AutoSYS™ GC-FID @ 330°C

Table I Recovery and reproducibility for aliphatics and aromatics via Massachusetts TPH SPE fractionation, using new Restek SPE cartridges.

	Aliphatic Fraction			Aromatic Fraction		
	% Rec.ov.	Std. Dev.	RSD	% Recov.	Std. Dev.	RSD
1. nonane (C9)	86.4	9.11	10.5			
2. decane (C10)	84.7	7.17	8.5			
3. naphthalene				82.3	6.09	7.4
4. dodecane (C12)	83.8	8.33	9.9			
5. 2-methylnaphthalene				89.1	5.50	6.2
2-fluorobiphenyl (surrog.)				92.1	6.70	7.3
6. tetradecane (C14)	90.7	6.29	6.9			
7. acenaphthylene				91.6	7.63	8.3
2-bromonaphthalene (surrog.)				84.9	6.82	8.0
8. acenaphthene				93.4	6.32	6.8
9. fluorene				92.4	6.19	6.7
10. hexadecane (C16)	90.9	4.37	4.8			
11. phenanthrene				90.4	5.55	6.1
12. octadecane (C18)	94.9	3.45	3.6			
13. anthracene				91.5	5.29	5.8
14. nonadecane (C19)	91.1	3.63	4.0			
o-terphenyl (int. std.)				96.4	3.43	3.6
15. eicosane (C20)	89.8	2.64	2.9			
16. fluoranthene				93.4	3.16	3.4
1-chlorooctadecane (int. std.)	83.1	5.02	6.0			
17. pyrene				95.1	3.84	4.0
18. docosane (C22)	85.2	3.97	4.7			
19. tetracosane (C24)	85.0	3.23	3.8			
20. benzo(a)anthracene				91.2	2.38	2.6
21. chrysene				90.9	2.56	2.8
22. hexacosane (C26)	85.8	2.97	3.5			
23. octacosane (C28)	85.7	2.51	2.9			
24. benzo(b)fluoranthene				91.3	2.23	2.4
25. benzo(k)fluoranthene				90.8	2.10	2.3
26. benzo(a)pyrene				91.0	2.67	2.9
27. triacontane (C30)	86.0	2.49	2.9			
28. dibenzo(a,h)anthracene				90.9	1.78	2.0
29. indeno(1,2,3-cd)pyrene				91.4	1.48	1.6
30. benzo(ghi)perylene				90.7	2.21	2.4
31. hexatriacontane (C36)	78.6	3.95	5.0			

n=4 (2 analyses on each of 2 lots of SPE cartridges)

Analytical Conditions

Column:	Rtx®-5 30m, 0.32mm ID, 0.25µm (cat. #10224)
Sample:	50µL Mass EPH Surrogate Spike Mix (cat.# 31479) diluted to 400µg/mL 1mL MA Fractionation Check Mix (cat.# 31481), 25µg/mL in hexane 1mL MA Fractionation Surrogate Spike Mix (cat.# 31480), diluted to 40µg/mL in hexane
Inj.:	0.5µL splitless (hold 0.75 min.), Precision™ split inlet liner with wool (cat.# 21027)
Inj. temp.:	290°C
Inj. press. prog.:	pressure pulse to 50cm/sec. @ -0.71min. pressure to 35cm/sec. @ 0.8 min.
Carrier gas:	helium
Linear velocity:	35cm/sec., constant velocity
Oven temp.:	40°C (hold 1 min.) to 310°C @ 15°C/min.
Det.:	PerkinElmer AutoSYS™ GC-FID @ 330°C

SPE Method

Tube:	Massachusetts TPH 20mL/5g, cat.# 26065
Tube conditioning:	30mL hexane; do not allow top frit or bed to dry.
Sample:	Add 1mL EPH sample in hexane.
Elution #1:	Using gravity or very low vacuum, pass 18mL hexane through tube.* Do not allow top frit or bed to dry; collect this aliphatic fraction in a clean sample container. Reduce eluate to 1mL under gentle nitrogen purge or other concentration technique. Do not concentrate to less than 1mL or allow eluate to dry before analysis.
Elution #2:	Using gravity or low vacuum, pass 20mL methylene chloride through tube. Do not allow top frit or bed to dry; collect this aromatic fraction in a clean sample container. Reduce to 1mL (see above) and analyze.

*Note that the volume of hexane will vary, and should be verified in each laboratory.
For details concerning the SPE method, refer to the original method in Reference 1.

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.

2 Total Petroleum Hydrocarbons, TNRCC Method 1005, Revision 03 (June 1, 2001); Draft TNRCC Method 1006 (May 2000) Texas Natural Resource Conservation Commission.

low level quantification. Consequently, quality must be assured for each lot of cartridges and, sometimes, even within lots.

We have always specially treated our Massachusetts TPH SPE cartridges (cat.# 26065) to ensure minimum background extractables and maximum silica activity. Now, a new process has allowed us to reduce extractables even further, and assure greater reliability of fractionation. Larger uniform lots of silica will reduce the frequency with which a lab will need to verify fractionation results. New packaging ensures reduced levels of coextractables and better protection from environmental humidity.

Figure 1C shows the background of a typical previous lot of cartridges, compared to the significantly lower background from the new product, in Figure 1B. All cartridges were extracted with 15mL of hexane, with no prior conditioning. The hexane was evaporated, o-terphenyl and 1-chlorooctadecane were added, and samples were reconstituted to 1mL for analysis by GC-FID. Fractionation, extraction efficiency, and reproducibility also are excellent, as shown by the summary in Table I. Details of the extraction method, based on the Massachusetts procedure, also are presented in Table I.

If you are conducting Massachusetts EPH analyses, or similar analyses, and have been concerned about the quality and uniformity of the SPE cartridges you have been using, we think you will be as impressed as we are with the quality of our new product.

Massachusetts TPH SPE Cartridges

20mL, 5g, 20-pk. 26065

Rtx®-5 Column (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

MA EPH Surrogate Spike Mix

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

MA Fractionation Check Mix

(31 components)
25µg/mL each in hexane, 1mL/ampul
cat. # 31481 (ea.)

MA Fractionation Surrogate Spike Mix

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

New Reference Mix of Canadian Drinking Water Volatiles

By Jason Thomas, Environmental Innovations Chemist

- New, complete mix includes 19 volatiles on Canadian Drinking Water List.
- Simple purge and trap GC/MS analysis.
- Rtx®-VMS column provides sharp peaks for early eluters, resolves heavier compounds.

Much like the US Environmental Protection Agency's regulation of environmental contaminants in drinking water through the Safe Drinking Water Act, Canada has its own stipulations regarding drinking water. These mandates are laid out in the *Guidelines for Canadian Drinking Water Quality* published by Health Canada's Water Quality and Health Bureau. Regulation falls under the jurisdiction of the individual provinces and territories, which use these guidelines to establish water quality requirements for municipal water sources.[1]

Here, we illustrate the analysis of the volatiles portion of the Canadian contaminants list, now available from Restek as Canadian Drinking Water Volatiles Mix (cat.# 30610). We analyzed a 25mL water sample containing 50ppb each analyte, using an OI Analytical 4660 purge and trap system, with autosampler, and an HP 5890/5971 GC/MS system. A 30m x 0.25mm ID x 1.4µm Rtx®-VMS column (cat.# 19915), in conjunction with a Siltek® deactivated 1mm ID split inlet liner and a 35:1 split, affords good peak shape for the early-eluting components, as well as good resolution for the heavier compounds. The Rtx®-VMS column is an excellent choice for many other volatiles applications as well.

Reference

[1]http://www.hc-sc.gc.ca/ewh-semt/water-eau/drink-potab/index_e.html

Rtx®-VMS Column (fused silica)

(proprietary Crossbond® phase)

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

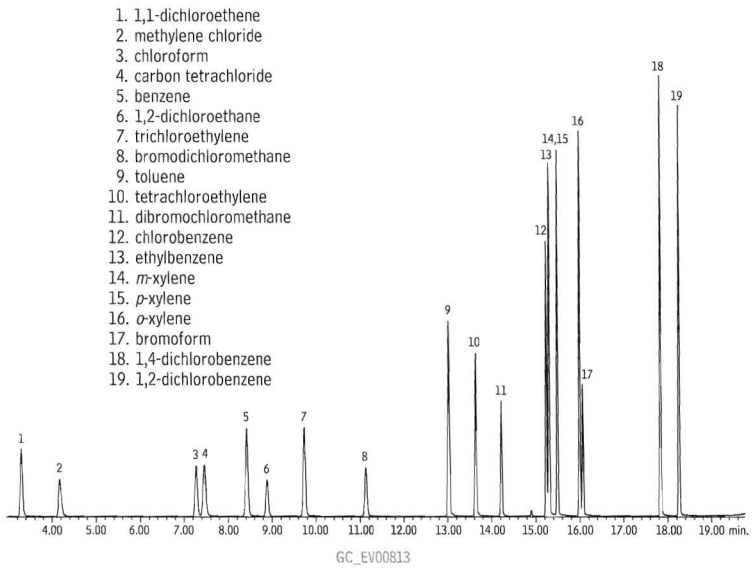
Canadian Drinking Water Volatiles Mix new!

(19 components)

benzene
bromodichloromethane
bromoform
carbon tetrachloride
chlorobenzene
chloroform
dibromochloromethane
1,2-dichlorobenzene
1,4-dichlorobenzene
1,2-dichloroethane
1,1-dichloroethylene
ethylbenzene
methylene chloride
tetrachloroethylene
toluene
trichloroethylene
m-xylene
o-xylene
p-xylene

2,000µg/mL each in P&T methanol, 1mL/ampul
cat. # 30610 (ea.)

Figure 1 Canadian Drinking Water Volatiles Mix analyzed on an Rtx®-VMS column.



Column:	Rtx®-VMS 30m, 0.25mm ID, 1.4µm (cat.# 19915)
Sample:	50 ppb each analyte in 25mL water, prepared from Canadian Drinking Water Volatiles Mix (cat.# 30610), 2000µg/mL each component in purge & trap methanol
Inj.:	split, 35:1, 1mm ID Siltek®-treated split inlet liner (cat.# 20972-214.1)
Inj. temp.:	200°C
Carrier gas:	helium
Linear velocity:	30cm/sec. @ 35°C, constant pressure
Oven temp.:	35°C (5 min.), to 70°C @ 5°C/min., to 220°C @ 20°C/min. (hold 3 min.)
Det.:	MS
Transfer line temp.:	150°C
Scan range:	35-250 amu
Ionization:	EI
Mode:	scan

Purge and Trap Conditions

Instrument:	OI 4660 Eclipse Purge & Trap
Trap:	#10 (Tenax®/silica gel/carbon molecular sieve)
Sample temp.:	40°C
Purge:	11 min. @ 40mL/min.
Desorb preheat:	185°C
Desorb:	0.5 min. @ 190°C
Desorb flow rate:	35.0mL/min.
Bake:	6 min. @ 210°C
Interface:	split injector
Transfer line temp.:	150°C

Analyze Hydrocarbons on OPN/Res-Sil™ C Bonded GC Packing

Superior Replacement for Porasil® Packings

By Barry Burger, Petroleum Chemist

- Unique separations of saturated and unsaturated hydrocarbons.
- Innovative bonding chemistry for batch-to-batch reproducibility, excellent thermal stability, and long life.
- Other bonded phases available.

For years, Porasil® C and Porasil® B, modified with covalently attached liquid phases such as OPN (cyanopropyl) or *n*-octane functional groups, offered important advantages, relative to conventional GC packings, in analyses of C1-C4 hydrocarbons: faster separations, higher thermal stability, shorter conditioning times, and longer lifetimes. Porasil® C / Porasil® B products were discontinued in the 1980s, however, and inventories have been depleted, forcing those that use these packings to search for comparable materials.

Restek chemists solved the problem by developing Res-Sil™ C and Res-Sil™ B bonded packings. These packings afford all of the advantages of the Porasil® C and Porasil® B materials, with the added advantage of consistent batch-to-batch performance - and they are readily available for immediate delivery. Compared to diatomaceous earth media, Res-Sil™ C has a small surface area, good inertness, low friability, and less reactivity.

Unique Selectivity for Process GC and High-Speed Analysis

Speed of analysis is crucial in process GC, and in laboratory gas analyzers in which multiple columns and valve switching are used to separate complex gas mixtures. Res-Sil™ C bonded packings are ideal for resolving the difficult-to-separate saturated and unsaturated C4 hydrocarbons under these demanding conditions. Figure 1 illustrates the unique selectivity of OPN on Res-Sil™ C packing for eluting *cis*-2-butene before 1,3-butadiene. When used in series with other columns, this unique material provides petroleum and petrochemical method developers with a powerful tool for fast determination of C1-C4 hydrocarbons.[1]

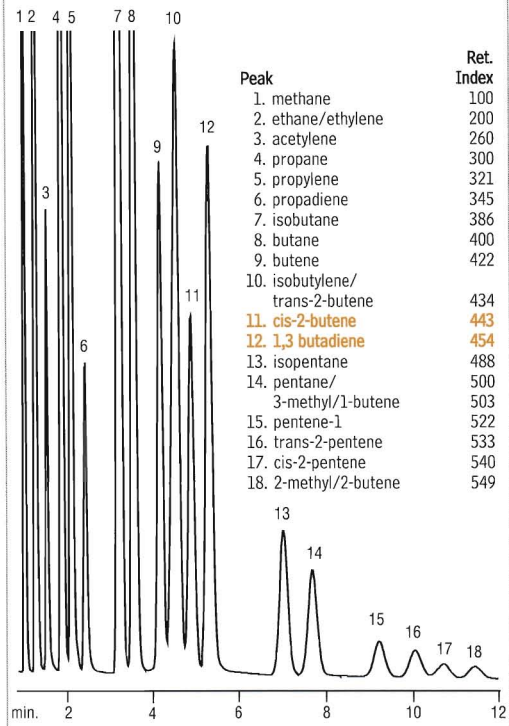
Stringent QA Assures Batch-to-Batch Consistency

Historically, one of the problems with bonded phases on Porasil® media has been batch-to-batch variations in the amount of liquid stationary phase incorporated on the silica support. Through our new synthesis pathways, we precisely control the amount of bonded liquid phase on Res-Sil™ C in every batch of packing, assuring reproducible retention times and separations. Each batch of packing is tested with a complex mixture of hydrocarbons, to confirm it meets demanding retention time and retention index specifications. We evaluate column bleed at the recommended maximum temperature, 150°C, to ensure that there are no retention shifts or high baselines.

In addition to OPN on Res-Sil™ C packing, we bond *n*-octane and Carbowax® 1540 phases to Res-Sil™ C. Each of these packings offers a conditioning time of less than 30 minutes, low bleed, long lifetime, and consistent batch-to-batch reproducibility. For details about *n*-octane or Carbowax® on Res-Sil™ C, and for bonded phase packings on Silcoport™ deactivated diatomaceous earth, refer to our current chromatography supplies catalog. We test every batch of every Restek bonded phase packing for bleed, efficiency, retention index, and retention time reproducibility. In addition, we make a broad range of packed and micropacked columns in specially-deactivated Silcosteel® tubing, for superior inertness and efficiency.

If you have been looking for a replacement for a Porasil® C or Porasil® B packing, we invite you to contact us. Your search should end here.

Figure 1 OPN on Res-Sil™ C packing has unique selectivity for *cis*-2-butene and 1,3-butadiene.



Peak	Ret. Index
1. methane	100
2. ethane/ethylene	200
3. acetylene	260
4. propane	300
5. propylene	321
6. propadiene	345
7. isobutane	386
8. butane	400
9. butene	422
10. isobutylene/ trans-2-butene	434
11. cis-2-butene	443
12. 1,3 butadiene	454
13. isopentane	488
14. pentane/ 3-methyl/1-butene	500 503
15. pentene-1	522
16. trans-2-pentene	533
17. cis-2-pentene	540
18. 2-methyl/2-butene	549

Reference standard courtesy of DCG Partnerships, Ltd., Pearland, TX.

12' x 2mm ID x 1/8" OD Silcosteel® column packed with OPN on Res-Sil™ C 80/100 mesh.
20µL on-column injection of refinery gas

Oven Temp.:	50°C
Inj. temp.:	200°C
Det. temp.:	200°C
Flow rate:	30mL/min., He

Res-Sil™ C Packings

Description	cat.#
Res-Sil™ C, 80/100 mesh, 10g*	25028
OPN on Res-Sil™ C, 80/100 mesh, 10g*	25042
n-Octane on Res-Sil™ C, 80/100 mesh, 10g*	25030
2% Carbowax® 1540 on Res-Sil™ C, 80/100 mesh, 10g*	25044

Reference

1 Saha, N.C., S.K. Jain, and R.K. Dua. *J. Chromatogr. Sci.* 16: 323-328 (1978).
Reference not available from Restek.

Sensitive GC/MS Analysis for Drugs of Abuse

Rxi™-5ms Column Resolves Acidic/Neutral or Basic Drugs

By Kristi Sellers, Clinical/Forensic Innovations Chemist

- New stationary phase, inert to acidic or basic drugs.
- Unique deactivation for low column bleed at 330°C.
- Column technology specially developed for GC/MS.

new **column** technology!

GC/MS is considered the standard for confirming the presence of abused drugs in body fluids, including acidic drugs (e.g., methaqualone), neutral drugs (e.g., phenobarbital), and basic drugs (e.g., methamphetamine). These methods are well established, and the positive identifications mass spectral data generate are accepted as confirming evidence in courts of law. The accepted stationary phase for these analyses is a 5% phenyl / 95% methyl polysiloxane phase, because it provides the best selectivity for separating the drugs and their metabolites. Unfortunately, not all 5% phenyl columns provide the inertness needed to accurately quantify low concentrations of reactive acidic or basic drugs.

Now, Restek's R&D chemists have developed a new 5% phenyl stationary phase and a unique column deactivation technology specifically for GC/MS. The product of this combination - the Rxi™-5ms column - ensures enhanced inertness for acidic or basic compounds, while maintaining the selectivity of a conventional 5% phenyl column.

Using mixtures of acidic/neutral drugs and basic drugs in their free base form, at an on-column concentration of 50ng for each drug, we evaluated a 30m, 0.25mm ID, 0.25µm Rxi™-5ms column for resolution, inertness, and bleed. Figure 1 shows chromatography for acidic/neutral drugs and Figure 2 shows basic drugs. In either analysis, all compounds are resolved to baseline and exhibit Gaussian peak shapes. Furthermore, there is no interference from column bleed - not even at 330°C. Note that a Siltek® treated inlet liner contributes to these results: our unique Siltek® surface passivation process assures the liner will have the inertness needed for accurate low-level analyses of reactive acids or bases.

In combination, an Rxi™-5ms column and a Siltek® treated inlet liner represent a complete solution for analyzing acidic, neutral, and basic drugs by GC/MS. For additional dimensions of Rxi™-5ms columns, and for Siltek® treated inlet liners for your chromatograph, please refer to the 2006 Restek catalog - or visit our website.

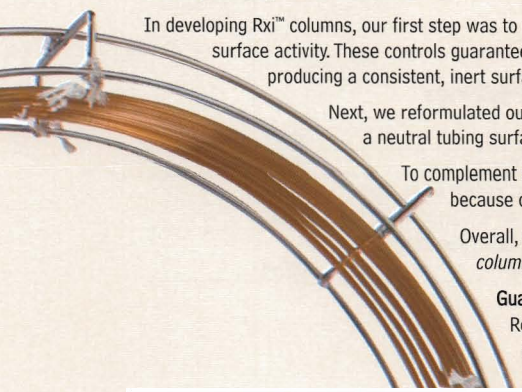
Rxi™ Columns, Ultimate High Performance Capillary GC Columns

Rxi™ columns were created at Restek's cutting-edge research facility, Restek West, in California. Our senior polymer chemists developed new column technology, based on our Crossbond® chemistry, to create this new column line. The columns we produce as a result of their work exhibit exceptional inertness and unsurpassed reproducibility, from column to column and lot to lot. Acidic or basic compounds chromatograph beautifully, at sub-nanogram on-column levels, with no peak tailing. Ultra-low bleed assures compatibility with sensitive detectors or in trace-level GC/MS analysis. We tuned this unique chemistry until polymer selectivity was locked in, to allow install-and-run use of Rxi™ columns with retention time-locking software.

Restek West
Shawn Reese, Gianna Barlupi, Roy Lautamo

What makes Rxi™ columns different from other columns?

First, and foremost, unique deactivation and our modified Crossbond® chemistry create columns with superior performance. The raw materials we use in the manufacturing process - both tubing and chemicals - are strictly controlled. Cleanliness and precision are critical to every step in the process. In addition, we looked in-depth at all other aspects of the column manufacturing process, to establish a highly reproducible process. In both performance and column-to-column consistency, Rxi™ columns are surpassed by no other columns.



In developing Rxi™ columns, our first step was to work with our fused silica tubing supplier to establish rigorous controls on internal diameter, outer diameter, ovality, and surface activity. These controls guarantee our tubing is a known starting point. Then, we treat this highly uniform tubing with our unique deactivation chemistry, producing a consistent, inert surface on which to apply the polymer.

Next, we reformulated our polymers, taking steps to ensure neutrality and to fine tune selectivity for retention time locking. A neutral polymer and a neutral tubing surface are important contributors toward excellent peak shape for both acidic and basic compounds.

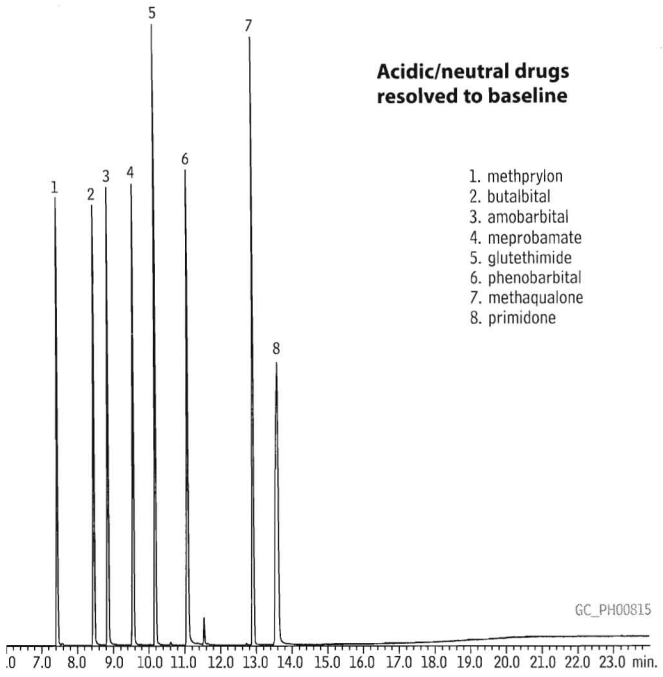
To complement these efforts, we developed a new column manufacturing process that creates a very reproducible product. This is critical, because our customers' workdays are simplified when every new column they purchase performs exactly as its predecessor.

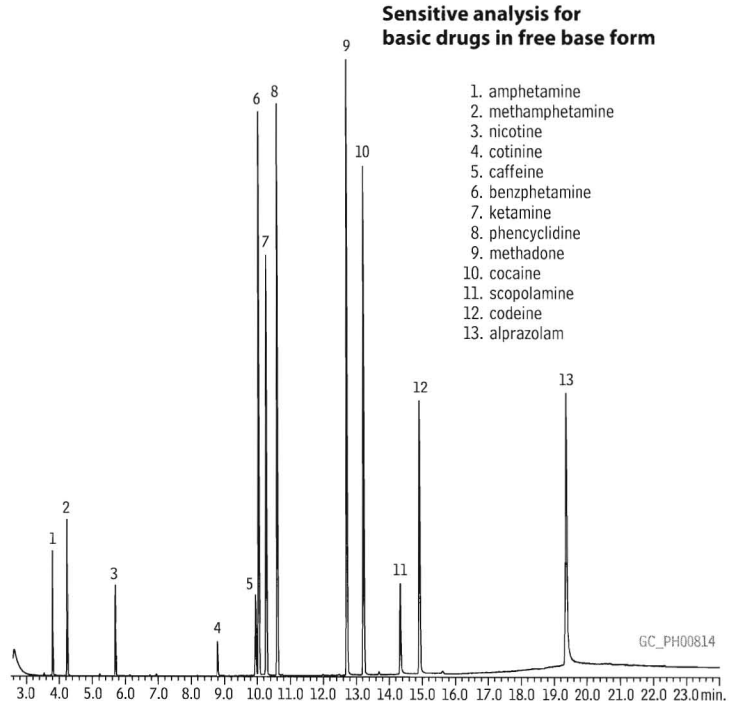
Overall, the results of these efforts are columns that define *unsurpassed inertness*, *ultra low bleed*, and *totally reliable column-to-column performance*.

Guaranteed Quality and Reliability

Restek is committed to supplying the most reliable GC columns in the industry. Every Rxi™ column is individually challenged to pass our stringent requirements for film thickness, coating efficiency, selectivity, inertness, and bleed. We believe Rxi™ column technology produces the most reliable columns available, anywhere, and we promise that every Rxi™ column you

Figure 1 Analyze acidic drugs or basic drugs under the same conditions, using an Rxi™-5ms column.





Column:	Rxi™-5ms 30m, 0.25mm ID, 0.25µm (cat.# 13423)
Sample:	1000µg/mL each in methanol
Inj.:	1.0µL split (50:1), 20ng each compound on column; Siltek® treated 4mm gooseneck splitless inlet liner (cat # 20799-214.5)
Inj. temp.:	250°C
Carrier gas:	helium, constant pressure
Linear velocity:	30cm/sec.
Oven temp.:	100°C to 220°C @ 15°C/min., to 330°C @ 10°C/min. (hold 5 min.)
Det.:	Agilent 5973 MSD
Transfer line temp.:	300°C
Scan range:	35-550
Tune:	PFTBA
Ionization:	EI

Exempted Drug of Abuse Reference Materials

new!

1,000µg/mL in P&T methanol (*except where noted), 1mL/ampul

Compound	Individual cat.#
Benzodiazepines	
alprazolam	34042
bromazepam	34043
chlordiazepoxide HCL	34044
clobazam	34045
clonazepam	34046
diazepam	34047
flunitrazepam	34049
flurazepam di-HCL	34050
lorazepam	34051
nitrazepam	34053
oxazepam	34054
prazepam	34055
temazepam	34056
triazolam	34057
Cocaine & Metabolites	
cocaine HCL	34015
benzoylecgonine	34016
ecgonine	34017
ecgonine methyl ester	34018
Methadone & Metabolites	
methadone HCL	34005
Amphetamines & Metabolites	
d-amphetamine	34020
(+)methamphetamine	34021
Opiates & Metabolites	
codeine	34000
hydrocodone	34002
hydromorphone	34063
morphine	34006
oxycodone	34007
oxymorphone	34065
Cannabinoid & Metabolites	
cannabidiol	34011
cannabinol	34010
Barbiturates	
amobarbital	34028
aprobarbital	34029
barbital	34030
butabarbital	34031
butalbital	34032
DL-glutethimide	34058
hexobarbital	34033
mephobarbital	34034
methohexital	34035
pentobarbital	34036
phenobarbital	34037
secobarbital	34038
talbutal	34039
thiamylal	34040
thiopental	34041
Other	
benzphetamine	34022
cocaethylene*	34066
fenfluamine	34023
levorphanol	34003
meperidine	34004
meprobamate	34059
methaqualone	34064
methyprylon	34060
pentazocine	34062
phencyclidine	34027
phendimetrazine	34025
phenmetrazine	34026
phentermine	34024
dextro-propoxyphene	34008
thebaine	34009

Rxi™-5ms Column (fused silica)

new!

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

ID	df (µm)	temp. limits	length	cat. #
0.25mm	0.25	-60 to 330/350°C	30-Meter	13423

RP-HPLC Analysis of Selective Serotonin Reuptake Inhibitors

Using Allure™ Basix and Ultra PFP Polar Stationary Phases

By Rick Lake, Pharmaceutical Innovations Chemist

- Good retention and selectivity without ion-pairing chromatography.
- Practical at acidic pH (Ultra PFP phase) or neutral pH (Allure™ Basix phase).
- Improved peak shape for basic compounds, compared to alkyl phases.

Selective serotonin reuptake inhibitors (SSRIs) are a novel class of antidepressants that have gained much acceptance in the medical community. Although they have been found to be no more effective than the "older" tricyclic antidepressants, they produce fewer side effects. Historically, SSRIs have been analyzed using ion-pairing chromatography (IPC) on alkyl stationary phases (e.g., C18). IPC is a good alternative when reversed phase chromatography (RPC) on hydrophobic alkyl phases cannot provide adequate separation. IPC has disadvantages, however, including artifactual peaks, slow column equilibrium, poor peak shape, and incompatibility with MS detection. Because of these downsides to IPC, we evaluated the use of polar stationary phases, including the Allure™ Basix and Ultra PFP phases, for analysis of SSRIs.

The chemical structures of SSRIs (Figure 1) reveals that these compounds are polar bases capable of ionic separations. To ensure complete ionization, a pH value approximately 2 units from an analyte's pKa should be used. SSRIs have high pKa values (fluvoxamine maleate: 8.7, fluoxetine: 9.1, sertraline HCl: 9.5), however, and two pH units above these analytes' pKa values will be outside the acidic to neutral operating range for silica-based columns. Because SSRIs are basic, their retention can be increased by increasing the mobile phase pH. According to acid-base equilibria, as pH decreases, bases gain a proton (ionize), making them more hydrophilic and less retained by RPC. Thus, the greatest retention of SSRIs would occur at neutral pH, rather than at an acidic pH.

At neutral pH, an Allure™ Basix column shows good retention, selectivity, and peak shape for SSRIs (Figure 2). This stationary phase and pH are a good choice if optimum retention and selectivity are desired. Adding an amine modifier can alter selectivity and improve peak shape (Figure 3). As the concentration of amine modifier is increased, the retention of basic analytes decreases, and the peaks sharpen. This could be an effective way to produce alternate selectivity and enhance peak

Figure 1 Selective serotonin reuptake inhibitors (SSRIs) are a chromatographic challenge.

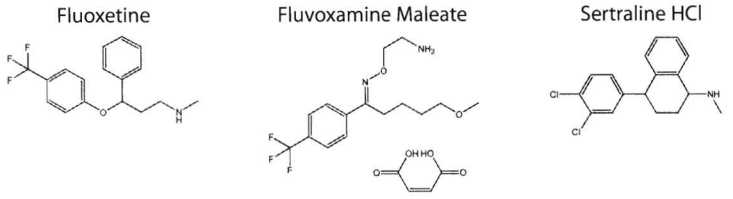
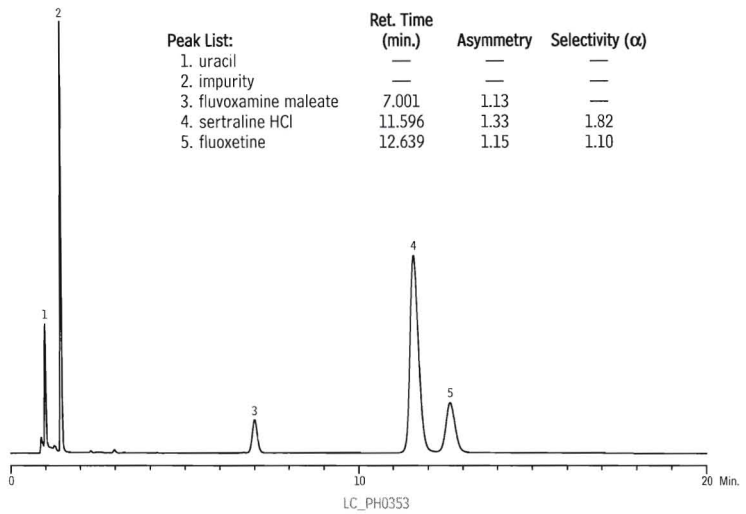


Figure 2 At neutral pH, an Allure™ Basix column provides good retention, selectivity, and peak shape for SSRIs.



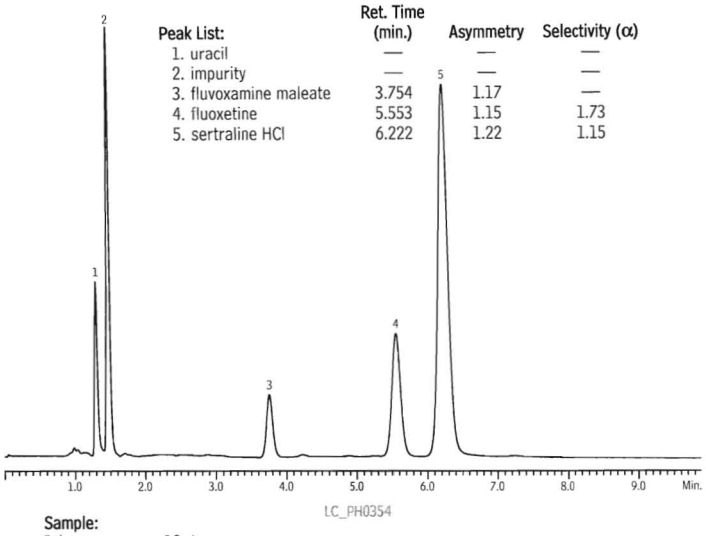
Peak List:	Ret. Time (min.)	Asymmetry	Selectivity (α)
1. uracil	—	—	—
2. impurity	—	—	—
3. fluvoxamine maleate	7.001	1.13	—
4. sertraline HCl	11.596	1.33	1.82
5. fluoxetine	12.639	1.15	1.10

Sample:
Inj.: 10µL
Conc.: 100µg/mL each component
Sample diluent: water:acetonitrile, 50:50

Column: Allure™ Basix (cat.# 9161565)
Dimensions: 150 x 4.6 mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: 20mM potassium phosphate dibasic in water (pH 7):acetonitrile, 40:60
Flow: 1mL/min.
Temp.: 30°C
Det.: UV @ 230 nm

Figure 3 An amine modifier improves peak shape and changes selectivity for SSRIs on an Allure™ Basix column.

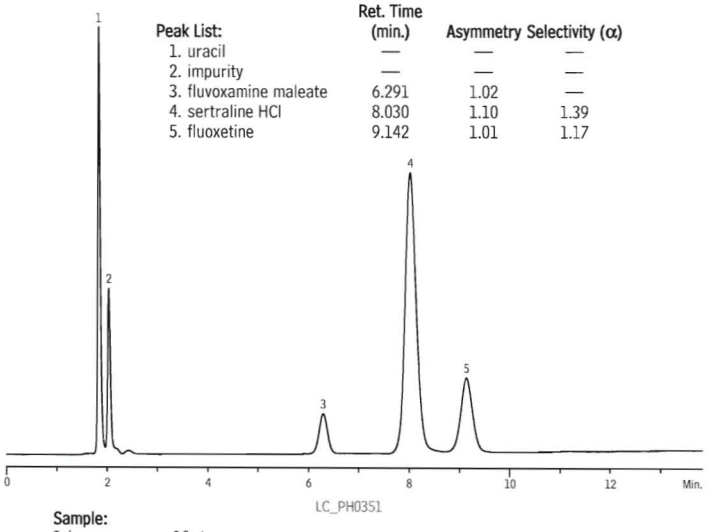


Sample:
Inj.: 10µL
Conc.: 100µg/mL each component
Sample diluent: water:acetonitrile, 50:50

Column: Allure™ Basix (cat.# 9161565)
Dimensions: 150 x 4.6 mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: 1% triethylamine in water (pH 6):acetonitrile, 50:50
Flow: 1mL/min.
Temp.: 30°C
Det.: UV @ 230 nm

Figure 4 An Ultra PFP column shows good performance at acidic pH.



Sample:
Inj.: 10µL
Conc.: 100µg/mL each component
Sample diluent: water:acetonitrile, 50:50

Column: Ultra PFP (cat.# 9176565)
Dimensions: 150 x 4.6 mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: 20mM potassium phosphate monobasic in water (pH 3):acetonitrile, 70:30
Flow: 1mL/min.
Temp.: 30°C
Det.: UV @ 230 nm

shape in reversed phase mode. However, amine additives work by blocking ionizable silanols, which can vary from column to column, so be sure the amine concentration is high enough to suppress all potential silanol effects.

The Ultra PFP phase shows the best performance at an acidic pH (Figure 4).

These analyses reveal that polar stationary phases can effectively replace IPC in analyses of SSRIs. Overall, in RPC, polar stationary phases provide better peak shape than alkyl phases for basic analytes. When analyzing SSRIs at neutral pH, the Allure™ Basix phase is a good choice. When analyzing SSRIs at an acidic pH, the Ultra PFP phase is the better candidate.

Allure™ Basix Column

5µm Column, 4.6mm	cat. #
150mm	9161565

Ultra PFP Column

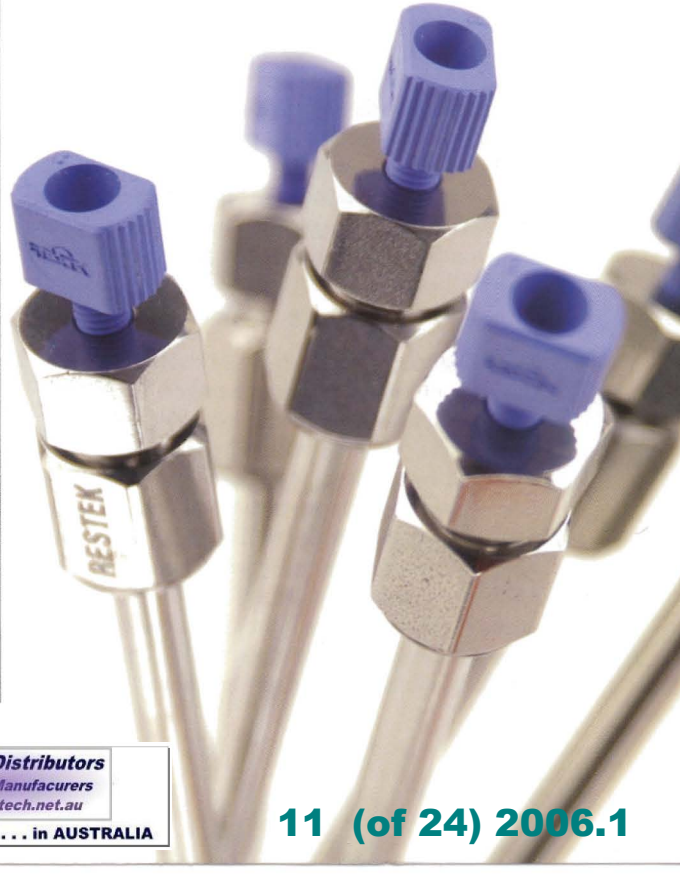
5µm Column, 4.6mm	cat. #
150mm	9176565

ordering note

To order a 2.1mm, 3.2mm, or 4.6mm ID column with a Trident™ Integral Inlet Fitting, add "-700" to the catalog number for the column.

Nominal additional charge

For guard cartridges and XG-XF guard cartridge fittings for these columns, visit our website at **www.restek.com**.



Assaying Tetracyclines by HPLC

Using the Allure™ Biphenyl Stationary Phase

By Rick Lake, Pharmaceutical Innovations Chemist, and Sherry Moyer, Innovations Chemist

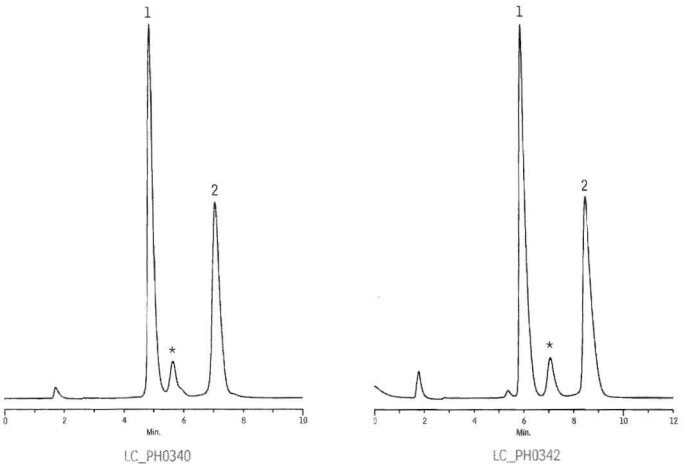
- Superior selectivity and efficiency, using an Allure™ Biphenyl column.
- Simplified analysis for high-throughput potency and stability-indicating assays.
- More easily achievable system suitability criteria.

Tetracyclines are a widely used class of antibiotics whose applications range from topical acne medications for humans to premix feed additives for livestock. Because of their widespread and liberal use, tetracyclines are manufactured in large quantities, which generates the need for a large number of potency and stability-indicating assays. These assays must be completed at regular intervals, in a timely manner, over extended periods of time. Consequently, it is critical that simple, rugged, and selective methods be developed. By selecting a stationary phase that produces optimum selectivity, less demand to produce selectivity is placed on the mobile phase, and a simple isocratic analysis is possible. Among the stationary phases we tested, the Allure™ Biphenyl and Allure™ PFP Propyl stationary phases showed the best performance (Table 1 and Figure 1).

Developing a simple mobile phase for this application was a major concern. Ideally, to achieve ionization equilibrium, choose a mobile phase pH 2 units from the analytes' pKa. But two units below the pKa values for the tetracyclines (approximately 3.3) would be below the recommended pH limit for traditional silica-based columns, pH 2. Consequently, we chose a pH of 2.5, and we added a buffer to maintain pH. Because tetracyclines form chelates with metal ions, we chose a non-metal organic salt - ammonium phosphate - and, to minimize surface metal content, we used only columns made from high-purity Type B silica. Lastly, we chose acetonitrile as the organic solvent, because of its eluting strength and limited effect on pKa: increasing the organic composition increases pKa for acidic analytes and decreases pKa for basic analytes, but a small amount of acetonitrile lessens the effect, relative to a larger amount of methanol.

We evaluated several silica-based stationary phases, using the mobile phase described above, UV detection, and isocratic conditions. The first selection criteria we used was selectivity, which we measured by analyzing oxytetracycline and tetracycline (an impurity in oxytetracycline formulations) and determining the USP resolution and selectivity (α) between the two compounds. The Allure™ Biphenyl and Allure™ PFP Propyl stationary phases showed the best performance among the columns we tested (Table 1 and Figure 1). These results suggest that the Allure™ Biphenyl

Figure 1 Tetracycline and oxytetracycline have low pKa values that make the analysis a challenge.



Peak List	Ret. Time (min.)
1. oxytetracycline	4.92
* degradation peak	5.66
2. tetracycline	7.10

Column:	Allure™ Biphenyl
Cat. #:	9166565
Dimensions:	150 x 4.6 mm
Particle size:	5µm
Pore size:	60Å

Peak List	Ret. Time (min.)
1. oxytetracycline	5.94
* degradation peak	7.08
2. tetracycline	8.57

Column:	Allure™ PFP Propyl
Cat. #:	9169565
Dimensions:	150 x 4.6 mm
Particle size:	5µm
Pore size:	60Å

Sample:

Inj.:	20µL (Allure™ Biphenyl column) or 30µL (Allure™ PFP Propyl column)
Conc.:	100µg/mL each component
Sample diluent:	methanol

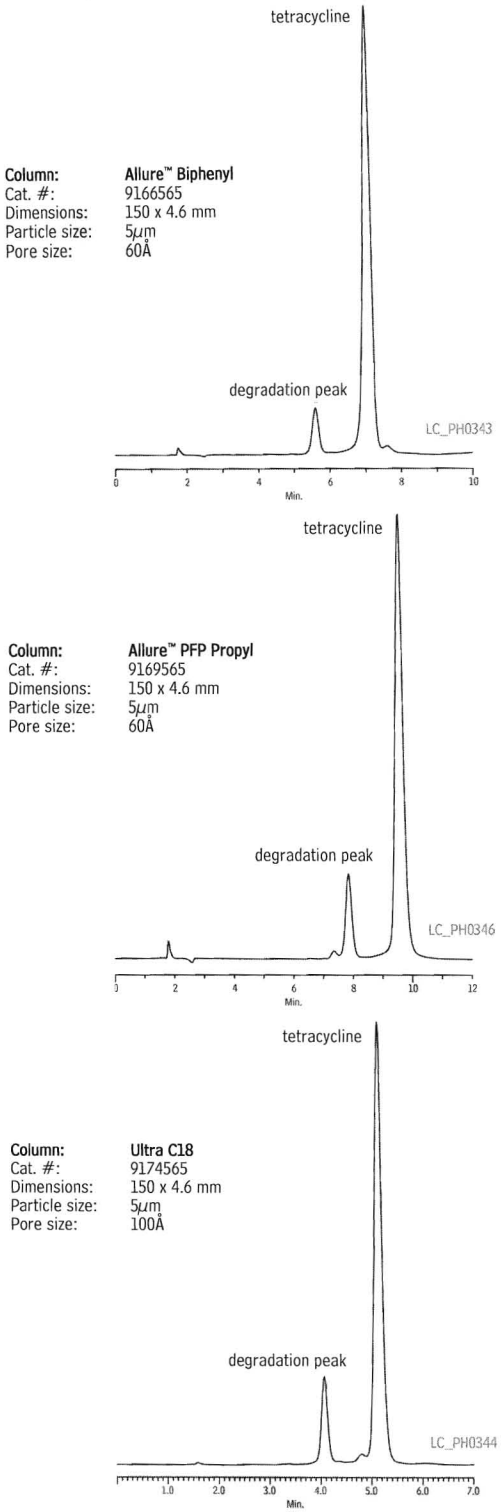
Conditions:

Mobile phase:	20mM ammonium phosphate (pH 2.5):acetonitrile, 80:20
Flow:	1mL/min.
Temp.:	ambient
Det.:	UV @ 254 nm

Table 1 Among tested columns, Allure™ Biphenyl and Allure™ PFP Propyl columns show the best combination of resolution and selectivity for tetracycline and oxytetracycline.

Stationary Phase	USP Resolution	Selectivity (α)
Allure™ Biphenyl	5.28	1.61
Allure™ PFP Propyl	4.49	1.59
Ultra C18	3.31	1.50
Allure™ Basix	NA	1.34
Ultra C8	NA	0.47
Ultra PFP	NA	NA

Figure 2 Overall, the Allure™ Biphenyl column is the best choice for assaying the tetracycline antibiotics.



Sample:
Inj.: 5µL
Conc.: 100µg/mL each component
Sample diluent: methanol

Conditions:
Mobile phase: 20mM ammonium phosphate (pH 2.5):acetonitrile, 80:20
Flow: 1mL/min.
Temp.: ambient
Det.: UV @ 254 nm

Table 2 Allure™ Biphenyl, Allure™ PFP Propyl, and Ultra C18 columns provide excellent repeatability.

	Peak Area	Retention Time (min.)	Capacity Factor (k')	USP Tailing
Allure™ Biphenyl	best choice for tetracycline antibiotics			
Mean	2509475	7.08	5.68	1.06
Std. Dev.	36397.39	0.02	0.03	0.01
%RSD	1.45	0.36	0.45	0.49
Allure™ PFP Propyl				
Mean	2483972	9.52	8.12	1.28
Std. Dev.	22202.94	0.04	0.04	0.01
%RSD	0.89	0.45	0.53	0.43
Ultra C18				
Mean	2399803	5.13	3.73	1.21
Std. Dev.	21171.76	0.02	0.02	>0.00
%RSD	0.88	0.33	0.45	0.34

stationary phase exhibits π-π bonding with the ring structures of the tetracyclines, and the embedded polarity of the fluorinated Allure™ PFP Propyl phase interacts with tetracycline moieties. Either of these separation mechanisms increases retention, compared to a mechanism based on hydrophobicity, as exhibited by the alkyl chain of a C18 phase.

Tetracycline drug products are produced under cGMP protocols and, therefore, manufacturers are required to use validated or compendial methods, either of which require the completion of system suitability criteria (e.g., tailing factors, capacity factors, and repeatability). Consequently, we further evaluated the three stationary phases that produced the best initial results, using system suitability criteria, by assaying tetracycline.

Overall, all three columns provided excellent repeatability (Table 2). The Allure™ PFP Propyl column exhibited the greatest retention and capacity for the analytes, but exhibited the highest degree of peak tailing under these conditions (Figure 2). The Ultra C18 column also exhibited a high degree of peak tailing, and the weakest analyte retention (Figure 2). Altering the mobile phase likely would improve peak shape, but capacity factors would suffer accordingly. The Allure™ Biphenyl column proved to be the best overall choice for the tetracyclines - it exhibited good capacity, high selectivity, and the least peak tailing (Figure 2).

By selecting the stationary phase that provides the best selectivity and efficiency for tetracycline analytes - the Allure™ Biphenyl phase - analysts can exercise more control over separation and other method conditions, ultimately creating a simple, rugged, and selective method.

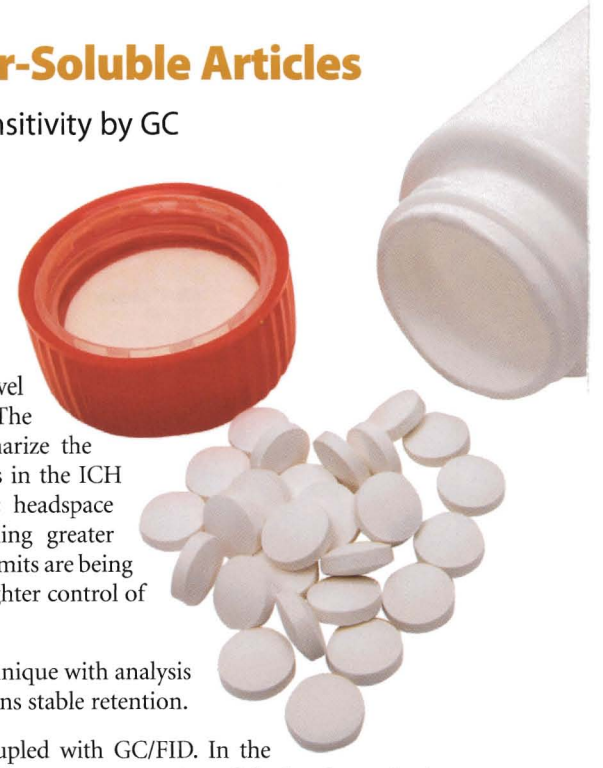
Allure™ Biphenyl Column

5µm Column, 4.6mm	cat. #
150mm	9166565

Analyzing Residual Solvents in Water-Soluble Articles

Dynamic Headspace Sampling Enhances Sensitivity by GC

By Rick Lake, Pharmaceutical Innovations Chemist



- Sensitivity increased 13X-30X for residual solvents (OVIs) in water.
- Excellent resolution and stable retention times, using an Rtx®-G43 column.
- Greater sensitivity makes smaller samples possible.

Residual solvents, or organic volatile impurities (OVIs), in pharmaceuticals are trace-level leftover solvents that were used in the manufacture of drug products or excipients. The International Conference on Harmonization (ICH) provides guidelines that summarize the allowable concentrations of common solvents. However, some of the detection limits in the ICH guidelines are not easily achieved through the normal sampling technique, static headspace analysis, and pharmaceutical manufacturers are becoming concerned with attaining greater sensitivity. As more toxicity data become available, maximum allowable concentration limits are being lowered. And, as active ingredient and excipient markets are becoming more global, tighter control of impurities is needed.

In our investigations, we have found that coupling a dynamic headspace sampling technique with analysis on an Rtx®-G43 column greatly increases sensitivity for residual solvents, and maintains stable retention.

Analyses for residual solvents typically are performed using headspace sampling coupled with GC/FID. In the commonly used static headspace technique, a pressurized or ballast loop system is used to extract a portion of the headspace in the sample vial for introduction into the GC. Another, more novel, technique for headspace sampling is the dynamic headspace technique. In this technique, the entire content of the vial headspace is swept onto an activated trap, which collects and concentrates the target analytes, then desorbs the analytes into the GC carrier flow. Dynamic headspace increases the sensitivity of the analysis, but high concentrations of organic solvents will cause contamination and lifetime problems with the trap and, therefore, this technique is not compatible with the use of organic solvents as diluents for water-insoluble articles. On the other hand, the technique is well suited to, and easily performed in, analyses of residual solvents in water-soluble articles.

We evaluated the sensitivity of the static and dynamic headspace techniques, using solvents in an aqueous matrix, to compare responses as they might relate to pharmaceutical analysis of residual solvents in water-soluble articles. We prepared reference standards containing the USP467 solvents at their regulatory limits in water, by adding 100µL of our USP 467 Calibration Mix #5 (cat.# 36007) to 5mL of deionized water in a 22mL headspace sampling vial. We also added approximately 1 gram of an inorganic salt, sodium sulfate, to each sample to decrease the solubility of polar compounds. This is critical for highly water-soluble volatiles, like 1,4-dioxane, as it promotes analyte transfer into the gaseous phase in the sample vial.

First, we used a traditional static headspace (loop) technique to assay a system suitability set comprised of 6 replicates (Figure 1A). The sample vial was heated, mixed, and pressurized. A six-port valve was used to fill a specified loop volume with an aliquot of the headspace, then the valve was switched to redirect the gas flow, flushing the sample into the transfer line and ultimately mixing with the GC carrier gas flow. Next, we used a dynamic headspace (trap) technique to analyze an equivalent 6-replicate system suitability set (Figure 1B). The sample vial was heated and mixed under the same conditions as used in the loop method, then a gas flow was introduced into the headspace

Table 1 Dynamic headspace sampling greatly increases sensitivity for OVIs.

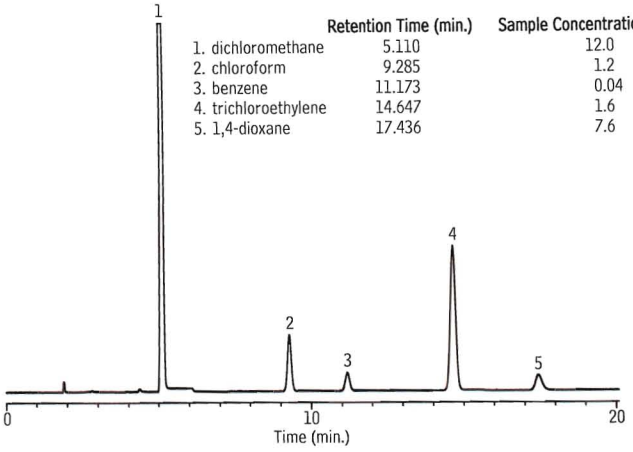
Analyte	Sample Conc. (ppm)	Concentration at Regulatory Limit (ppm)	Mean Peak Area Response		Increase in Sensitivity with Dynamic Headspace
			Static Headspace	Dynamic Headspace	
dichloromethane	12.0	600	619	18679	30X
chloroform	1.2	60	39	783	20X
benzene	0.04	2	15	313	21X
trichloroethene	1.6	80	141	3479	25X
1,4-doxane	7.6	380	20	272	13X

Table 2 Solvent retention times and resolution are equivalent for static or dynamic headspace sampling and analysis on an Rtx®-G43 column.

Solvent		Static Headspace		Dynamic Headspace	
		Retention Time (min.)	Resolution	Retention Time (min.)	Resolution
dichloromethane	Mean	5.092	—	5.139	—
	Std. Dev.	0.01	—	>0.00	—
	%RSD	0.25	—	0.04	—
chloroform	Mean	9.250	23.02	9.263	22.18
	Std. Dev.	0.02	0.26	>0.00	0.07
	%RSD	0.23	1.11	0.04	0.31
benzene	Mean	11.134	7.67	11.145	7.72
	Std. Dev.	0.03	0.08	>0.00	0.01
	%RSD	0.23	1.04	0.03	0.11
trichloroethene	Mean	14.592	11.87	14.599	11.86
	Std. Dev.	0.03	0.06	>0.00	0.01
	%RSD	0.23	0.46	0.04	0.10
1,4-dioxane	Mean	17.388	7.91	17.411	—
	Std. Dev.	0.04	0.10	0.09	—
	%RSD	0.20	1.23	0.50	—

Figure 1 USP 467 solvents by headspace analysis/GC on an Rtx®-G43 column.

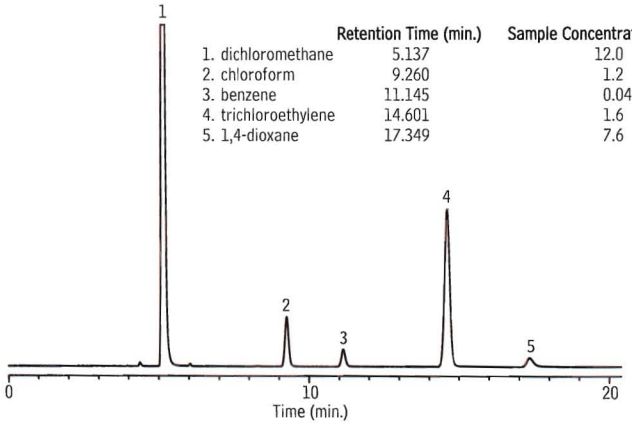
A) Static headspace (loop) technique



	Retention Time (min.)	Sample Concentration (µg/ml)
1. dichloromethane	5.110	12.0
2. chloroform	9.285	1.2
3. benzene	11.173	0.04
4. trichloroethylene	14.647	1.6
5. 1,4-dioxane	17.436	7.6

Instrument:	Teledyne Tekmar HT3	Platen/Sample Temp.:	80°C
Sample Equilibr. Time:	15.00 min.	Platen Temp. Equilibr. Time:	2.00 min.
Mixing:	level 5, 2.00 min.	Pressurize:	15 psi, 2.00 min.
Mixer Stabilize Time:	0.50 min.	Pressurize Equilibr. Time:	0.50 min.
Valve Oven Temp.:	150°C	Loop Fill Pressure:	5 psi
Transfer Line Temp.:	150°C	Loop Fill Time:	2.00 min.
Standby Flow Rate:	10mL/min.	Loop Fill Equilibr. Time:	0.50 min.
		Inject Time:	1.00 min.

B) Dynamic headspace (trap) technique



	Retention Time (min.)	Sample Concentration (µg/ml)
1. dichloromethane	5.137	12.0
2. chloroform	9.260	1.2
3. benzene	11.145	0.04
4. trichloroethylene	14.601	1.6
5. 1,4-dioxane	17.349	7.6

Instrument:	Teledyne Tekmar HT3	Trap Standby Temp.:	40°C
Sample Equilibr. Time:	15.00 min.	Platen/Sample Temp.:	80°C
Mixing:	level 5, 2.00 min.	Sweep Flow:	75mL/min., 3.00 min.
Mixer Stabilize Time:	0.50 min.	Dry Purge:	50mL/min., 5.00 min., 25°C
Valve Oven Temp.:	150°C		
Transfer Line Temp.:	150°C	Desorb Preheat:	245°C
Standby Flow Rate:	10mL/min.	Desorb:	1.00 min., 250°C
		Trap Bake:	450mL/min., 6.00 min., 300°C

Chromatography Conditions

Column:	Rtx®-G43 30m, 0.53mm ID, 3.0µm (cat.# 16085-126)
Sample:	100µL USP 467 Calibration Mixture #5 (cat.# 36007) in dimethylsulfoxide, 5mL water, ~1.0g sodium sulfate in 22mL headspace vial. Concentrations listed on figure.
Inj.:	static headspace or dynamic headspace
Inj. temp.:	180°C
Carrier gas:	helium, split 2:1
Linear velocity:	5mL/min., constant flow
Oven temp.:	40°C (20 min.), to 240°C @ 25°C/min. (hold 10 min.)
Det.:	FID @ 250°C hydrogen flow: 40mL/min.; air flow: 450mL/min.; make-up flow: 45mL/min.

of the vial, to sweep the analytes onto an activated trap. The trap, with the concentrated analytes, was dry purged to remove the water vapor, then was heated without flow to desorb the analytes. After the analytes were desorbed, the trap was back-flushed to direct the concentrated analytes onto the analytical column. Between analyses, the trap was baked at high temperature to remove all residue compounds.

When we compared the results of the system suitability analyses for the two headspace techniques, we determined that, based on area responses, the dynamic headspace method greatly enhanced sensitivity for the target OVIs: area counts were, on average, 22 times larger than for the static headspace method (Table 1). We also noted that the Rtx®-G43 capillary column provided excellent resolution among analytes, with very little drift in retention time or resolution (Table 2).

As with purge and trap systems, or other dynamic sampling systems, certain system controls must be taken into account when using a dynamic headspace technique. Factors to consider include sweeping and desorbing times and flows, adsorbent materials used to trap the analytes, and water management. In this specific application, we observed that either prolonged sample heating at 80°C or extended vial sweep times increased the water content in the sample headspace, ultimately resulting in poor peak shape for 1,4-dioxane and, if excessive, extinguishing the FID. 1,4-Dioxane has a notoriously poor partitioning efficiency and proved to be the limiting factor when setting system operating conditions. For samples heated at 80-85°C in a water matrix, a sweep time of 5 minutes or less enhanced sensitivity for all compounds while assuring proper water management.

From this work, we conclude that coupling a dynamic headspace sampling technique with analysis on an Rtx®-G43 column greatly increases sensitivity for residual solvents, and makes stable retention possible. These enhancements can lead to more achievable system suitability criteria and lower detection limits, or to effective results with smaller samples.

Rtx®-G43 Column
(fused silica with 5-meter Integra-Guard™)
(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.53mm	3.00	-20 to 240°C	30-Meter	16085-126

trans Fat: Resolving *cis* and *trans* FAME Isomers by GC

By Julie Kowalski, Innovations Chemist

- Highly polar Rt-2560 column resolves individual *cis* and *trans* FAME isomers.
- Analytical reference mixes for quantifying FAMEs in foods and dietary supplements.
- Use column and reference mixes to meet new *trans* fat labeling regulations.

Concern over the detrimental effects of diets high in *trans* fats has prompted the US Food and Drug Administration (FDA) to require *trans* fat content to be reported separately on food labels after January 2006. The FDA estimates that by 2009 this rule will save $900 million to $1.8 billion per year in medical costs and lost productivity. The monetary savings will far more than offset the FDA-estimated $140-250 million in one-time costs of determining amounts of *trans* fats, revising Nutrition Facts panels, and voluntarily reducing amounts of *trans* fats[1] that the food industry will incur to comply with the rule.

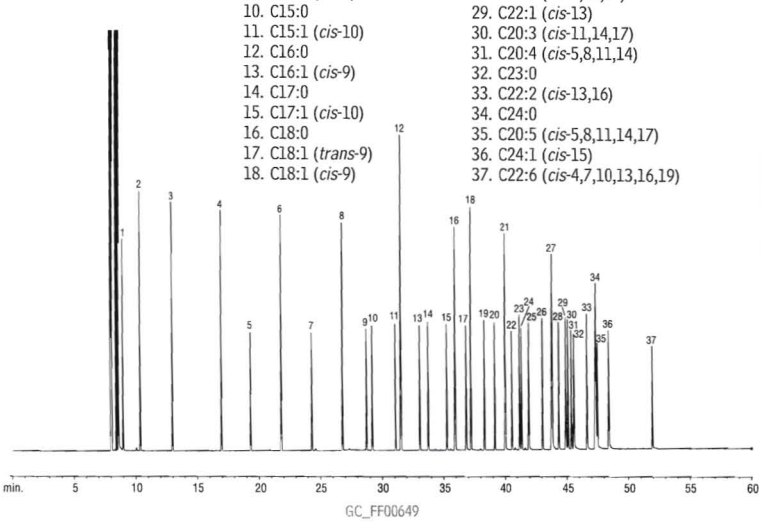
The highly polar Rt-2560 biscyanopropyl stationary phase has the selectivity needed for resolving *cis* and *trans* FAME isomers to comply with the FDA guidelines. Individual *cis* and *trans* isomers are resolved on a 100-meter Rt-2560 GC column (cat.# 13199), making this the column of choice for analyzing partially hydrogenated fats. The *trans* isomers elute before the *cis* isomers (Figure 1), a reverse of the elution order on Carbowax®-based phases such as FAMEWAX™ or Rtx®-Wax. AOAC method 996.06[2] specifies the determination of total fat content based on the fatty acid content after conversion of the fatty acids to the methyl esters, and is the accepted analytical method for determining total fat content for nutritional labeling. A 100-meter Rt-2560 column meets the requirements of this procedure, and also allows quantification of the total *trans* fat content.

To calibrate the GC system for these assays, we recommend a carefully formulated FAME mixture, such as our 37-component Food Industry FAME Mix (cat.# 35077, Figure 1) or our 28-component NLEA FAME Mix (cat.# 35078). Each of these mixes includes a gravimetric certificate of analysis to help ensure accurate quantification. To ensure correct identifications of individual C18:1 *cis* or *trans* isomers, use our *cis*/*trans* FAME Mix (cat.# 35079), as shown in Figure 1.

An Rt-2560 column is the column of choice when determining *trans* fat content and total fat content in food products. Whatever your fatty acid analysis requirements, Restek can provide the consistent-performance analytical columns and reference materials that will help you to accurately characterize your materials.

Figure 1 Food Industry FAME Mix (cat.# 35077) resolved on an Rt-2560 column.

Compound		
1. C4:0	14. C17:0	27. C22:0
2. C6:0	15. C17:1 (*cis*-10)	28. C20:3 (*cis*-8,11,14)
3. C8:0	16. C18:0	29. C22:1 (*cis*-13)
4. C10:0	17. C18:1 (*trans*-9)	30. C20:3 (*cis*-11,14,17)
5. C11:0	18. C18:1 (*cis*-9)	31. C20:4 (*cis*-5,8,11,14)
6. C12:0	19. C18:2 (*trans*-9,12)	32. C23:0
7. C13:0	20. C18:2 (*cis*-9,12)	33. C22:2 (*cis*-13,16)
8. C14:0	21. C20:0	34. C24:0
9. C14:1 (*cis*-9)	22. C18:3 (*cis*-6,9,12)	35. C20:5 (*cis*-5,8,11,14,17)
10. C15:0	23. C20:1 (*cis*-11)	36. C24:1 (*cis*-15)
11. C15:1 (*cis*-10)	24. C18:3 (*cis*-9,12,15)	37. C22:6 (*cis*-4,7,10,13,16,19)
12. C16:0	25. C21:0	
13. C16:1 (*cis*-9)	26. C20:2 (*cis*-11,14)	



Rt-2560, 100m, 0.25mm ID, 0.2µm (cat.# 13199)
Sample: Food Industry FAME Mix (cat.# 35077), 30mg/mL total FAMEs in methylene chloride
Inj.: 2.0µL split (split ratio 200:1), 4mm inlet liner (cat.# 20814)
Inj. temp.: 225°C
Carrier gas: hydrogen, constant flow
Flow rate: 1.2mL/min.
Oven temp.: 100°C (4 min. hold) to 240°C @ 3°C/min. (10 min. hold)
Det.: FID @ 250°C

References
[1] *http://www.cfsan.fda.gov/~dms/qatrans2.html#s5q1*
[2] *Official Methods of Analysis, 17th edition, AOAC International, 2000.*

free literature

High Resolution Analyses of Fatty Acid Methyl Esters (FAMEs) by Gas Chromatography

lit. cat.# 59584A

Free on request from Restek, or from your Restek distributor.

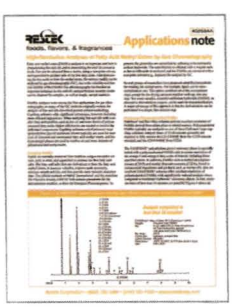
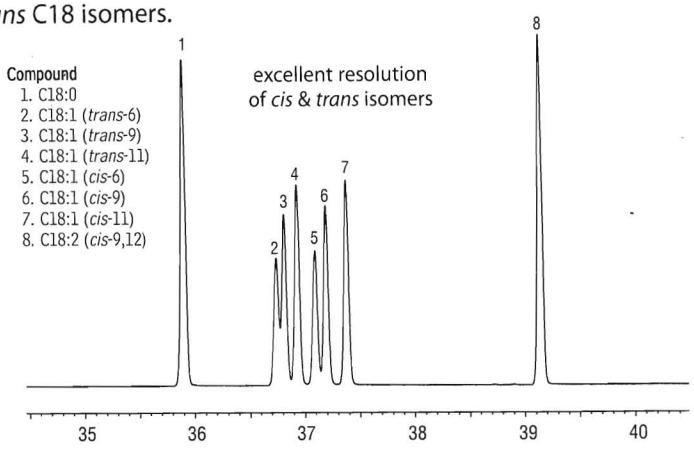


Figure 2 An Rt-2560 column resolves *cis* C18 FAME isomers from *trans* C18 isomers.



Rt-2560, 100m, 0.25mm ID, 0.2μm (cat.# 13199)
Sample: *cis/trans* FAME Mix (cat.# 35079), 10mg/mL total FAMEs in methylene chloride
Inj.: 1.0μL split (split ratio 20:1), 4mm inlet liner (cat.# 20814)
Inj. temp.: 225°C
Carrier gas: hydrogen, constant flow
Flow rate: 1.2mL/min.
Oven temp.: 100°C (4 min. hold) to 240°C @ 3°C/min. (10 min. hold)
Det.: FID @ 250°C

Rt-2560 column (fused silica)

(biscyanopropyl polysiloxane)

ID	df (μm)	temp. limits	length	cat. #
0.25mm	0.20	20 to 250°C	100-Meter	13199

Food Industry FAME Mix (37 components)
30mg/mL total in methylene chloride, 1mL/ampul
cat. # 35077 (ea.)

NLEA FAME Mix (28 components)
30mg/mL total in methylene chloride, 1mL/ampul
cat. # 35078 (ea.)

cis/trans **FAME Mix** (8 components)
10mg/mL total in methylene chloride, 1mL/ampul
cat. # 35079 (ea.)

For analysis of NLEA FAME Mix on an Rt-2560 column, please refer to our 2006 catalog or visit our website.

Genuine Restek Replacement Parts for Shimadzu HPLC Systems

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

- Keep your Shimadzu HPLC systems in top condition!
- All parts designed to meet or exceed original equipment performance.
- New items constantly being added - check our website for our complete HPLC product offering.

Restek Replacement Parts for Shimadzu HPLC Systems

Description		Model #	Similar to Shimadzu part #	qty.	cat.#
Inlet Check Valve		LC-6A, LC-10AS	228-12353-91	ea.	25287
Inlet Check Valve		LC-600, LC-9A, LC-10AD	228-18522-91	ea.	25295
Inlet Check Valve	new!	LC-10ADvp	228-39093-92	ea.	24984
Outlet Check Valve		LC-6A, LC-10AS	228-09054-93	ea.	25288
Outlet Check Valve Rebuild Kit		LC-6A, LC-10AS	228-11200-91	2-pk.	25289
Outlet Check Valve		LC-600, LC-9A, LC-10AD	228-18522-92	ea.	25282
Outlet Check Valve	new!	LC-10ADvp, LC-10ATvp	228-34976-91	ea.	24983
Plunger Seal		LC-6A	228-11999-00	ea.	25285
Plunger Seal, Polyethylene		LC-10AS	228-21975-00	ea.	25290
Plunger Seal		LC-600, LC-9A, LC-10AD	228-18745-00	ea.	25293
Plunger Seal	new!	LC-10ADvp	228-35146-00	ea.	24980
Plunger Seal, Gold	new!	LC-10ADvp	228-32628-00	ea.	24981
Plunger Seal	new!	SIL-10ADvp, LC-10ATvp	228-35145-00	ea.	24985
Plunger Rinse Seal		LC-10AS	228-28499-00	ea.	25292
Sapphire Plunger		LC-6A	228-12904-93	ea.	25286
Sapphire Plunger		LC-10AS	228-17019-93	ea.	25291
Sapphire Plunger		LC-600, LC-9A, LC-10AD	228-18523-91	ea.	25294
Needle Seal		SIL-10A, 10XL, 10ADvp	228-33355-04	ea.	25468
Rotor Seal	new!	SIL-10ADvp	228-21217-97	ea.	24986
Rotor Seal Assembly		SIL-10A, 10AXL	228-21217-91	ea.	25469
Stator Assembly		SIL-10A, 10AXL	228-21220-91	ea.	25470
Syringe, 500μL		SIL-10A, 10AXL	228-25237-04	ea.	25471
Plunger Assembly, Ceramic		LC-10ADvp	228-35601-91	ea.	25472
Plunger Assembly, Ceramic		LC-10ATvp	228-35009-92	ea.	25473
Plunger Assembly, Sapphire	new!	LC-10ADvp	228-35601-92	ea.	24982
Deuterium Lamp		SPD-6A	062-65056-02	ea.	25283
Deuterium Lamp		SPD-10A, 10AV	228-34016-02	ea.	25284

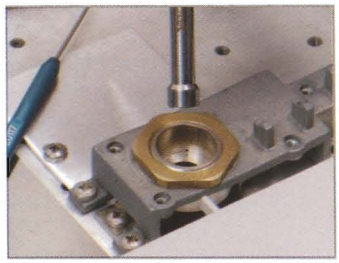
Cool Tools!

Restek Innovations Save You Time and Money

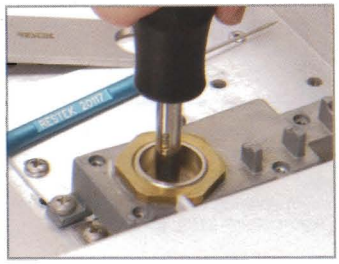
FID Jet Removal Tool for Agilent 5890/6890/6850 FIDs

- Securely grips jet in socket for easy removal or installation.
- Unique, ergonomic handle—easy to hold.

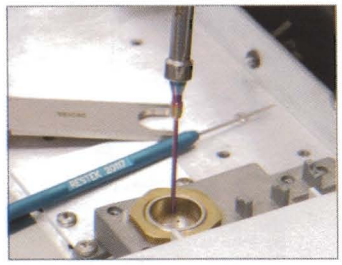
restek innovation!



Slip tool over FID jet...



loosen jet...



and remove.

Description	qty.	cat.#
FID Jet Removal Tool for Agilent 5890/6890/6850 FIDs	ea.	22328

Septum Nut Removal Tool for Agilent 5890/6890/6850 GCs

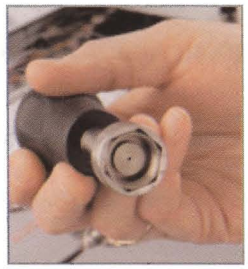
- Easily remove the septum nut without touching the heated nut—no more burned fingers!
- Unique, ergonomic handle—easy to grip.

restek innovation!

Slip tool over septum nut...

loosen nut...

and remove, avoiding hot metal surfaces.



Septum nut remains in tool until reinstalled.

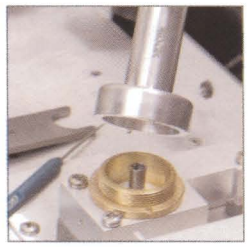
Description	qty.	cat.#
Septum Nut Removal Tool for Agilent 5890/6890/6850 GCs	ea.	24918

Spanner Wrench for Agilent 5890/6890/6850 FID Collector Assembly

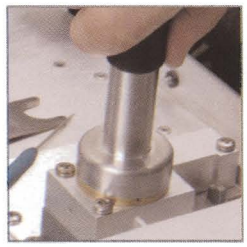
- Easily remove the nut from the FID collector without damaging the nut.
- Unique, ergonomic handle—easy to grip.

restek innovation!

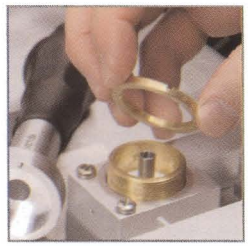
Remove FID ignitor castle.



Easily loosen nut by aligning two pins on bottom of wrench with two open slots on nut...



turn counterclockwise...



and remove.

Description	Replaces Agilent part #	qty.	cat.#
Spanner Wrench for Agilent 5890/6890/6850 FID Collector Assembly	19231-00130	ea.	22329

Headspace Vials;
Hand-held, Rechargeable, Crimpers and Decappers

By Donna Lidgett, GC Accessories Product Marketing Manager

Headspace Autosampler Vials

Description	100-pk.	1000-pk.
6mL Clear Vial	21166	21167
10mL Clear Vial, Flat Bottom	24683	24684
10mL Clear Vial, Rounded Bottom	21164	21165
20mL Clear Vial, Flat Bottom	24685	24686
20mL Clear Vial, Rounded Bottom	21162	21163
27mL Clear Vial	21160	21161

20mm Aluminum Seals w/Septa, Assembled

Description	100-pk.	1000-pk.
Silver Seal w/ PTFE/Gray Butyl Rubber	21761	21762
Silver Seal w/ PTFE/Silicone	21763	21764
Pressure Release Silver Seal w/ PTFE/Gray Butyl Rubber Septum	21765	21766
Pressure Release Silver Seal w/ PTFE/Silicone Septum	21767	21768

Hand-held Rechargeable Crimpers and Decappers

new!

- Easy to use; comfortable grip.
- Hundreds of operations from one charge.
- Adjustable crimping force.

Powered by a standard Black & Decker Versapak® battery, these electronic tools for 11mm or 20mm caps will cycle hundreds of times on a single charge. The cycle is controlled precisely, through an internal counter, and is adjusted with two buttons on the side of the case. The tools fit comfortably in the hand and weigh approximately 600 grams. The jaws can be positioned easily around closely-spaced vials in standard autosampler trays. Each kit includes the tool, a Versapak® Gold rechargeable battery, which uses environmentally friendly NiMH technology, and a charger. Recharging generally takes 6-9 hours.

Decapping has never been easier!

one...

two...

three!

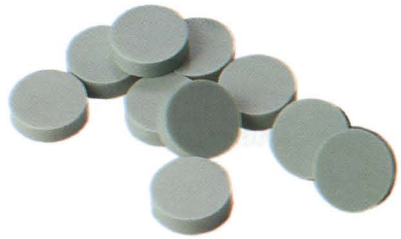
The Electronic Crimper fits around vials in standard autosampler trays. The adjustable setting provides a precision crimp, vial after vial.

Description	qty.	cat.#
11mm Electronic Crimper (110 volt battery charger)	kit	22853
11mm Electronic Crimper (220 volt European battery charger)	kit	22853-EUR
11mm Electronic Crimper (220 volt UK battery charger)	kit	22853-UK
11mm Electronic Decapper (110 volt battery charger)	kit	22854
11mm Electronic Decapper (220 volt European battery charger)	kit	22854-EUR
11mm Electronic Decapper (220 volt UK battery charger)	kit	22854-UK
20mm Electronic Crimper (110 volt battery charger)	kit	22851
20mm Electronic Crimper (220 volt European battery charger)	kit	22851-EUR
20mm Electronic Crimper (220 volt UK battery charger)	kit	22851-UK
20mm Electronic Decapper (110 volt battery charger)	kit	22852
20mm Electronic Decapper (220 volt European battery charger)	kit	22852-EUR
20mm Electronic Decapper (220 volt UK battery charger)	kit	22852-UK

Peak Performers

Avoid Septum Problems

By Donna Lidgett, GC Accessories Product Marketing Manager



- Handle septa carefully, to prevent contamination.
- Use low-bleed septa.

handy septum size chart

Instrument	Septum Diameter (mm)
Agilent (HP)	
5880A, 5890, 6890, 6850, PTV	11
5700, 5880	9.5/10
On-Column Injection	5
CE Instruments (TMQ)	
TRACE™ GC	17
Finnigan (TMQ)	
GC 9001	9.5
GCQ	9.5
GCQ w/TRACE™, PTV	17
QCQ™	9.5
TRACE™ 2000	9.5
Fisons/Carlo Erba (TMQ)	
8000 series	17
Gow-Mac	
6890 series	11
All other models	9.5
PerkinElmer	
Sigma series	11
900,990	11
8000 series	11
Auto SYS™	11
Auto SYS™ XL	11
Pye/Unicam	
All models	7
Shimadzu	
All models	Plug
SRI	
All models	Plug
Tracor	
540	11.5
550,560	9.5
220,222	12.5
Varian	
Injector type:	
Packed column	9.5/10
Split/splitless	
1078/1079	10/11
1177	9
1075/1077	11

did you know?

Restek's new Thermolite® and IceBlue™ septa are now precision molded to ensure consistent, accurate fit.

Septum Handling

All septa, regardless of their composition, puncturability, or resistance to thermal degradation, will be a source of problems if they are mishandled. Always use clean forceps or wear clean cotton gloves when handling septa; do not handle them with bare fingers, nor with powdered latex gloves—contaminants such as finger oils, perfumes, make-up, fingernail polish, skin creams, hand soaps, and talcum can be absorbed into the septum and will bleed from the septum during your analyses.

Also, follow septum and instrument manufacturers' recommendations when installing a septum. Overtightening a septum nut invariably will reduce septum lifetime by increasing septum coring and splitting problems.

Septum Bleed

All septa contain various amounts of volatile materials (e.g., silicone oils, phthalates) that can be released when the septum is heated to analysis temperatures. Septum bleed occurs when these volatiles from the septum collect on the column, then elute from the column and create baseline disturbances or extraneous (ghost) peaks in the chromatogram. This problem is prevalent in temperature-programmed analyses, because the septum volatiles collect on the column during the oven cool-down and initial hold periods. Capillary columns require much lower gas flow rates than packed columns, therefore septum volatiles are more concentrated, and bleed problems are more pronounced in capillary GC systems.

Why are Low-Bleed Septa Important?

Either baseline rise or extraneous peaks caused by septum bleed can interfere with identification and quantification of target analytes. And, because septum bleed is inconsistent, method reproducibility can be a problem. Using low-bleed septa can minimize these effects and help produce more reliable results.

Why Does Septum Puncturability Matter?

A septum that can be penetrated cleanly and easily by a syringe needle has a longer life, and consistent injections made through such a septum help ensure accurate results. The soft silicone rubber from which all Restek septa are manufactured is specially formulated for chromatographic performance, which ensures our septa are easy to puncture.

What Septum Configurations are Available, and for Which GCs?

Restek has fashioned septa for all major brands of gas chromatographs and injectors. Use the septum size chart to determine the septum diameter for your instrument or, contact us.

Which Septa Should I Use?

Thermolite® septa are a proven low-bleed champion. With a maximum temperature of 340°C, there are very few applications for which Thermolite® septa are not suitable.

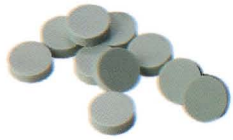
IceBlue™ septa are ideal for analysts using inlet temperatures of 250°C or below, or using solid phase microextraction (SPME) sampling techniques. IceBlue™ septa will accommodate puncturing from the large needles used in SPME, and still assure consistent injections and long lifetime.

BTO® septa are bleed and temperature optimized with a maximum temperature of 400°C, for the most demanding GC and GC/MS applications. They retain remarkable softness and pierceability at high temperatures. The CenterGuide™ can help reduce coring when used with tapered (rounded-tip) needles.

Restek Septa

- Precision molding assures consistent, accurate fit.
- Ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.

Septum Diameter	25-pk.	50-pk.	100-pk.
Thermolite® Septa			
5mm (3/16")	27120	27121	27122
6mm (1/4")	27123	27124	27125
7mm	27126	27127	27128
8mm	27129	27130	27131
9mm	27132	27133	27134
9.5mm (3/8")	27135	27136	27137
10mm	27138	27139	27140
11mm (7/16")	27141	27142	27143
11.5mm	27144	27145	27146
12.5mm (1/2")	27147	27148	27149
17mm	27150	27151	27152
Shimadzu Plug	27153	27154	27155
IceBlue™ Septa			
9mm		27156	27157
9.5mm (3/8")		27158	27159
10mm		27160	27161
11mm (7/16")		27162	27163
11.5mm		27164	27165
12.5mm (1/2")		27166	27167
17mm		27168	27169
Shimadzu Plug		27170	27171
BTO® Septa new!			
5mm CenterGuide™		27100	27101
6mm (1/4")		27102	27103
9mm CenterGuide™		27104	27105
9.5mm (3/8")		27106	27107
10mm		27108	27109
11mm (7/16") CenterGuide™		27110	27111
11.5mm CenterGuide™		27112	27113
12.5mm (1/2") CenterGuide™		27114	27115
17mm CenterGuide™		27116	27117
Shimadzu Plug		27118	27119



Thermolite® Septa
- Usable to 340°C inlet temperature.
- Excellent puncturability.

IceBlue™ Septa
- Usable to 250°C inlet temperature.
- General-purpose septa.
- Excellent puncturability.
- Ideal for SPME.

BTO® Septa
- CenterGuide™ design—requires less force for initial penetration.
- Usable to 400°C inlet temperature.
- Each batch GC-FID tested.
- Bleed and temperature optimized; ideal for demanding GC and GC/MS applications.

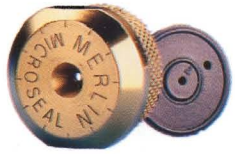
Septum Puller

- Keep several on hand in your laboratory—can be used in many different ways.
- Use hooked end for removing septa and O-rings; pointed end for removing stuck ferrules or fragments.

Description	qty.	cat.#
Septum Puller	ea.	20117

Merlin Microseal™ Septa

- Allow operation from 2 to 100psi (400 Series) or 2 to 30psi (300 Series).
- Top wiper rib improves resistance to particulate contamination; can be taken apart for cleaning.
- High resistance to wear—greatly reduces shedding of septum particles into the injection port liner, eliminating a major source of septum bleed and ghost peaks.
- Longer life—reduces the risk of septum leaks during extended automated runs.
- Maximum temperature—Agilent 6890, 5890 Series II: 325°C; Agilent 5890A: 300°C.



Microseal™ High-Pressure Septa, 400 Series (100psi)	Merlin#	Similar to Agilent#	cat.#
Standard kit (nut, 2 septa)	404	Not offered	22810
Starter kit (nut, 1 septum)	405	5182-3442	22811
Nut kit (1 nut, fits 300 & 400 series septa)	403	5182-3445	22809
High-pressure replacement septum (1 septum)	410	5182-3444	22812
Microseal™ Septa, 300 Series (30psi)			
Standard kit (nut, 2 septa)	304	5181-8833	22813
Starter kit (nut, 1 septum)	305	5181-8816	22814
Microseal replacement septum (1 septum)	310	5181-8815	22815
Replacement PTFE washers (2-pk.)	311	5182-0853	22808

Click-On Inline Super-Clean™ Traps

by Donna Lidgett, GC Accessories Product Marketing Manager

Click-On Inline Super-Clean™ Traps

- High-purity output ensures 99.9999% pure gas.
- Click-On fittings for easy, leak-tight cartridge changes; available in brass or stainless steel, 1/4" or 1/8".
- Helium-Specific Triple Trap is ideal for GC/MS.

Using the same features and benefits as Super-Clean™ base-plates and filters (see our 2006 catalog), Click-On Inline Super-Clean™ adaptor connectors allow cartridges to be exchanged without introducing air. Spring-loaded check valves seal when a filter is removed and open only when a new filter has been locked in place. There is no longer a need for loosening and tightening fittings every time a trap is changed, and your system will not become contaminated during the process.

The Triple Trap is ideal for purifying carrier gas—it contains oxygen, moisture, and hydrocarbon scrubbers in one cartridge.

please note

Super-Clean™ traps are recommended for purifying non corrosive gases with low concentrations of contaminants. For oxygen traps, the maximum concentration of oxygen in the incoming gas stream is 0.5%.

The Fuel Gas Trap is ideal for purifying flame ionization detector (FID) fuel gases, removing both moisture and hydrocarbons.

The Helium-Specific Triple Trap is ideal for purifying helium in GC/MS systems. This trap is packed and purged under helium and contains oxygen, moisture, and hydrocarbon scrubbers in one cartridge.

Trap replacement depends on the quality of the incoming gas. Use the double connector and install an indicating cartridge after the trap to indicate when a trap should be replaced.

Filter Type	Gas Quality at Outlet	Maximum Pressure	Maximum Flow (L/min.)	Use For	Capacity H_2O (g)	Capacity O_2 (mL)	Capacity Hydrocarbons (g)	Estimated Lifetime (years)
Moisture cat.#22467	>99.9999	11 bar 160psi	25	Inert carrier gas, helium, air, H_2	21	NA	NA	>3
Oxygen cat.#22468	>99.9999	11 bar 160psi	25	Inert carrier gas	NA	3000	NA	>3
Hydrocarbon cat.#22466	>99.9999	11 bar 160psi	25	Inert carrier gas, helium, air, H_2	NA	NA	36[3]	>3
Fuel Gas[1] cat.#22465	>99.9999	11 bar 160psi	25	Inert carrier gas, helium, air, H_2	10	NA	18[3]	>2
Triple[2] cat.#22464	>99.9999	11 bar 160psi	25	Inert carrier gas	6	1000	12[3]	>2

[1]Removes hydrocarbons, moisture.
[2]Removes hydrocarbons, moisture, oxygen.
[3]As *n*-butane.

Click-On Inline Super-Clean™ Trap and Connector Kits

Description	qty.	cat.#
Carrier Gas Purification Kit, 1/8" Stainless Steel Includes (2) 1/8" SS connectors and (1) oxygen/moisture/hydrocarbon triple trap	kit	22456
Carrier Gas Purification Kit, 1/8" Brass Includes (2) 1/8" brass connectors and (1) oxygen/moisture/hydrocarbon triple trap	kit	22457
Carrier Gas Purification Kit, 1/4" Stainless Steel Includes (2) 1/4" SS connectors and (1) oxygen/moisture/hydrocarbon triple trap	kit	22458
Carrier Gas Purification Kit, 1/4" Brass Includes (2) 1/4" brass connectors and (1) oxygen/moisture/hydrocarbon triple trap	kit	22459
Fuel Gas Purification Kit, 1/8" Stainless Steel Includes (4) 1/8" SS connectors and (2) hydrocarbon/moisture traps	kit	22460
Fuel Gas Purification Kit, 1/8" Brass Includes (4) 1/8" brass connectors and (2) hydrocarbon/moisture traps	kit	22461
Fuel Gas Purification Kit, 1/4" Stainless Steel Includes (4) 1/4" SS connectors and (2) hydrocarbon/moisture traps	kit	22462
Fuel Gas Purification Kit, 1/4" Brass Includes (4) 1/4" brass connectors and (2) hydrocarbon/moisture traps	kit	22463

Click-On Inline Super-Clean™ Replacement Traps

Description	qty.	cat.#
Click-On Super-Clean™ Triple Trap (removes oxygen, moisture and hydrocarbons)	ea.	22464
Click-On Super-Clean™ Fuel Gas Trap (removes moisture and hydrocarbons)	ea.	22465

Click-On Inline Super-Clean™ Ultra-High Capacity Traps

Description	qty.	cat.#
Ultra-High Capacity Hydrocarbon Trap	ea.	22466
Ultra-High Capacity Moisture Trap	ea.	22467
Ultra-High Capacity Oxygen Trap	ea.	22468

Helium-Specific Click-On Inline Super-Clean™ Trap Kits

Description	qty.	cat.#
Kits		
Helium-Specific Carrier Gas Cleaning Kit, 1/8" Stainless Steel Includes (2) 1/8" SS connectors and (1) oxygen/moisture/hydrocarbon Helium-Specific Triple Trap	kit	22469
Helium-Specific Carrier Gas Cleaning Kit, 1/8" Brass Includes (2) 1/8" brass connectors and (1) oxygen/moisture/hydrocarbon Helium-Specific Triple Trap	kit	22470
Helium-Specific Carrier Gas Cleaning Kit, 1/4" Stainless Steel Includes (2) 1/4" SS connectors and (1) oxygen/moisture/hydrocarbon Helium-Specific Triple Trap	kit	22471
Helium-Specific Carrier Gas Cleaning Kit, 1/4" Brass Includes (2) 1/4" brass connectors and (1) oxygen/moisture/hydrocarbon Helium-Specific Triple Trap	kit	22472
Replacement Trap		
Helium-Specific Triple Trap (removes oxygen, moisture and hydrocarbons)	ea.	22473

did you know?

The Helium-Specific Click-On Inline Super-Clean™ Trap is designed specifically for purification of helium in GC/MS systems!

Click-On Inline Super-Clean™ Indicator

- Color changes: oxygen-green to grey; moisture-beige to clear

Description	qty.	cat.#
Click-On Inline Super-Clean™ Indicator (oxygen, moisture plus adsorbents and hydrocarbons)	ea.	22474

tech tip

Install an indicator after the Click-On inline trap so there is no confusion about when to replace the trap.

Click-On Inline Super-Clean™ Connectors

Description	qty.	cat.#
1/8" Brass Click-On Inline Super-Clean™ Connectors	2-pk.	22475
1/8" Stainless Steel Click-On Inline Super-Clean™ Connectors	2-pk.	22476
1/4" Brass Click-On Inline Super-Clean™ Connectors	2-pk.	22477
1/4" Stainless Steel Click-On Inline Super-Clean™ Connectors	2-pk.	22478

Click-On Inline Super-Clean™ Double Connector

Description	qty.	cat.#
Click-On Inline Super-Clean™ Double Connector, stainless steel (connects trap and indicator)	ea.	22479

Wall-Mounting Clamps for Click-On Inline Super-Clean™ Traps

Description	qty.	cat.#
Wall-Mounting Clamps for Click-On Inline Super-Clean™ Traps	4-pk.	22480

Replacement O-Rings for Click-On Inline Connectors

Description	qty.	cat.#
Replacement O-Rings for Click-On Connectors	10-pk.	22481

New Rxi™ Fused Silica Columns *Continued from page 3.*

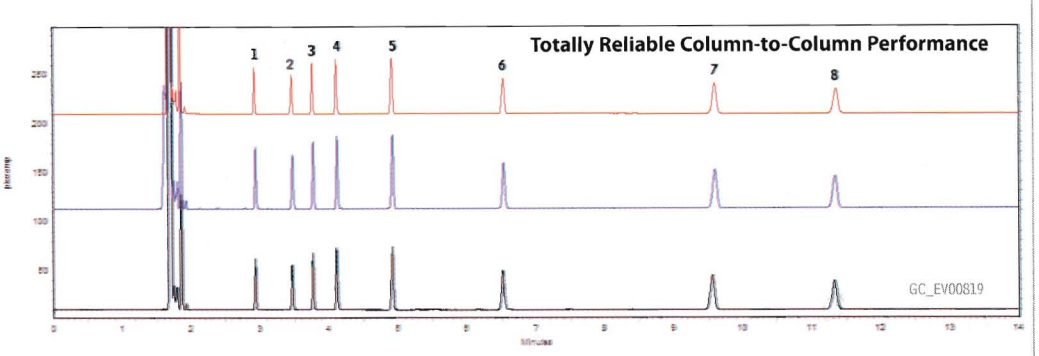
for more info

Analyses of acidic/neutral and basic drugs on Rxi™-5ms columns are described on pages 8-9 of this *Advantage*.

Totally Reliable Column-to-Column Performance

Chromatographers need to know every column they receive is going to perform in the same way as the column it replaces. Rxi™ column technology has enabled us to tighten our quality control standards, and guarantee reproducibility. Columns from each of three manufacturing batches show the excellent reproducibility assured by the new manufacturing process.

Figure 3 Three manufacturing batches of Rxi™ columns show excellent reproducibility.



Guaranteed Quality and Reliability

Rxi™ columns are already proving to be the best columns on the market, for inertness, ultra-low bleed, and column-to-column uniformity. It is our promise and commitment to you that every Rxi™ column you receive will be *exactly* as good as the one it replaces.

Typical Applications

Alcohols, amines, aromatic hydrocarbons, bile acids, drugs, EPA Methods, esters, fatty acid methyl esters (FAMEs), flavors and aromas, glycerides, halogenated hydrocarbons, herbicides, hydrocarbons, organic acids, oxygenates, polynuclear aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, phenols, polymers, solvents, steroids, sugars, sulfur compounds

Rxi™-5ms Columns (fused silica) new!

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

- Nonpolar 5% dimethyl / 95% dimethylpolysiloxane phase, equivalent to USP Phase G27.
- Operating temperature range: -60 to 330/350 °C.
- Most widely used general purpose column.

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.25	-60 to 330/350°C	13420	13423
	0.50	-60 to 330/350°C	13435	13438
	1.00	-60 to 330/350°C	13450	13453
0.32mm	0.25	-60 to 330/350°C	13421	13424
	0.50	-60 to 330/350°C	13436	13439
	1.00	-60 to 330/350°C	13451	13454

For other dimensions, and additional information about Rxi™ columns, please visit our website: **www.restek.com/rxi**

RESTEK 20 YEARS

Lit. Cat.# 580035-INT

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