

THE RESTEK ADVANTAGE

Turning Visions into Reality™

2005 vol. 1

Viva™ HPLC Silica: Ideal for Separating Large Molecules

new!

New Wide Pore Silica, Designed and Manufactured by Restek

by Vernon Bartlett, HPLC Manager, Bruce Albright, HPLC Chemist,
and Rebecca Wittrig, Ph.D., HPLC Product Marketing Manager

- 67% of available surface area can interact with proteins, peptides, other large molecules.
- Larger surface area than other commercially available 300Å materials.
- Manufactured by Restek, quality controlled by Restek.



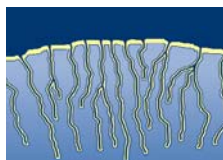
Numerous HPLC grade silica materials currently are available in the marketplace, but these silicas differ greatly from one manufacturer to

another. Some of the most important factors affecting the selectivity of a substrate are surface area, pore volume, and pore diameter distribution. We have determined these physical properties of our new Viva™ 300 Ångstrom silica, and have compared this silica to other commercially available 300Å silicas.

Of the silicas tested, Viva™ 300Å silica shows the largest available surface area and the greatest percentage of pores narrowly distributed around a mean diameter of 300Å (Table I). These characteristics ensure greater accessibility to larger molecules, relative to other materials. They also are important because silicas with excessive numbers of pores smaller than 200Å can become more easily fouled with larger molecular weight debris, and silicas with excessive numbers of pores larger than 500Å can be impractically fragile for conventional HPLC applications.

Figure 1 depicts a typical porous silica particle. In general, as the number of pores in a silica increase, surface area and pore volume

Figure 1 A typical porous silica substrate: as the number of pores increase, surface area and pore volume increase.



increase. Also, as pore width increases, pore volume increases. For a fixed pore volume, materials having the smallest pore diameters have the largest available surface area (Table II). While smaller pores (e.g., 60Å) maximize retention of small molecules, larger pores are necessary when analyzing higher molecular weight analytes, such as proteins and peptides, because retention will be maximized if an analyte can enter into the pores of the material. Theoretically, the more pores to which an analyte has access, the longer the retention. For analytes with molecular weights greater than 3000, silica materials with pore diameters in the 250-350Å range, or larger, should yield the highest retention. In addition, a narrow pore diameter distribution is desirable,

Table I Viva™ silica has the highest percentage of available surface area from 200-300Å pores, allowing the greatest interaction with large molecules.

Silica	Total Surface Area (m ² /g)	% of Total Surface Area		
		<200Å	200-300Å	>300Å
Viva™ 300Å	128.0	2.5	67.3	30.2
(7) 300Å	51.8	65.6	18.5	15.9
(6) 300Å	87.2	53.6	22.2	24.2
(5) 300Å	105.8	56.3	22.3	21.4
(3) 300Å	83.5	40.5	24.5	35.0
("B") 200Å	231.5	66.1	33.1	0.8
("B") 300Å	118.1	8.3	34.3	57.4

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because this can aid in separating closely related analytes that differ only slightly in hydrodynamic size (size in solution). In developing Viva™ silica, we found some "wide pore" materials do not possess sufficiently large pore volume in the pore diameter range needed for effectively separating large molecules.

Table II For a fixed pore volume, the smaller the pores in a silica particle, the larger the surface area.

Pore Diameter (Å)	Surface Area (m ² /g)
60	300-600
100	150-300
200	75-150
300	50-75
500	30-40
1000	20-30

RESTEK 20 YEARS

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In addition to our Viva™ 300Å silica, we evaluated 300Å materials from five other vendors, and one 200Å material, determining pore characteristics and surface area for each. We used nitrogen gas porosimetry, BET measurements¹, and BJH calculation² to determine the surface area,

pore volume, and pore diameter distribution of each material. Figures 2 and 3 show the pore volume and pore area for each material; Table I indicates the available surface area for a given pore diameter range. Viva™ 300Å silica shows, by far, the greatest available surface area rep-

resented by 200-300Å pores. None of the other materials evaluated comes close to the 67.3% value obtained for Viva™ 300Å silica.

In selecting a wide pore material, it is important to know the available surface area, the pore volume, and the pore diameter distribution, because these are the critical factors in determining retention. The exceptionally large available surface area of Viva™ 300Å silica, and a highly desirable pore volume and pore diameter distribution, will help ensure effective retention of peptides, proteins, or other large molecules, making Viva™ 300Å products an excellent choice for your analyses.

References

1. Barton, T., et al., *Tailored Porous Materials* Chem. Mater. 11: 2633-2656 (1999).
2. Webb, P.A. and C. Orr, *Analytical Methods in Fine Particle Technology* Micrometrics, Georgia, 1997, pp. 53-152.



free literature

HPLC Columns and Accessories

General-purpose and unique special-purpose columns, guard columns, column kits, and bulk materials, plus instrument parts, innovative tools, accessories, and many example chromatograms. Our five silicas have characteristics tailored to meet specific analytical requirements. Request lit. cat.# 59241B (100 pages).

Figure 2 Pore volume vs pore diameter for commercial wide pore silicas (BJH desorption). Only Viva™ silica has a sharp distribution around 300Å. (Change in scale for plots at right.)

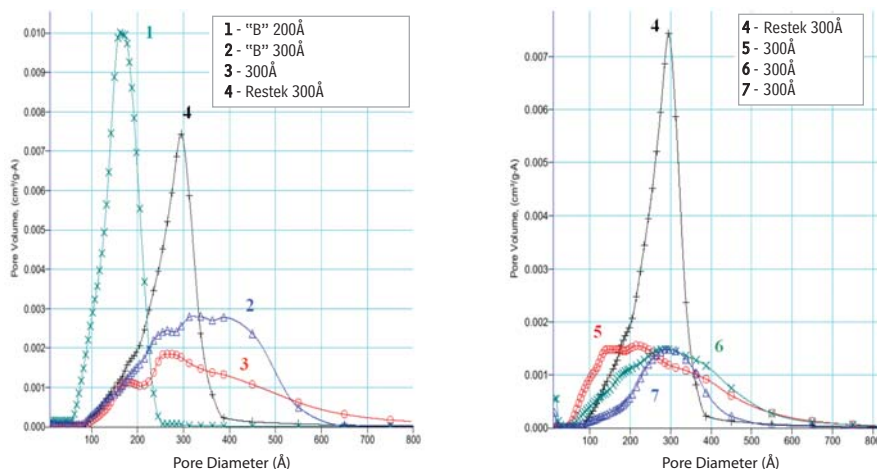
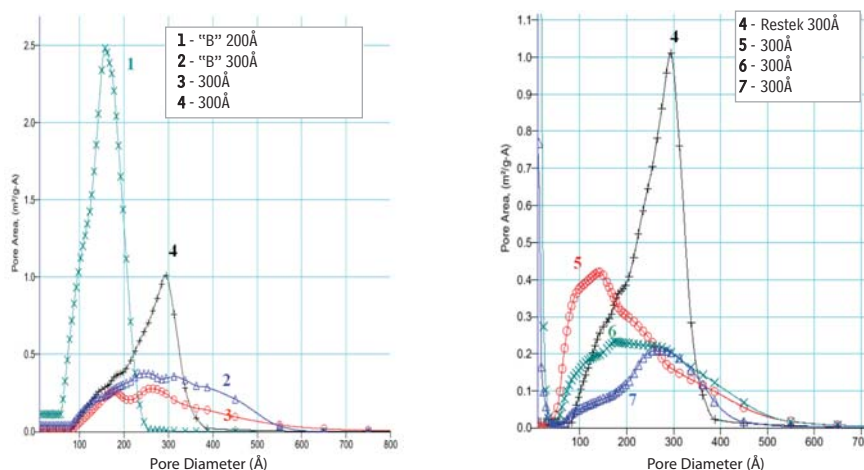


Figure 3 Pore area vs pore diameter for commercial wide pore silicas (BJH desorption). Viva™ silica shows a highly desirable distribution. (Change in scale for plots at right.)



Viva™ Wide Pore C18 Columns

Length	3.2mm ID cat.#	4.6mm ID cat.#
5µm Columns		
100mm	9514513	9514515
150mm	9514563	9514565
250mm	9514573	9514575

Viva™ Wide Pore Silica Columns

Length	3.2mm ID cat.#	4.6mm ID cat.#
5µm Columns		
100mm	9510513	9510515
150mm	9510563	9510565
250mm	9510573	9510575

Viva™ wide pore silica packings are available in bulk. Please inquire: 800-356-1688 or 814-353-1300, ext. 4, or contact your Restek representative.

Replacement Parts for Dionex ASE® Systems

by Neil Mosesman, SPE Product Marketing Manager, and Brad Rightnour, Instrument Innovations Manager

- Designed to meet or exceed performance of original manufacturer's parts.
- Siltek® treated parts assure maximum inertness.
- Save time—order parts when you order GC or HPLC columns and consumables.
- Renowned Restek Plus 1™ service.

Accelerated solvent extraction has become a common technique for fast and reliable extraction of organic materials from solid matrices. Restek now offers several direct replacement parts for ASE® extraction systems, including extraction cells, caps, frits, and PEEK® washers. In addition to stainless steel extraction cells—equivalent to original parts—we offer cells, caps, and frits finished with our innovative Siltek® treatment. Siltek® treatment reduces the surface activity of stainless steel, greatly improving inertness and, therefore, the reliability of analytical results for active compounds. All of our replacement parts for ASE® systems are designed to meet or exceed original equipment manufacturer's performance. Look to Restek to keep your extraction systems running smoothly.

Kits and Parts for ASE® Extraction Units

Each kit includes: 1 extraction chamber, 2 end caps, 2 frits, and 2 PEEK® washers.

Description	Similar to Dionex		
	part #	qty.	cat.#
Extraction Cell Kit for ASE® 200 Extraction Unit, 22mL Tubes	048764	kit	26094
Extraction Cell Kit for ASE® 200 Extraction Unit, Siltek®-Treated, 22mL Tubes	—	kit	26095
Replacement Caps for ASE® 200 Extraction Unit, Universal	049450	2-pk.	26096
Replacement Caps for ASE® 200 Extraction Unit, Siltek®-treated, Universal	—	2-pk.	26097
Extraction Tube for ASE® 200 Extraction Unit, 22mL	048821	ea.	26098
Extraction Tube for ASE® 200 Extraction Unit, Siltek®-treated, 22mL	—	ea.	26099
Replacement Frits for ASE® 200 Extraction Unit, Universal	049453	6-pk.	26100
Replacement Frits for ASE® 200 Extraction Unit, Siltek®-treated, Universal	—	6-pk.	26101

PEEK® Washers for ASE® Extraction Unit

Meet original equipment manufacturer's performance.

Description	Similar to Dionex		
	part #	qty.	cat.#
PEEK® Washers for ASE® 200 Extraction Unit	049454	12-pk.	25256
PEEK® Washers for ASE® 200 Extraction Unit	049454	48-pk.	25257
PEEK® Washers for ASE® 300 Extraction Unit	061687	12-pk.	25393
PEEK® Washers for ASE® 300 Extraction Unit	061687	48-pk.	25394

new!

Siltek® Treated
Parts for ASE®
Systems



Untreated Parts for
ASE® Systems



PEEK® Washers for ASE®
Extraction Unit

Solid Phase Extraction Tubes for Extracting Nitrosamines from Drinking Water

by Neil Mosesman, SPE Product Marketing Manager

- Meet the requirements of EPA Method 521.
- Batch tested charcoal ensures consistent recoveries.

Newly proposed US EPA Method 521 is designed for the analysis of 7 nitrosamines in drinking water. This method employs a 6mL solid phase extraction (SPE) tube packed with 2 grams of coconut charcoal to extract and concentrate the nitrosamines from the aqueous matrix.

We perform a batch test on each lot of coconut charcoal we use to prepare these new tubes, to ensure consistent recoveries and low background. High quality polypropylene tubes and frits are used to minimize interferences.

SPE Tubes for US EPA Method 521

Description	Applications	Tube Volume, Bed Weight	qty.	cat.#
EPA Method 521	For use in EPA Method 521, Nitrosamines in Drinking Water. This method uses large volume injection and CI, MS-MS. Activated charcoal for NDMA.	6mL, 2g	30-pk.	26032

new!



Cartridges may be processed by any one or all of these techniques: positive pressure, sidearm flask, centrifuge, or vacuum manifold.

Analysis of Organochlorine Pesticides

Using 2D-GC with Rtx®-5 and Rtx®-200 Capillary GC Columns

by Frank Dorman, Ph.D., Director of Technical Development

- GCxGC analysis combines primary column and confirmation column results.
- Separate target compounds from co-extracted contaminants in sample extracts.
- Analyte refocusing effect increases sensitivity.
- Combination of Rtx®-5 and Rtx®-200 columns resolves all target pesticides.

By using application-specific capillary GC columns, such as our Rtx®-CLPesticides and Rtx®-CLPesticides2 columns, many laboratories analyzing organochlorine pesticides can separate all of the target compounds. But, with complex matrices, there still can be difficulties in resolving the target compounds from co-extracted interfering matrix components. Especially difficult are samples contaminated with chlorinated organic compounds, such as PCBs. Like the target analytes, these contaminants produce a signal on the electron capture detectors (ECDs)

commonly used for this application. In order to separate the target compounds from the co-extracted contaminants in many sample extracts, Restek chemists, in collaboration with colleagues at LECO Corporation, have investigated GCxGC technology.

Comprehensive GCxGC is a relatively new, exciting technique that increases chromatographic peak capacity by enabling the analyst to use two columns of differing selectivity in a single analysis. By coupling two columns in series, and

incorporating a modulation technique at the junction between the two columns (e.g., valving or cryomodulation), it is possible to get the benefit of each column, as in independent separations. This technique has been reviewed in depth by Professor John Dimandja¹, and the reader is urged to consult this reference for details. There are several manufacturers of commercial GCxGC instruments, and the technique can be adapted to conventional instrumentation.

In determining which column pair to use for a GCxGC application, it is important to choose stationary phases that differ in selectivity. For this application, we choose an Rtx®-5 column for a volatility-based separation, in series with an Rtx®-200 column which is selective for halogenated compounds. The second-dimension separation from this column ensemble is focused on retention of halogenated compounds, and separates the target compounds from some of the possible interferences in the sample matrix.

Figure 1 is a GCxGC chromatogram of 22 common organochlorine pesticides, obtained from the Rtx®-5 column/Rtx®-200 column ensemble in a LECO GCxGC/ECD instrument. Table I lists the compounds and the independent retention times observed in the two dimensions of separation. By having two independent retention times, from two different columns, we obtain a primary column separation and a secondary column confirmation for the target compounds, so this technique should be in compliance with any methodology requiring a primary column/confirmation column approach.

Additionally, an analysis of a spiked extracted food sample (tomato) shows we can separate the target compounds from many co-extracted interferences (Figure 2). Recovery values for the spiked sample, listed in Table II, are in agreement with "known" values, indicating little to no matrix interference with target compound quantification, even for a difficult matrix like a food.

A secondary benefit of using cryomodulation at the column junction is peak sharpening prior to "injection" of an analyte onto the second column. This has the effect of increasing sensitivity. Due to this analyte refocusing effect, we obtained linear calibration for these compounds over a 25-fold wider range of concentration than by conventional GC. The compounds for which detection is most sensitive (e.g., the hexachlorocyclohexanes, or BHCs) normally are calibrated from 5 to 80 pg/μL. We were able to calibrate from 0.2 to 80 pg/μL, thus greatly extending the reporting limit. We also were able to employ split injection, which typically reduces injector-related problems, such as analyte adsorption and breakdown.

Figure 1 GCxGC analysis of organochlorine pesticides combines primary column and confirmation column results.

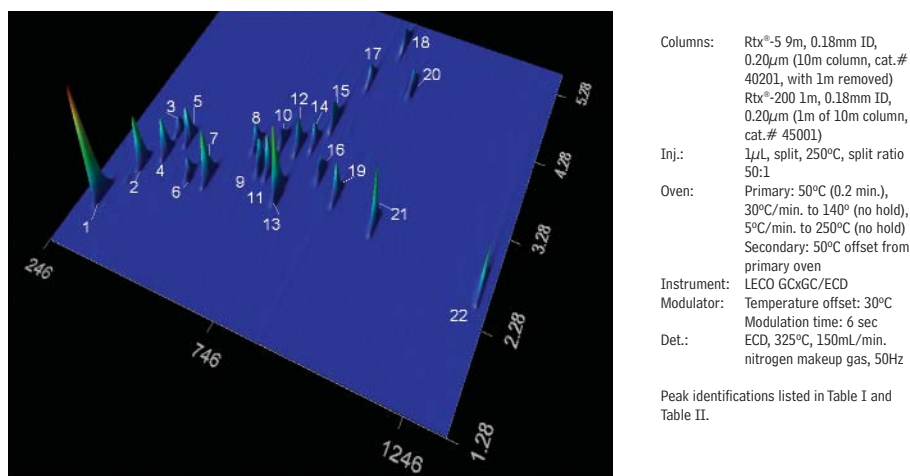
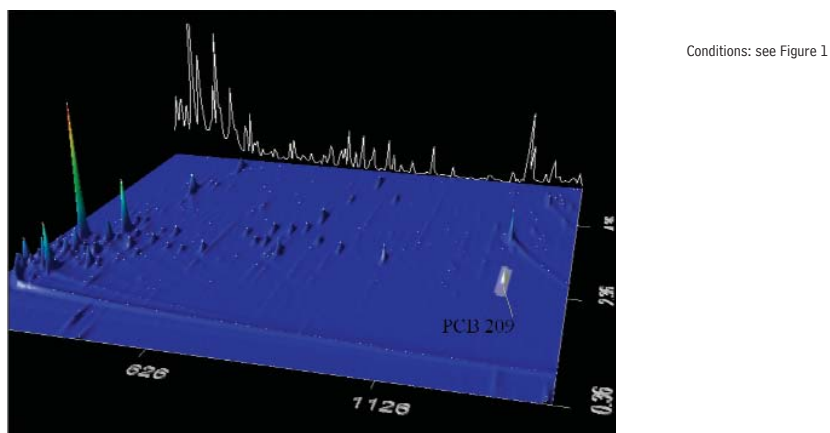


Figure 2 Organochlorine pesticides separated from interferences in tomato extract.



Pittcon® presentation

Jack Cochran, LECO Corporation, will present this information at the 2005 Pittsburgh Conference in Orlando, Florida. (1000-7)

For more information about this application, or about GCxGC in general, please contact our Technical Service chemists, or your Restek representative.

Reference

1. Dimandja, J., Anal. Chem. 76 (9): 167A - 174A (2004).

Acknowledgement

This investigation was conducted in collaboration with Jack Cochran, Director of Separation Science, LECO Corporation, Las Vegas, NV.

Table I Organochlorine pesticides and retention times in GCxGC separation.

Analyte	T _R (sec.)	
	Dimension 1	Dimension 2
1. tetrachloro- <i>m</i> -xylene	294	1.9
2. α -BHC	342	2.44
3. β -BHC	378	2.96
4. γ -BHC	384	2.66
5. δ -BHC	420	2.94
6. heptachlor	480	2.52
7. aldrin	534	2.58
8. heptachlor epoxide	606	3.16
9. γ -chlordane	648	2.96
10. endosulfan I	672	3.32
11. α -chlordane	678	2.96
12. dieldrin	720	3.34
13. 4,4'-DDE	732	2.72
14. endrin	756	3.46
15. endosulfan II	780	3.78
16. 4,4'-DDD	810	3.18
17. endrin aldehyde	816	4.5
18. endosulfan sulfate	864	5.2
19. 4,4'-DDT	882	2.96
20. endrin ketone	942	4.68
21. methoxychlor	1008	2.82
22. decachlorobiphenyl	1320	2.62

Table II Pesticide recovery values for a spiked sample tomato extract agree with theoretical values, indicating interference from matrix is minimal.

Analyte	Spike		Recovery (%)
	Sample Amount Quantified	Sample Amount Theoretical	
1. tetrachloro- <i>m</i> -xylene	0	14.4	90
2. α -BHC	0	7.8	98
3. β -BHC	0	7.1	89
4. γ -BHC	0	6.8	85
5. δ -BHC	0	6.5	81
6. heptachlor	0	9.3	116
7. aldrin	0	7.2	90
8. heptachlor epoxide	0	9.3	116
9. γ -chlordane	0	6.9	86
10. endosulfan I	9.5	18.7	115
11. α -chlordane	0	7.5	94
12. dieldrin	0	17.4	109
13. 4,4'-DDE	0	16.8	105
14. endrin	0	14.2	89
15. endosulfan II	15.4	27.9	78
16. 4,4'-DDD	0	13.9	87
17. endrin aldehyde	0	12.8	80
18. endosulfan sulfate	13.1	28.2	94
19. 4,4'-DDT	0	15.7	98
20. endrin ketone	0	13	81
21. methoxychlor	0	76.8	96
22. decachlorobiphenyl	0	16.2	101

Rtx®-5 Column (fused silica)

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

Temp. limits: -60 to 325/340°C

ID	df (μm)	length	cat. #
0.18mm	0.20	10-Meter	40201

Rtx®-200 Columns (fused silica)

(Crossbond® trifluoropropylmethyl polysiloxane)

Temp. limits: -20 to 310/330°C

ID	df (μm)	length	cat. #
0.18mm	0.20	10-Meter	45001

Organochlorine Pesticide Mix AB #2

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 (ea.)

Pesticide Surrogate Mix

decachlorobiphenyl
2,4,5,6-tetrachloro-*m*-xylene

200μg/mL each in acetone, 1mL/ampul
cat. # 32000 (ea.)

We're On the Road Again

2005 Restek Seminars—in the US and Internationally!

By Rick Parmely, Director of Technical Training

- Comprehensive Capillary GC
- Comprehensive HPLC
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How can I quickly choose the correct column for my application?

What is crucial to achieving reliable results from flash vaporization sample injection?

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If answers to questions like these don't come easily, let the experienced Restek chromatographers help! Our widely acclaimed seminars reduce guesswork, improve throughput and reliability, and raise confidence in results. Your investments of time and money will be very wisely spent.

In 2005, we are offering more topics as well. We have returned the popular Foods, Flavors, and Fragrances to our list of available subjects. This special-topic seminar zeros in on FFF applications, while incorporating some basic instruction on GC techniques for new operators. New GC Hands-On Maintenance and Troubleshooting

gives front-line chromatographers basic information to assist in preventing and troubleshooting problems with GC injection ports and flame ionization detectors. We can offer this hands-on course only at customer sites, to a limited enrollment. Please call for details.

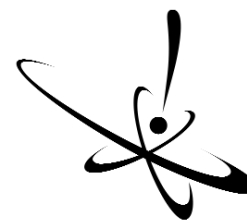
We present all of our seminars in a professional, non-promotional, engaging format, with animated illustrations and problem-solving exercises that reinforce understanding of important principles. Because of the range of information presented, both advanced users and beginners come away with important information of immediate value in the laboratory.

Choose the topic that best matches your work, at a convenient site near you. Or, we'll come to your facility: contact our Technical Service chemists, or your local Restek representative. See our most current schedule on line at www.restek.com/seminar

Superior Storage and Transfer of Sulfur Compounds

Sulfinert® Treated Systems Preserve ppb Levels of Active Compounds

Gary Barone, Restek Performance Coatings Division Manager, David Smith, RPC Chief Scientist,
and Martin Higgins, RPC Chief Engineer



**Restek
Performance
Coatings**

- Improve analytical accuracy and reduce system cycle times, using Sulfinert® treated products.
- Increase analytical confidence for low parts-per-billion sulfur compounds, using Sulfinert® treated sample cylinders.
- Transfer sulfurs in gas streams, without loss, using Sulfinert® treated electropolished tubing.



Accurate analyses for parts-per-million to parts-per-billion levels of organosulfur compounds in petrochemical streams are critical to meeting new regulations for lower levels of sulfur in diesel fuel and gasoline. Many sulfur compounds, including hydrogen sul-

fide, methyl mercaptan, and ethyl mercaptan, adsorb strongly to metal surfaces in sampling, storage, and transfer apparatus. In addition to causing inaccurate, falsely low values, adsorption can prolong analysis cycle times. To determine quantitative losses of active sulfur species, we sampled, stored, and transferred low ppmv to low ppbv concentrations of active sulfur gases, using control (untreated) and Sulfinert® treated system components.

Preventing Sulfur Compound Losses During Storage

Figure 1a depicts results from a comparison in which a gas containing 17ppbv of hydrogen sulfide was stored for 7 days in untreated or in Sulfinert® treated stainless steel sample cylinders. The response ratio for hydrogen sulfide, relative to a stable reference material, dimethyl sulfide, is steady at approximately 1:1 for at least seven days in Sulfinert® treated cylinders. The data show a Sulfinert® treated system will reliably store ppb levels of the active sulfur-containing compound during transport from the sampling site to the analytical laboratory. In contrast, hydrogen sulfide degraded rapidly in the untreated cylinder, and was lost totally within 24 hours.

In a similar study in which gas containing 18.8ppbv methyl mercaptan was stored for 60 hours in Sulfinert® treated sample cylinders,

recovery of the active sulfur compound was equally high relative to the stable reference material, dimethyl mercaptan, as shown in Figure 1b.

Sample Transfer: Adsorption of Sulfur Compounds to Tubing

Comparison of Sulfinert® treated electropolished stainless steel tubing (TrueTube™ EPS tubing, surface roughness average (RA): 5-10, O'Brien Corporation, St. Louis, MO), untreated electropolished stainless steel tubing (TrueTube™ EP tubing, RA 5-10, O'Brien Corporation), and raw commercial grade stainless steel tubing (RA 23-27) showed only the Sulfinert® treated electropolished tubing has the inertness necessary for quantitatively transferring low ppmv to low ppbv concentrations of sulfur compounds. Figures 2 and 3 depict the results (seamless 316L stainless steel, 1/8" OD, 0.020" wall). Tests were performed at room temperature, using a gas flow rate of 40cc/minute.

Figure 1 Sulfur compounds are stable in Sulfinert® treated stainless steel systems

- a) 17ppbv hydrogen sulfide in 500mL cylinders
b) 18.8ppbv methyl mercaptan in 300mL cylinders

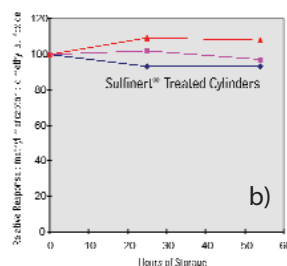
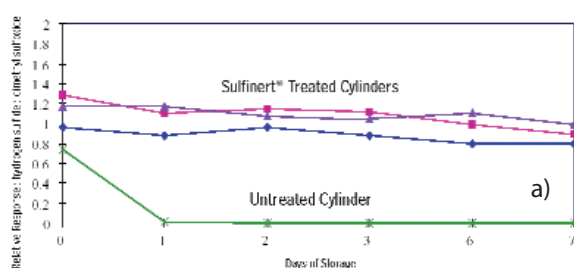


Figure 2 Sulfinert® treated electropolished seamless stainless steel tubing (red) does not adsorb methyl mercaptan (500ppbv). Blue-untreated electropolished tubing; violet-raw tubing.

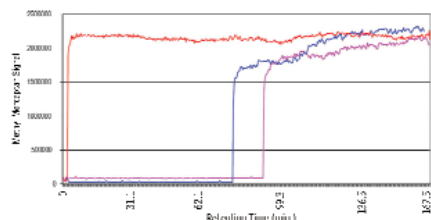
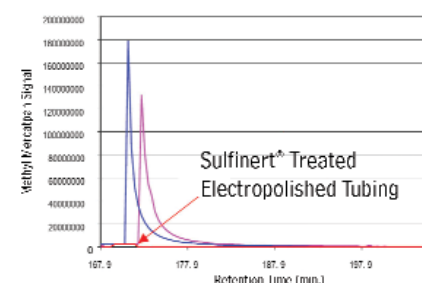


Figure 3 Sulfur memory is prolonged in raw commercial grade stainless steel tubing (violet). Red-Sulfinert® treated electropolished tubing; blue-untreated electropolished tubing. (500ppbv methyl mercaptan in helium)



To confirm whether an active sulfur-containing compound in a gas stream passing through 100-foot (30.5-meter) lengths of tubing would adsorb to active sites on the tubing surface, we measured the amount of time elapsed before values for the sulfur content exiting the tubing were stable and accurate, using helium containing 0.500ppmv methyl mercaptan. Figure 2 shows Sulfinert® treated electropolished tubing did not adsorb methyl mercaptan to any measurable extent, delivering a representative sample with no delay. Untreated electropolished tubing, in contrast, totally adsorbed methyl mercaptan for more than 75 minutes, and the sulfur gas level did not stabilize until approximately 130 minutes. Conventional 316L seamless tubing totally adsorbed methyl mercaptan for more than 90 minutes, and the sulfur gas level did not stabilize until approximately 140 minutes.

When adsorption of sulfur-containing compounds is prolonged, desorption from the surface also is slow. This "memory" of adsorbed compounds can cause long delays in equilibrating a sample stream. In Figure 3, Sulfinert® treated tubing shows the lowest retention of sulfur compounds, by several orders of magnitude. Samples can be evaluated, with accurate results, with no delay between them.

Reference

1. Application of TrueTube™ in Analytical Measurement
Cardinal UHP August 2004

Available at www.restekcoatings.com or by contacting us at 800-356-1688, ext. 4. Request lit. cat.# 59088.

Economic Value of an Inert Pathway
Sulfinert® treated sampling and transfer systems offer more accurate results and faster cycle times. Improved accuracy and reliability of data for sulfur compounds allow improvements in downstream process control, with associated cost savings. Shorter cycles translate directly into more samples collected and analyzed in a given period of time. Savings accrued from shorter cycles can be calculated by looking at typical per-hour costs of operating processes that rely on accurate quantification of sulfur compounds: a one-hour delay in an 800,000 tons-per-year ethylene plant can cost \$50,000; a 250,000 tons-per-year EBSM styrene plant stands to lose \$33,000/hour; even for a

200,000 tons-per-year anti-freeze grade production process, the loss can be \$3,600/hour.¹

In these studies, we obtained accurate data, with no delay between samples, by using Sulfinert® treated electropolished tubing in the sampling-storage-transport system. In contrast, we obtained significantly less accurate data, even with delays of more than two hours between samples, by using untreated tubing. Analysts charged with monitoring sulfur levels in process streams can significantly improve process control, and profitability, by using Sulfinert® treated system components and Sulfinert® treated electropolished tubing transport lines.

Sulfinert®-Treated Electropolished Tubing

ID	OD	cat.#	5-24 ft.	25-99 ft.	100-299 ft.	> 300 ft.
0.085"	1/8"	22538				
0.180"	1/4"	22539				

Coiled Sulfinert®-Treated Seamless 316 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				

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.
** 0.035" wall thickness.

please note We can cut and finish tubing at nominal additional charge—please inquire.

Sulfinert®-Treated Sample Cylinders

D.O.T. rated to 1800psi at room temperature.

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



award-winning innovation!



Restek Surface Technology Earns R&D 100 Award

Our newest surface treatment, Silcosteel®-UHV, has been recognized by a panel of independent judges and editors of R&D Magazine as one of the 100 most technologically significant products introduced in 2003. Silcosteel®-UHV treatment minimizes the migration of water and oxygen molecules from the surfaces of ultra high vacuum system components into the vacuum chamber, allowing the system to be evacuated much more quickly, with less costly pumping equipment.

tech tip

Minimum Bend Radius

For tubing treated with Restek coatings

OD	Min. Bend Radius
≤ 1/16"	1" (2.5cm)
1/8"	2" (5.1cm)
1/4"	4" (10.2cm)

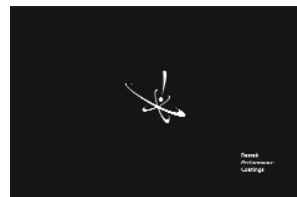


We offer Sulfinert®-treated Swagelok® and Parker fittings, and treated valves and sample loops.

For more information and ordering, call us or visit us online.

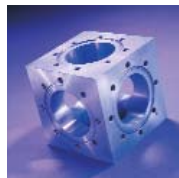
Find us on the web!

www.restekcoatings.com



free literature

Learn more about our precisely applied, highly durable surface treatments: request our new 38-page brochure today (lit. cat.# 59493).



did you know?

Restek surface treatments have many uses, in addition to analytical chemistry.

Silcosteel®

A general-purpose passivation layer for steel and stainless steel.
U.S. patent 6,511,760.

Silcosteel®-AC

Dramatically reduces carbon buildup on stainless steel components.
U.S. patent 6,444,326.

Silcosteel®-CR

A corrosion resistant layer that increases the lifetime of system components in acidic environments containing hydrochloric acid, nitric acid, sulfuric acid, or seawater.
Patent pending.

Silcosteel®-UHV

Greatly reduces outgassing from components of ultra-high vacuum systems. Patent pending.

Siltek®

The ultimate passivation for treated components, from glass to high nickel alloys of steel.
U.S. patent 6,444,326.

Sulfinert®

A required treatment for metal components when analyzing for parts-per-billion levels of organo-sulfur compounds.
U.S. patent 6,444,326.

Nine-Minute Analysis of Semivolatile Organic Compounds

Using an Rtx®-5Sil MS Capillary GC Column in Combination with TOFMS

by Frank Dorman, Ph.D., Director of Technical Development

- Monitor 81 analytes and internal standards in 9 minutes.
- Excellent resolution of critical target compounds.
- At least 20 scans for each peak.
- Use split injection, to minimize injection problems and extend reporting limits.

Analysts in many environmental laboratories struggle to increase sample throughput. Fast GC techniques have enabled analysis times to be decreased, but methods employing mass spectrometric detection often can't make use of these techniques, due to scan-speed limitations of commonly used instruments. While some manufacturers have improved the scan rates of their instruments, methods employing either quadrupole or ion-trap mass filters are limited by the residence time of an ion as it passes through the detector. In most cases, the scan-speed limitations of these devices preclude very rapid analyses of a wide range of compounds, such as the semivolatiles in environmental matrices, even though current capillary column and gas chromatograph technology would allow fast separations.

In order to adequately characterize a chromatographic peak as it elutes from the column, most methods require, at a minimum, 6 to 7 data points (scans) across the peak. Certainly, additional data points yield a better peak profile, and thus improved precision, so it is always better to have more than the 6 to 7 scan minimum. For a typical semivolatiles analysis, this correlates to a minimum scan rate of approximately 2 scans/second, with peak widths of 3 to 5 seconds considered "typical." It is important to note that this rate must be maintained over the entire expected mass range, or identifications, especially for unknown compounds, will be compromised. As faster GC techniques are investigated, peak widths are reduced and, as a result, the detector struggles to collect data at a rate that is fast enough to adequately characterize the peak profile. Unfortunately, for most GC/MS systems, this dictates a total analysis time of about 15 minutes, or longer, given the characteristics of most instruments used in this application.

In a recent collaboration, Restek and LECO Corporation developed a much faster analysis of common semivolatile organic compounds by taking advantage of both fast GC column technology and the speed of acquisition of the time-of-flight mass spectrometer (TOFMS). Using a 10 meter, 0.18 μ m ID, 0.18 μ m film Rtx®-5Sil MS fast GC column (phase optimized for semivolatiles analysis; low bleed) and TOFMS, the analysis time for this separation was less than 10 min-

utes, and at least 20 scans were recorded for each peak. Table 1 lists the retention times for the semivolatile target compounds, in seconds, and each compound had approximately a 1-second peak width at the base.

Figure 1 is the total ion chromatogram of a mid-level calibration standard of these compounds, analyzed under the conditions listed with the figure. Another valuable benefit of TOFMS is that there is a sensitivity improvement relative to most scanning instruments, enabling the analyst to use split injection. Split injection typically creates fewer maintenance issues than splitless injection, due to the much shorter residence time of the analytes in the injector, and produces narrower peaks, increasing resolution. For this analysis, the TOFMS system offers sensitivity sufficient to allow calibration beyond the 20 to 160ng/ μ L "normal" calibration range, to a range of 0.2 to 160ng/ μ L, even at a 50:1 split ratio, thus allowing laboratories to extend reporting limits (sensitivity) to lower levels.

Finally, extracts of actual samples were analyzed using this method, and results were compared to values obtained by a commercial environmental laboratory using conventional GC/MS. The results compared well, even for samples with high levels of non-target contaminants. Detailed information about this work is available on request, and will be presented at the 2005 Pittsburgh Conference.¹

If your laboratory is analyzing semivolatile organic compounds by GC/MS, and you are interested in significantly increasing sample throughput by reducing analysis time to less than 10 minutes, we urge you to request a copy of the complete report of this work, and/or attend our presentation at the Pittsburgh Conference.

Pittcon® presentation

1. Improved Sensitivity and Analysis Time for Semivolatile Organic Compounds, Using GC-TOFMS: Can this Analysis Really be Performed in Less Than 10 Minutes? Frank L. Dorman, Jack W. Cochran (LECO Corporation), Gary B. Stidsen, Chris M. English, Michael S. Wittrig
PittCon 2005, Monday, Feb. 28. Oral Session 380, Room S210C, presentation 380-3, 2:10 pm.

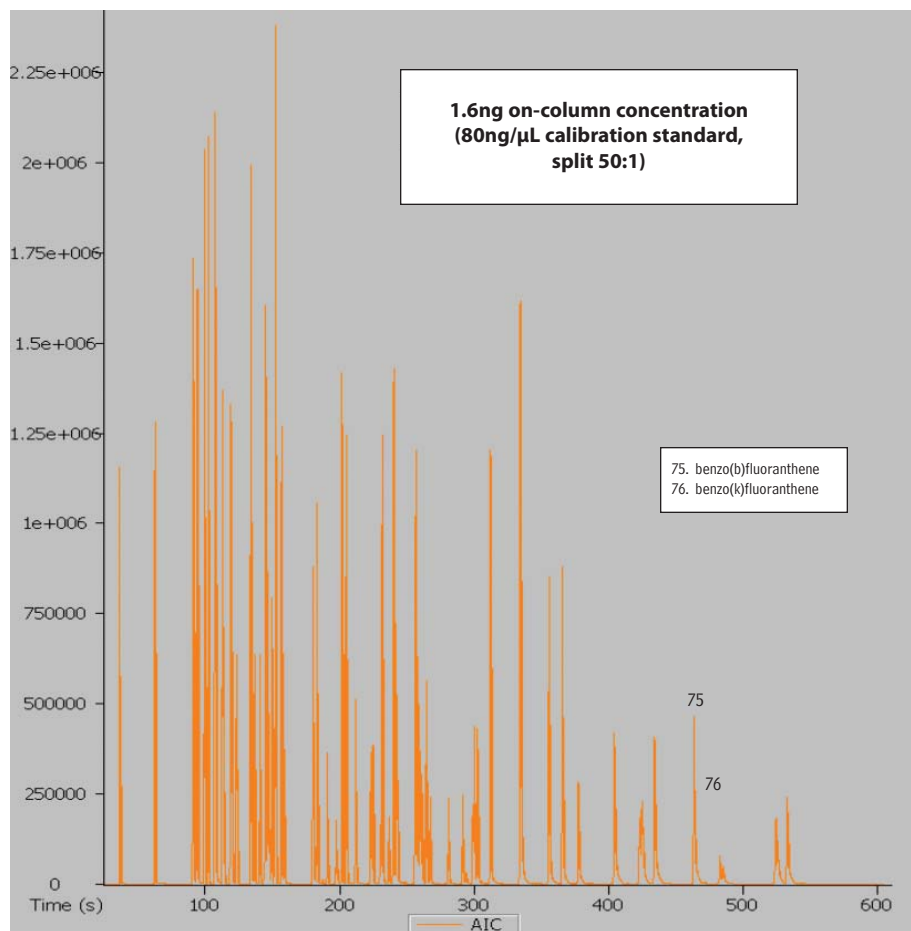
Acknowledgement

This investigation was conducted in collaboration with Jack Cochran, Director of Separation Science, LECO Corporation, Las Vegas, NV.

Table 1 Absolute retention times for semivolatile target compounds, in seconds.

Compound	T _R (sec.)
1. N-nitrosodimethylamine	36.5
2. 2-fluorophenol	62.7
3. phenol-d6	90.9
4. phenol	91.3
5. 2-chlorophenol-d4	93.9
6. bis(2-chloroethyl) ether	94.2
7. 2-chlorophenol	94.5
8. 1,3-dichlorobenzene	99.6
9. 1,4-dichlorobenzene-d4	101.8
10. 1,4-dichlorobenzene	102.4
11. 1,2-dichlorobenzene-d4	107.0
12. 1,2-dichlorobenzene	107.6
13. benzyl alcohol	108.1
14. 2-methylphenol	112.9
15. bis(2-chloroisopropyl) ether	113.7
16. N-nitrosodipropylamine	118.8
17. 4-methylphenol	119.3
18. hexachloroethane	119.8
19. nitrobenzene-d5	123.1
20. nitrobenzene	123.9
21. isophorone	134.1
22. 2-nitrophenol	136.7
23. 2,4-dimethylphenol	140.7
24. bis(2-chloroethoxy) methane	145.0
25. 2,4-dichlorophenol	146.8
26. benzoic acid	148.0
27. 1,2,4-trichlorobenzene	149.8
28. naphthalene-d8	151.6
29. naphthalene	152.5
30. 4-chloroaniline	156.8
31. hexachlorobutadiene	159.1
32. 4-chloro-3-methyl phenol	180.3
33. 2-methylnaphthalene	183.4
34. hexachlorocyclopentadiene	190.9
35. 2,4,6-trichlorophenol	197.5
36. 2,4,5-trichlorophenol	198.5
37. 2-fluorobiphenyl	201.7
38. 2-chloronaphthalene	205.1
39. 2-nitroaniline	212.1
40. dimethyl phthalate	222.9
41. acenaphthylene	223.5
42. 2,6-dinitrotoluene	224.8
43. acenaphthene-d10	230.3
44. 3-nitroaniline	231.6
45. acenaphthene	231.9
46. 2,4-dinitrophenol	236.6
47. dibenzofuran	240.3
48. 4-nitrophenol	242.2
49. 2,4-dinitrotoluene	243.0
50. fluorene	256.0
51. diethyl phthalate	256.7
52. 4-chlorophenyl phenyl ether	258.5
53. 4-nitroaniline	260.2
54. 4,6-dinitro-2-methylphenol	261.3
55. N-nitrosodiphenylamine	264.6
56. 2,4,6-tribromophenol	267.4
57. 4-bromophenyl phenyl ether	280.8
58. hexachlorobenzene	281.0
59. pentachlorophenol	291.5
60. phenanthrene-D10	299.0
61. phenanthrene	300.2
62. anthracene	302.6
63. carbazole	312.2
64. dibutyl phthalate	334.5
65. fluoranthene	355.7
66. pyrene	365.7
67. p-terphenyl-d14	377.5
68. butyl benzyl phthalate	404.4
69. benzo(a)anthracene	423.0
70. chrysene-d12	423.4
71. chrysene	424.6
72. 3,3'-dichlorobenzidine	425.4
73. bis(2-ethylhexyl) phthalate	434.3
74. di-n-octyl phthalate	463.6
75. benzo(b)fluoranthene	470.2
76. benzo(k)fluoranthene	471.4
77. benzo(a)pyrene	483.0
78. perylene-d12	485.1
79. indeno(1,2,3-cd)pyrene	524.4
80. dibenzo(a,h)anthracene	526.0
81. benzo(ghi)perylene	533.0

Figure 1 Monitor 81 semivolatile compounds and internal standards in 9 minutes.



Column: Rtx®-5SiIIMS, 10m x 0.18mm x 0.18μm, cat.# 42703
 Sample: 1μL containing SV Calibration Mix #1 (cat.# 31007), SV Calibration Mix #2 (cat.# 31008), SV Calibration Mix #3 (cat.# 31009), SV Calibration Mix #4 (cat.# 31010), SV Calibration Mix #5 (cat.# 31011), SV Calibration Mix #7 (cat.# 31013), 3,3'-Dichlorobenzidine (cat.# 31026); acids, bases, and neutrals 80ng each, internal standards 50ng each.
 Inj.: 1μL, split, 4mm Siltek® treated inlet liner with Siltek® treated wool, 250°C, split ratio 50:1, 25 sec. solvent delay
 Carrier gas: helium
 Flow rate: 2mL/min., constant flow
 Oven: 40°C (0.1 min.) to 340°C @ 30°C/min. (no hold)
 Mass spectrometer: LECO Pegasus 3 ToF-MS
 Source temp.: 250°C
 Electron ionization: 70 eV
 Stored mass range: 35 - 500 u
 Acquisition rate: 20 spectra/sec.
 Total run time: 10 min.

Rtx®-5SiI MS Columns (fused silica)

(Selectivity equivalent to Crossbond® 5% diphenyl / 95% dimethyl polysiloxane)

ID	df (μm)	temp. limits	10-Meter	20-Meter
0.18mm	0.18	-60 to 325°C	42703	42702
ID	df (μm)	temp. limits	15-Meter	30-Meter
0.25mm	0.10	-60 to 330/350°C	12705	12708
	0.25	-60 to 330/350°C	12720	12723
	0.50	-60 to 330/350°C	12735	12738
	1.00	-60 to 325/350°C	12750	12753
0.28mm	0.25	-60 to 330/350°C	12790	12793
	0.50	-60 to 330/350°C	12791	12794
	1.00	-60 to 325/350°C	12792	12795

Dr. Frank Dorman will present this information in detail at the 2005 Pittsburgh Conference in Orlando, FL. See Reference 1 on page 8 for details.

Reference Mixes

Use the mixes listed in the conditions for Figure 1, or replace the six SV calibration mixes with 8270 MegaMix™.

8270 MegaMix™ (76 components)

acenaphthene	2,4-dinitrophenol
acenaphthylene	2,4-dinitrotoluene
aniline	2,6-dinitrotoluene
anthracene	di- <i>n</i> -butyl phthalate
azobenzene ¹	di- <i>n</i> -octyl phthalate
benzo(a)anthracene	diphenylamine ²
benzo(a)pyrene	fluorene
benzo(b)fluoranthene	fluoranthene
benzo(ghi)perylene	hexachlorobenzene
benzo(k)fluoranthene	hexachlorobutadiene
benzyl alcohol	hexachlorocyclopentadiene
benzyl butyl phthalate	hexachloroethane
bis 2-ethylhexyl adipate	indeno(1,2,3- <i>cd</i>)pyrene
bis(2-chloroethoxy)methane	isophorone
bis(2-chloroethyl)ether	1-methylnaphthalene
bis(2-chloroisopropyl)ether	2-methylnaphthalene
bis(2-ethylhexyl)phthalate	2-methylphenol
4-bromophenyl phenyl ether	3-methylphenol*
carbazole	4-methylphenol*
4-chloroaniline	naphthalene
4-chloro-3-methylphenol	2-nitroaniline
2-chloronaphthalene	3-nitroaniline
2-chlorophenol	4-nitroaniline
4-chlorophenyl phenyl ether	nitrobenzene
chrysene	2-nitrophenol
dibenzo(a,h)anthracene	4-nitrophenol
dibenzofuran	N-nitrosodimethylamine
1,2-dichlorobenzene	N-nitroso-di- <i>n</i> -propylamine
1,3-dichlorobenzene	pentachlorophenol
1,4-dichlorobenzene	phenanthrene
2,4-dichlorophenol	phenol
diethyl phthalate	pyrene
dimethyl phthalate	pyridine
2,4-dimethylphenol	2,3,4,6-tetrachlorophenol
1,2-dinitrobenzene	2,3,5,6-tetrachlorophenol
1,3-dinitrobenzene	1,2,4-trichlorobenzene
1,4-dinitrobenzene	2,4,5-trichlorophenol
4,6-dinitro-2-methylphenol	2,4,6-trichlorophenol

1,000μg/mL each (except where noted) in methylene chloride, 1mL/ampul

cat. # 31850 (ea.)

*3-methylphenol and 4-methylphenol concentration is 500μg/mL.

¹1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).

²N-nitrosodiphenylamine (8270-listed analyte) decomposes to diphenylamine (mix component).

3,3'-Dichlorobenzidine

3,3'-dichlorobenzidine

2,000μg/mL in methanol, 1mL/ampul

cat. # 31026 (ea.)

please note

Many other calibration mixes, internal standards, and surrogates for analysis of semivolatile compounds are described in our catalog and on our website.

High Resolution GC/MS Separations of Dioxin or Furan Congeners

Using an Rtx®-Dioxin2 Column

by Gary Stidsen, GC Columns Marketing Manager

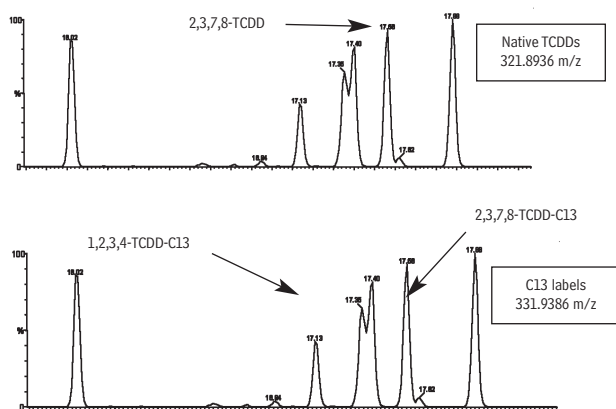


- Resolves all 2,3,7,8-substituted dioxins from each other and from non-toxic congeners.
- Resolves furan congeners from chlorodiphenyl ethers.
- Eliminates need for a second column.
- Low bleed stationary phase, stable to 320°C.

An analysis for dioxins or furans typically includes extensive sample extract cleanup, followed by high-resolution mass spectrometry, and a primary requirement of the analytical column is complete separation of the toxic dioxin or furan congeners (substitutions in the 2, 3, 7, and 8 positions). Unfortunately, separation of the toxic congeners from the non-toxic congeners proves difficult on almost all stationary phases.

Quantification for some target congeners is inaccurately high, due to coelution with non-toxic congeners. The coelution issue has resulted in the use of confirmation columns, most commonly high cyanopropyl stationary phases, in order to more accurately quantify the toxic congeners. Unfortunately, cyanopropyl columns exhibit poor thermal stability, and therefore offer poor lifetime in this application.

Figure 1 2,3,7,8-Tetrachlorodibenzodioxin resolved from other TCDD congeners, using an Rtx®-Dioxin2 column.



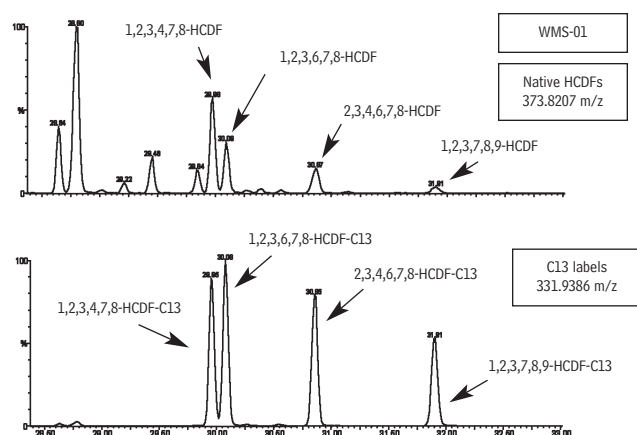
Column and Conditions for Figures 1 and 2:

Column: 60m, 0.25mm ID, 0.25µm Rtx®-Dioxin2 (cat. # 10758)

Oven temp.: 130°C (hold 1 min.) to 205°C @ 45°C/min. to 305°C @ 6°C/min. (hold 30 min.);
Dead time: 2.89 min.; Carrier gas: helium at 1.5mL/min., constant flow

GC_EV00702

Figure 2 Hexachlorodibenzofuran congeners resolved by an Rtx®-Dioxin2 column.



Chromatograms courtesy of Karen MacPherson and Eric Reiner, Ontario Ministry of the Environment, Etobicoke, Ontario, Canada.

WMS-01 and HCDF reference material courtesy of Wellington Laboratories, Guelph, Ontario, Canada.

GC_EV00703

Rtx®-Dioxin2 Columns (fused silica)

ID	df (µm)	temp. limits	40-Meter	60-Meter
0.18mm	0.18	20°C to 320°C	10759	—
0.25mm	0.25	20°C to 320°C	—	10758

With these problems in mind, Restek chemists developed the Rtx®-Dioxin2 capillary GC column. This column effectively resolves the 2,3,7,8-substituted congeners from each other and from non-toxic congeners. Figure 1 shows the separation of the tetrachlorodibenzodioxins on a 60-meter Rtx®-Dioxin2 column. 2,3,7,8-TCDD is well resolved from the other congeners in this group and can be quantified accurately.

Coelutions of toxic and non-toxic congeners also can make quantification of the hexachlorodibenzofurans difficult, but an Rtx®-Dioxin2 column resolves furan congeners as effectively as dioxins. Figure 2, a chromatogram for the HCDF congener group in reference material WMS-01, shows the congeners are very well resolved. Values for 1,2,3,4,7,8-hexachlorodibenzofuran, or for other congeners, compare favorably with values from the other columns typically used for this analysis.

We can provide elution orders for all of the commonly analyzed congeners, and chromatograms for each congener group in the WMS-01 reference material. If you would like this detailed information, or additional information about Rtx®-Dioxin2 columns, please contact us.

Pittcon® presentation

This information will be presented by Dr. Frank Dorman, Oral Session 1000, Tuesday afternoon, March 1.

Plan to attend Dioxin 2005, and visit Restek!



Stable, Low-Bleed Rtx®-XLB Columns

For Maximum Performance from High-Sensitivity GC/MS Systems

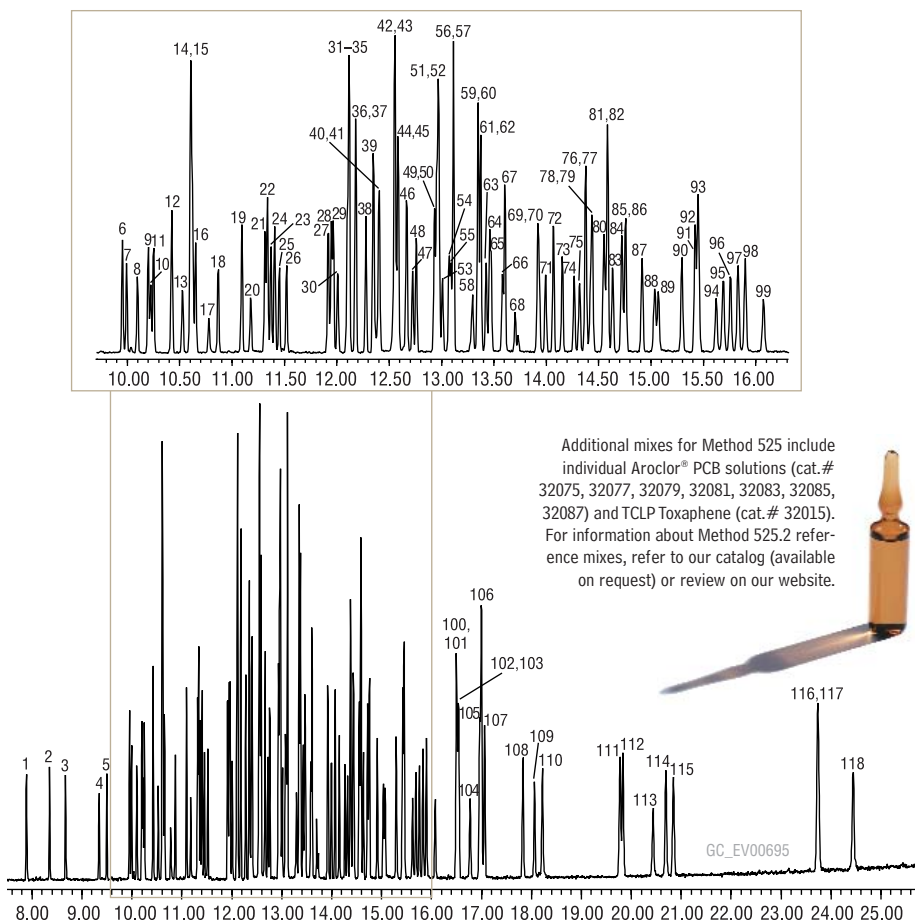
by Gary Stidsen, GC Columns Marketing Manager

- Unique low bleed polymer—ideal for low-level GC/MS.
- Excellent for semivolatiles analysis; equivalent to other “XLB” phases.
- Stable to 360°C; inert to active compounds.
- In stock, ready for delivery.

Use Rtx®-XLB columns in GC/MS analyses requiring a low bleed stationary phase at oven temperatures as high as 360°C. Applications for this stable, low polarity phase include

organochlorine pesticides and herbicides, PCBs, and mixed semivolatiles (Figure 1). The selectivity of Rtx®-XLB columns matches that of DB®-XLB columns.

Figure 2 An Rtx®-XLB column offers low bleed, inertness, and good resolution of semivolatile environmental pollutants.



Rtx®-XLB Columns (fused silica) (proprietary low-polarity phase)

ID	df (μm)	temp. limits*	15-Meter	30-Meter	60-Meter
0.25mm	0.10	30 to 340/360°C		12808	
	0.25	30 to 340/360°C	12820	12823	12826
	0.50	30 to 340/360°C		12838	
	1.00	30 to 340/360°C	12850	12853	
0.32mm	0.10	30 to 340/360°C		12809	
	0.25	30 to 340/360°C	12821	12824	12827
	0.50	30 to 340/360°C		12839	
	1.00	30 to 340/360°C		12854	
0.53mm	0.50	30 to 340/360°C		12840	
	1.50	30 to 340/360°C	12867	12870	
ID	df (μm)	temp. limits	12-Meter	20-Meter	25-Meter
0.18mm	0.18	30 to 340/360°C		42802	
0.20mm	0.33	30 to 340/360°C	42815		42820

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Every Rtx®-XLB column undergoes rigorous quality assurance evaluation for film thickness, bleed (at 340°C), inertness, plates per meter, and retention time indices, to ensure every column we ship meets exacting performance standards and performance is reproduced from column to column. These measures assure you of the highest quality GC/MS column on the market.

1. isophorone
2. 2-nitro-*m*-xylene
3. dichlorvos
4. hexachlorocyclopentadiene
5. EPTC
6. butylate
7. mevinphos
8. vernolate
9. pebulate
10. etridiazole (Terrazole®)
11. dimethylphthalate
12. acenaphthene
13. 2,6-dinitrotoluene
14. acenaphthene-d10
15. 2-chlorobiphenyl (BZ#1)
16. chloroneb
17. tebuthiuron
18. molinate
19. diethyl phthalate
20. 2,4-dinitrotoluene
21. propachlor
22. fluorene
23. ethoprop
24. cycloate
25. trifluralin
26. chlorpropham
27. 2,3-dichlorobiphenyl (BZ#5)
28. atraton
29. prometon
30. α-BHC
31. hexachlorobenzene
32. propazine
33. simazine
34. atrazine
35. metribuzin
36. diazinon
37. terbufos
38. pronamide
39. pentachlorophenol
40. β-BHC
41. disulfoton
42. terbacil
43. phenanthrene-d10
44. methyl parathion OA
45. phenanthrene
46. anthracene
47. γ-BHC (lindane)
48. 2,4,5-trichlorobiphenyl (BZ#29)
49. alachlor
50. prometryne
51. ametryn
52. simetryn
53. δ-BHC
54. heptachlor
55. chlorothalonil
56. di-*n*-butylphthalate
57. terbutryn
58. bromacil
59. chlorpyrifos
60. metolachlor
61. DCPA methyl ester (Dacthal®)
62. 2,2',4,4'-tetrachlorobiphenyl (BZ#47)
63. aldrin
64. triadimefon
65. cyanazine (Bladex)
66. MGK-264
67. diphenamid
68. merphos
69. 2,2',3',4,6-pentachlorobiphenyl (BZ#98)
70. heptachlor epoxide (isomer B)
71. heptachlor epoxide (isomer A)
72. butachlor
73. stirofos (tetrachlorvinphos)
74. fenamiphos
75. α-chlordane
76. napropamide
77. γ-chlordane
78. endosulfan I
79. *trans*-nonachlor
80. pyrene-d10
81. pyrene
82. 4,4'-DDE
83. 2,2',4,4',5,6'-hexachlorobiphenyl (BZ#154)
84. *p*-terphenyl-d14
85. dieldrin
86. carboxin
87. chlorbenzilate
88. tricyclozole
89. endrin
90. 4,4'-DDD
91. bis(2-ethylhexyl)adipate
92. butyl benzyl phthalate
93. endosulfan II
94. endrin aldehyde
95. norflurazon
96. 4,4'-DDT
97. triphenylphosphate
98. hexazinone
99. endosulfan sulfate
100. bis(2-ethylhexyl)phthalate
101. methoxychlor
102. 2,2',3,3',4,5,6,6'-octachlorobiphenyl (BZ#207)
103. 2,2',3,3',4,4',6-heptachlorobiphenyl (BZ#171)
104. endrin ketone
105. benzo(a)anthracene
106. chrysene-d12
107. chrysene
108. fenarimol
109. *cis*-permethrin
110. *trans*-permethrin
111. benzo(b)fluoranthene
112. benzo(k)fluoranthene
113. fluridone (Sonar®)
114. benzo(a)pyrene
115. perylene-d12
116. dibenzo(a,h)anthracene
117. indeno(1,2,3-*cd*)pyrene
118. benzo(ghi)perylene

Column: Rtx®-XLB, 30m, 0.25mm ID, 0.25μm (cat. # 12823)
 Sample: US EPA Method 525 analytes, 1μL 5ng per analyte
 reference mixes used: 31824, 32420, 32421, 32422, 32423, 31825, 31826, 31828, 32291, 32415, 32436.
 Inj.: pressure pulsed (0.4 min. @ 30psi), splitless (hold 0.4 min.), 4mm Drilled Uniliner® inlet liner (cat.# 21055)
 Inj. Temp.: 300°C
 Carrier Gas: helium, constant flow
 Flow Rate: 1.0mL/min.
 Oven Temp.: 35°C (hold 2 min.) to 260°C @ 20°C/min. (hold 0 min.) to 330°C @ 6°C/min. (hold 5 min.)
 Det: Agilent 5973 GC/MS
 Transfer Line Temp.: 280°C
 Scan Range: 45–550 amu
 Solvent Delay: 4.7 min.
 Tune: DFTPP

Enhanced Rtx®-1PONA GC Column Improves Detailed Hydrocarbon Analysis

Guaranteed Retention - Efficiency - Peak Symmetry - Selectivity

by Gary Stidsen, GC Columns Marketing Manager, and Barry Burger, Petroleum Applications Chemist

- Reduce analysis time by 30%!
- Selectivity specific for detailed hydrocarbon analysis (ASTM Method D-6730-0).
- Each column tested to meet method-specific resolution criteria.
- Unsurpassed peak symmetry for oxygenated compounds.

To meet the demanding resolution and retention criteria in American Society for Testing and Materials (ASTM) and Canadian General Standards Board (CGSB) methodology for detailed hydrocarbon analysis, Restek chemists reformulated the Rtx®-1PONA column. The enhanced column meets or exceeds all criteria in these standardized methods, in 30% less time: retention time for C13 is 97 minutes, using

helium as the carrier gas. Measured values for retention (k), efficiency (n), peak symmetry, and stationary phase selectivity (RI) are stringently controlled, enabling us to guarantee performance and reproducibility from column to column.

To achieve critical resolutions in detailed hydrocarbon analysis, a 5-meter 5% diphenyl/95% dimethyl polysiloxane tuning column is connect-

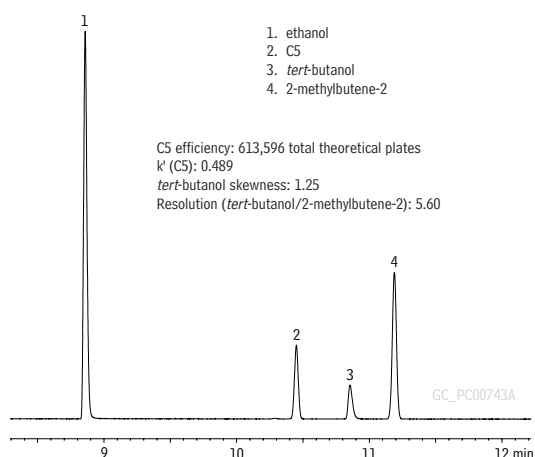
award-winning **innovation!**



**2004
Concluded
Research
Award goes to
Barry Burger**

The award was presented at the 2004 Gulf Coast Conference, for Restek's second generation Rtx®-1PONA capillary GC column for detailed hydrocarbon analysis according to American Society for Testing and Materials and Canadian General Standards Board methodology. For the full story, visit us online.

Figure 1 Sharp, symmetric peak for ethanol (gasoline oxygenate), using an Rtx®-1PONA column.



Rtx®-1PONA, 100m, 0.25mm ID, 0.5µm (cat.# 10195)
w/ Rtx®-5 tuning column, 2.62m, 0.25mm ID, 1.0µm,
connected via Press-Tight® connector (cat.# 20446)

Sample: custom detailed hydrocarbon analysis (DHA) mix, neat
Inj.: 0.01µL, split (split ratio 150:1),
4mm cup inlet liner (cat.# 20709)
Inj. temp.: 200°C
Carrier gas: helium, constant flow
Linear velocity: 28cm/sec. (2.3mL/min.)
Oven temp.: 35°C
Det.: FID @ 250°C

Pittcon® presentation

This information will be presented by Barry Burger, Poster Session 10, Sunday afternoon, February 27.

ed to the analytical column and adjusted to the needed length through a series of trial analyses.

This work earned the Restek Innovations chemists the Concluded Research Award, sponsored by DCG Partnership 1 Ltd., at the 2004 Gulf Coast Conference. When you use an Rtx®-1PONA column, we think you'll agree that the award is well justified.

Rtx®-1PONA Column (fused silica)

(Crossbond® 100% dimethyl polysiloxane)*

ID	df (µm)	temp. limits	100-Meter
0.25mm	0.50	-60 to 300/340°C	10195

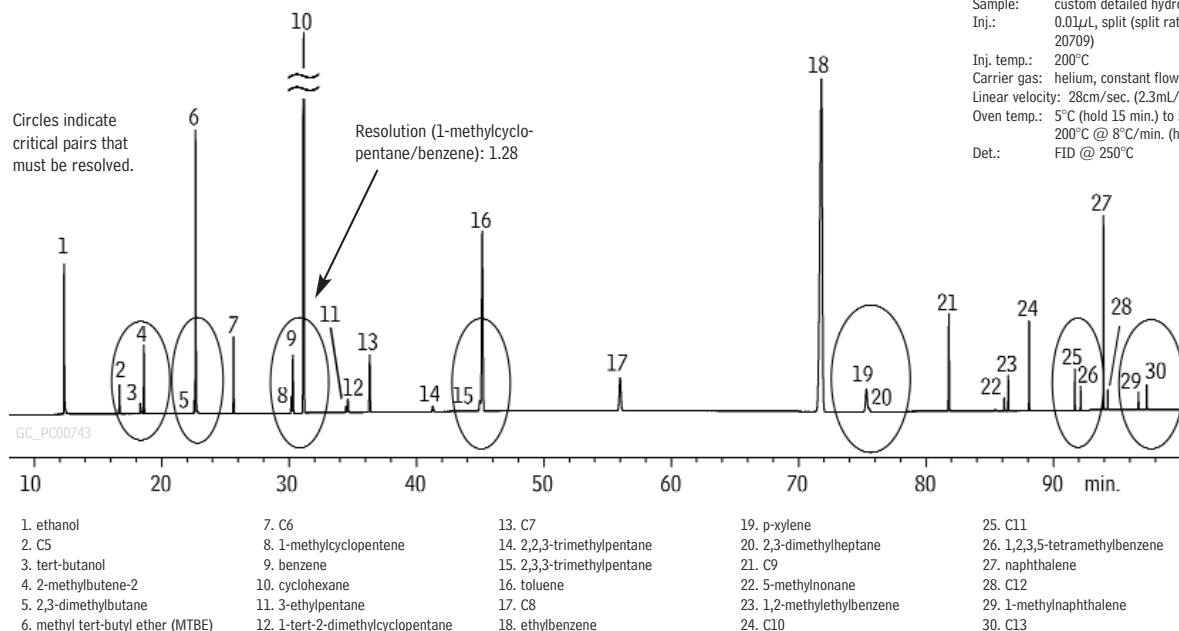
*Optimized phase for hydrocarbon analysis

Rtx®-5PONA Tuning Column (fused silica)

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

ID	df (µm)	temp. limits	5-Meter
0.25mm	1.0	-60 to 330/340°C	10196

Figure 2 Critical pairs of gasoline components resolved per ASTM specifications, using an Rtx®-1PONA column.



**Reliable connections
made simple!**
See page 22 for
information on
Vu2Union™
Connectors.

New GC Column for PCB Congeners or Aroclor® Mixes: Rtx®-PCB

Exclusive Polymer with Unique Selectivity

by Gary Stidsen, GC Columns Marketing Manager

- Unique polymer for PCBs analysis by GC/ECD or GC/MS.
- Good results for other semivolatiles.
- Low polarity and inertness for active compounds.
- Thermally stable to 340°C.

Rtx®-PCB columns contain a proprietary polymer that has provided unique separations for PCB congeners, and can be used with electron capture detection or mass spectrometry. Figure 1 shows the excellent peak shape obtained for the PCB congeners in several Aroclor® mixes. In our initial review of data for these columns, we dis-

covered that by using an Rtx®-PCB column in a GC/MS analysis, European PCB congeners can be analyzed without interference from other congeners. Table 1 is a shortened list of the PCB congeners, showing those that elute near the European PCB congeners, which are indicated by an "x".

Table 1 Coelutions do not interfere with analysis of European PCB congeners ("x") on Rtx®-PCB: only congeners not measurable by MS are indicated in boxes.

Eur	IUPAC #	Cl #	T _R (min.)	Resolution	Assessment
	53	4	14.11		
	31	3	14.14	0.5	
x	28	3	14.23	1.4	Measurable by MS
	33	3	14.27	0.6	
	51	4	14.29	0.4	
	20	3	14.30	0.1	
	45	4	14.54	3.8	
	46	4	14.71		
	43	4	14.88	2.7	
x	52	4	14.94	1.0	Measurable by MS
	48	4	15.01	1.2	
	49	4	15.08	1.0	
	89	5	17.29		
	84	5	17.30	0.2	
	56	4	17.34	0.6	
x	101	5	17.35	0.2	Measurable by MS
	99	5	17.50	2.3	
	60	4	17.52	0.4	
	123	5	19.18		
	109	5	19.19	0.2	
	134	6	19.24	0.7	
	133	6	19.28	0.7	
x	118	5	19.35	1.0	Measurable by MS
	131	6	19.35	0.0	
	146	6	19.47	1.9	
	122	5	19.53	1.0	
	114	5	19.65	1.8	
x	153	6	19.66	0.2	
	132	6	19.77	1.7	
	179	7	19.88	1.8	
	130	6	20.31		
	164	6	20.33	0.3	
	178	7	20.45	1.9	
x	138	6	20.47	0.3	Measurable by MS
	163	6	20.51	0.6	
	129	6	20.56	0.7	
	158	6	20.60	0.6	
	172	7	21.99		
	156	6	22.07	1.3	
	157	6	22.18	1.6	
x	180	7	22.19	0.3	Major congener 180 should not be terribly biased by 193.
	193	7	22.23	0.6	
	200	8	22.30	1.1	
	191	7	22.37	1.1	

Mix of Aroclor® 1242-1254-1262 used for resolution check.

Relaxed resolution criteria based on visual inspection of closely eluting congeners.

Rtx®-PCB Columns (fused silica)

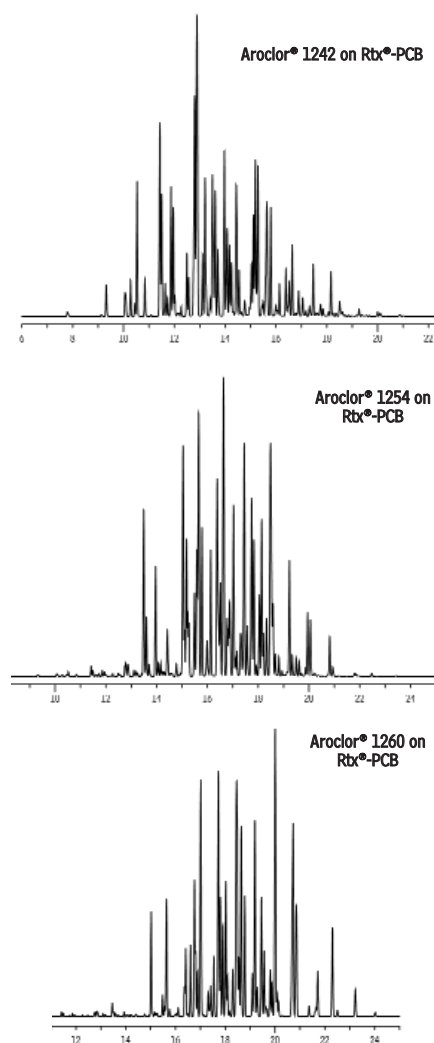
ID	df (μm)	temp. limits*	20-Meter	30-Meter	60-Meter
0.18mm	0.18	30°C to 320/340°C	41302		41304
0.25mm	0.25	30°C to 320/340°C		13223	13226
0.32mm	0.50	30°C to 320/340°C		13239	



new!
unique polymer
technology

Rtx®-PCB is the newest member of a family of new polymer phases that undergo rigorous quality assurance measures to ensure every column meets exacting standards and that performance is reproduced from column to column. Specified column parameters include film thickness, bleed (at 320°C), inertness, plates per meter, and retention time indices. These measures assure you of the highest quality columns available.

Figure 1 Excellent separation and peak shape for PCBs in three Aroclor® mixes, using an Rtx®-PCB column.



Column: Rtx®-PCB 30m, 0.25mm ID, 0.25μm (cat.# 13223)
 Sample: 200ng/mL Aroclor® 1242 (cat.# 32009); Aroclor® 1254 (cat.# 32011); Aroclor® 1260 (cat.# 32012)
 Inj.: 1.0μL splitless (hold 0.75 min.), 3.5mm ID single gooseneck inlet liner (cat.# 20962)
 Inj. temp.: 250°C
 Carrier gas: hydrogen, constant pressure
 Linear velocity: 71cm/sec. @ 110°C
 Oven temp.: 100°C (hold 1.0 min.) to 300°C @ 10°C/min. (hold 4 min.)
 Det.: ECD @ 310°C

Fast GC/MS Analysis of Semivolatile Organic Compounds

Using a 0.18mm ID / 0.36 μ m Film Rtx®-5Sil MS Column

by Gary Stidsen, GC Columns Marketing Manager

- Meets resolution and response factors for many methods, including EPA Method 8270.
- Split injections and thick phase film help prolong column life when analyzing dirty extracts.
- Low bleed, thermally stable.
- Column and method optimized for conventional scanning detectors, such as Agilent 5973.

A high sample throughput is important to most analysts, and is essential to those in environmental laboratories. Chromatographers following US EPA methods 8270, 625, or 525, or other methods for equivalent lists of semivolatile organic pollutants, now can take advantage of advances in mass spectrometer and GC column technology, to reduce analytical time and increase sample throughput while also obtaining good lifetimes from their columns.

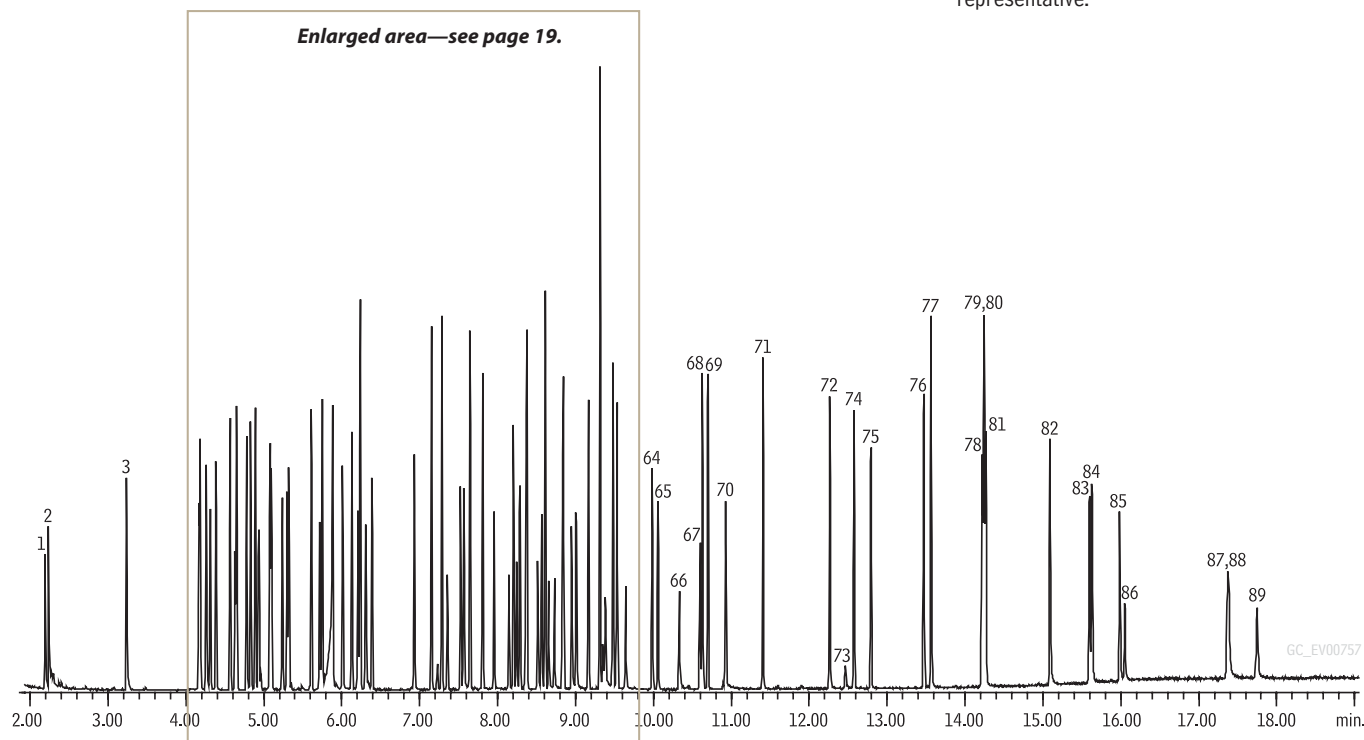
The new generation of high sensitivity mass spectrometers enables analysts to reduce the amount of sample injected onto the column. In analyses of semivolatiles, this has led to using split injections and columns having smaller internal diameter, resulting in shorter analysis times. Split injection contributes to good chromatographic resolution, but also helps improve sensitivity by reducing the degradation of acidic and basic compounds in the hot injection port.

Further, because less sample is transferred onto the column in split injections, transfer of non-volatile contaminants from the extracts is minimized, and column lifetime is prolonged.

In our laboratories, we have been evaluating various combinations of column length, ID, and film thickness for suitability for shortening analysis time while meeting the resolution requirements of the various methods. The latest product of our research is the 20m, 0.18mm ID, 0.36 μ m film Rtx®-5Sil MS column. Figure 1 shows the results of an analysis on this new column. Under the conditions listed, resolution of these 89 compounds meets the resolution criteria of method 8270, 625, or 525. A split injection was used to obtain Figure 1, but the column also can be used with splitless injections.

The 20m, 0.18mm ID, 0.36 μ m film Rtx®-5Sil MS column is the latest product of our application lab chemists' continuing efforts to combine rapid analysis with good column lifetime, and give you the best available column for analyzing semivolatile pollutants. To discuss this application, call our technical support staff at 800-356-1688 or 814-353-1300, ext. 4, or contact your Restek representative.

Figure1 89 semivolatile pollutants, surrogates, and internal standards separated in less than 18 minutes on a 20m, 0.18mm ID, 0.36 μ m film Rtx®-5Sil MS column.



- | | |
|----------------------------------|-------------------------------------|
| 1. N-nitrosodimethylamine | 75. <i>p</i> -terphenyl-d14 |
| 2. pyridine | 76. butyl benzyl phthalate |
| 3. 2-fluorophenol | 77. bis(2-ethylhexyl)adipate |
| peaks 4-63: see page 19 | 78. benzo(a)anthracene |
| 64. 4-bromophenyl phenyl ether | 79. chrysene-d12 |
| 65. hexachlorobenzene | 80. bis(2-ethylhexyl)phthalate |
| 66. pentachlorophenol | 81. chrysene |
| 67. phenanthrene-d10 | 82. di- <i>n</i> -octyl phthalate |
| 68. phenanthrene | 83. benzo(b)fluoranthene |
| 69. anthracene | 84. benzo(k)fluoranthene |
| 70. carbazole | 85. benzo(a)pyrene |
| 71. di- <i>n</i> -butylphthalate | 86. perylene-d12 |
| 72. fluoranthene | 87. indeno(1,2,3- <i>cd</i>)pyrene |
| 73. benzidine | 88. dibenzo(a,h)anthracene |
| 74. pyrene | 89. benzo(ghi)perylene |

Column: Rtx®-5Sil MS, 20m, 0.18mm ID, 0.36 μ m (cat.# 557810)
 Sample: US EPA Method 8270D analytes, 10ppm each (10ng on column); 8270 MegaMix™ (cat.# 31850); Benzoic Acid (cat.# 31415); 2,4-Dinitrophenol (cat.# 31291); Acid Surrogate Mix (4/89 SOW) (cat.# 31063); B/N Surrogate Mix (4/89 SOW) (cat.# 31062)
 Inj.: 1.0 μ L, splitless, 4mm ID gooseneck splitless inlet liner (cat.# 20798), splitless hold time 0.20 min., pressure pulse 0.15 min. @ 30psi
 GC: Agilent 6890
 Inj. temp.: 250°C
 Carrier gas: helium, constant flow
 Flow rate: 1.2mL/min.
 Oven temp.: 50°C(hold 0.5 min.) to 330°C @ 18°C/min. (hold 3 min.)
 Det.: Agilent 5973 GC/MS
 Transfer line temp.: 280°C
 Scan range: 35-550 amu
 Solvent Delay: 1 min.
 Tune: DFTPP
 Ionization: EI

New Rtx®-440 GC Column, for Rapid Analysis of Pesticides, PAHs or Other Semivolatiles

Exclusive Stationary Phase from Restek

by Gary Stidsen, GC Columns Marketing Manager

- Unique selectivity compared to other phases.
- Ideal polarity for pesticides, many other semivolatile compounds.
- Low bleed, thermally stable to 340°C—excellent for trace analysis by GC/MS.

new!

New Rtx®-440 stationary phase exhibits unique selectivity at an intermediate polarity.

Applications testing we have performed to date includes organochlorine pesticides, polycyclic

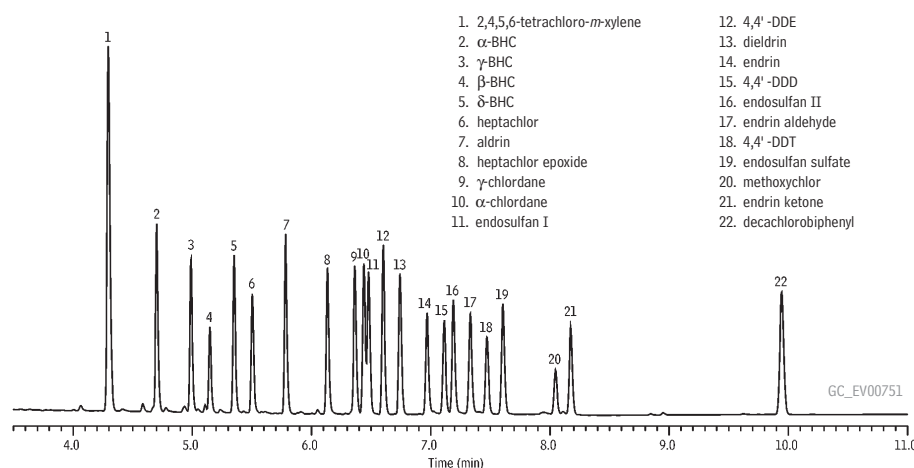
aromatic hydrocarbons (PAHs), and semivolatile environmental compounds (US EPA Method 8270). The new column resolves 20 commonly monitored organochlorine pesticides in 10 min-

utes (Figure 1), or the 34 organochlorine pesticides listed in US EPA Method 8081 in less than 13 minutes.

We also have had very promising results with analyzing PAHs on Rtx®-440 columns, as shown in Figure 2. Phenanthrene and anthracene (peaks 5 and 6), benzo(a)anthracene and chrysene (peaks 9 and 10) and benzo(b)fluoranthene and benzo(k)fluoranthene (peaks 11 and 12) are resolved well, as is a more difficult pair of analytes: indeno(1,2,3-cd)pyrene and dibenzo(a,h)anthracene (peaks 14 and 15). The analysis is complete in less than 18 minutes.

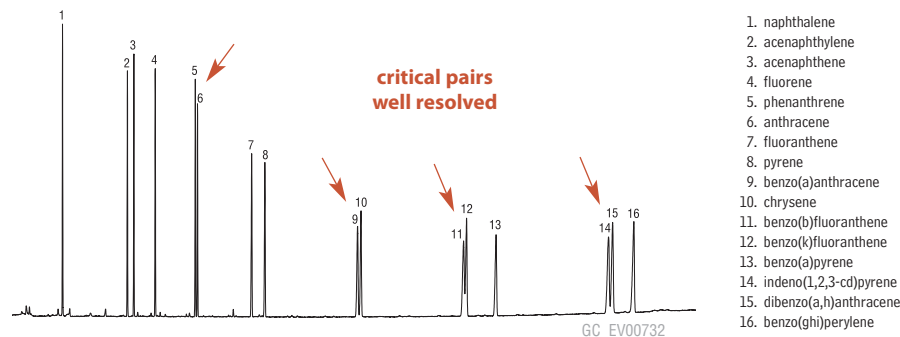
Our chemists are continuing their applications research with this new phase. Please call our Technical Service team to obtain the latest information about Rtx®-440 columns.

Figure 1 Separate 20 organochlorine pesticides in 10 minutes, using an Rtx®-440 column.



Column: Rtx®-440 30m, 0.32mm ID, 0.50 μ m (cat. # 12939)
 Sample: 50ng/mL Organochlorine Pesticides Mix AB standard (cat.#32291) & 50ng/mL Pesticide Surrogate Mix (cat.#32000) in hexane
 Inj.: 1.0 μ L splitless (hold 1.0 min.), 3.5mm ID single gooseneck inlet liner (cat.# 20962)
 Inj. temp.: 250°C
 Carrier gas: hydrogen, constant pressure
 Linear velocity: 71cm/sec. @ 110°C
 Oven temp.: 110°C (hold 0.5 min.) to 268°C @ 30°C/min. to 290°C @ 11°C/min. to 320°C @ 25°C/min. (hold 5 min.)
 Det.: ECD @ 310°C

Figure 2 Analyze 16 PAHs in 22 minutes, and resolve critical pairs, with an Rtx®-440 column.



Column: Rtx®-440 30m, 0.25mm ID, 0.25 μ m (cat.# 12923)
 Sample: 610 PAH Mix (cat.# 31011) diluted to 20ppm each compound in methylene chloride
 Inj.: 1.0 μ L splitless (hold 0.4 min.), 4mm splitless liner (cat.# 20772)
 Inj. temp.: 320°C
 Carrier gas: hydrogen, constant flow
 Flow: 3.6mL/min.
 Oven temp.: 40°C (hold 2 min.) to 240°C @ 30°C/min., to 320°C @ 8°C/min. (hold 5 min.)
 Det.: FID @ 320°C



Rtx®-440 Columns (fused silica)

(proprietary intermediate-polarity Crossbond® phase)
 Temp. limits: 20°C to 320/340°C

ID	df (μ m)	30-Meter
0.25mm	0.25	12923
	0.50	12938
0.32mm	0.25	12924
	0.50	12939
0.53mm	0.50	12940
	1.00	12955



Organochlorine Pesticide Mix AB #1

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

200 μ g/mL each in hexane:toluene (1:1), 1mL/ampul
 cat. # 32291 (ea.)

Pesticide Surrogate Mix

decachlorobiphenyl 2,4,5,6-tetrachloro-*m*-xylene
 200 μ g/mL each in acetone, 1mL/ampul
 cat. # 32000 (ea.)

SV Calibration Mix #5 / 610 PAH Mix

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

2,000 μ g/mL each in methylene chloride, 1mL/ampul
 cat. # 31011 (ea.)

New Reference Mixes for Determination of Chlorinated Disinfection Byproducts, Chlorinated Solvents, or Halogenated Pesticides in Drinking Water

by John Lidgett, Analytical Reference Materials Technical Specialist

- Complete set of high concentration reference materials for US EPA Method 551.1.
- Target pesticides/herbicides at equal concentration, for GC/MS analysis.
- Chloral hydrate and metribuzin offered as separate solutions, for assured stability.



Chlorine has been used to disinfect drinking water for many years. Chlorinating agents, however, can form harmful and potentially carcinogenic byproducts with organic compounds in water, and this potential led to US Environmental

Protection Agency regulation in 1979.¹ Extensive research has been done on the origination of disinfection byproducts (DBPs), and on preventing their formation. DBPs can form by reaction of chlorine with naturally present organic compounds in water, such as humic acid or fulvic acid - organic compounds found in water as a result of decomposition of plant matter. Disinfection byproducts include 3 groups of compounds: trihalomethanes (THMs), haloacetonitriles, and a mixed group that includes chloral hydrate, chloropicrin, and chloropropanones. Many other DBPs, including haloacetic acids, haloacetaldehydes, cyanogen halides, aldehydes, ketoacids, chlorite, bromate, and other organic and inorganic compounds also have been identified in chlorinated or ozonated drinking water.²

Several US EPA methods regulate the monitoring of drinking water, including methods 502.2, 524.1, 551.1, and 552.2. In addition to THMs, Method 551.1 is followed for monitoring chlorinated solvents and halogenated pesticides/herbicides. EPA Method 551 requires liquid-liquid extraction with methyl-*tert*-butyl ether (MTBE) as a primary extraction solvent and analysis by GC, using electron capture detection (ECD). The latest version of Method 551, Method 551.1, allows pentane to be used as the extraction solvent.

cyanopropylphenyl / 94% dimethyl polysiloxane stationary phase Restek column, Rtx®-1301 (30m, 0.25mm ID, 1.0µm film, cat.# 16053).

Restek chemists have formulated three new calibration mixes, Disinfection Byproducts & Chlorinated Solvents Mix (cat.# 30615), Disinfection Byproducts Mix (cat.# 30616), and Method 551.1 Pesticide/Herbicide Mix (cat.# 32438), to include all but two Method 551.1 target compounds, based on enhanced stability and the testing requirements of our customers. We prepare the three new solutions in acetone, because methanol causes degradation of most haloacetonitriles, and acetone should be used for primary dilution in preparing working solutions. Bromochloroacetonitrile, a target compound we include in two of our new mixes (Disinfection Byproducts & Chlorinated Solvents Mix, Disinfection Byproducts Mix) is not available commercially at purity higher than 89%. Dichloroacetonitrile and dibromoacetonitrile are both target compounds in the calibration mixes and impurities in bromochloroacetonitrile, at 0.5 to 2.2%. After careful review, we determined that Method 551.1 allows a 4% concentration error and, based on this information, we have included bromochloroacetonitrile in both reference materials after compensating for the impurities.

Because chloral hydrate is unstable, due to hydrogen-bond interactions with halide ions, we offer it as a separate solution. After several months of stability studies, using various solvents, we determined that chloral hydrate should be offered in acetonitrile, and we seal the reference material in light-resistant ampuls as defined in the United States Pharmacopoeia (USP). When using chloral hydrate all working solutions and glassware should be free of alkaline substances and the reference material

new!



For stability, we also offer another compound in this interest group, metribuzin, as a separate mix (cat.# 32436). In earlier studies we determined that metribuzin could react with certain pesticides/herbicides at high concentration.

We offer an internal standard, bromofluorobiphenyl, in acetone, per method recommendations. The method recommends the use of a laboratory performance check (LPC) solution in MTBE, the extraction solvent. The check solution is a mix of method analytes used to evaluate the performance of the instrument. The parameters evaluated are instrument sensitivity, chromatographic performance, column performance, and analyte breakdown. Special care must be taken when analyzing endrin, a component in our new pesticide/herbicide mix, because it can break down to aldo and keto derivatives on contact with active metal sites in the injection port. The analyzed value of each compound in the check solution should be 95% to 105% of its expected value. For analysts using pentane as the extraction solvent, we offer the laboratory performance check solution in pentane. Analysis of the LPC solution is especially difficult because of the substantial range in concentration (0.2 to 83 µg/mL) of the components. Because of the high sensitivity and narrow range of linear detection of the ECD, and the possibility of coelution of solvent impurities with some of the target compounds, we use high purity MTBE and pentane in preparing the LPC solutions.

Our complete set of reference materials for determining Method 551.1 target compounds is listed on page 17. If you are analyzing for disinfection byproducts, chlorinated solvents, or chlorinated pesticides/herbicides, we highly recommend these carefully prepared standards. We also offer Rtx®-1 and Rtx®-1301 capillary columns, which are ideal for the analysis, and are listed in Method 551.1.



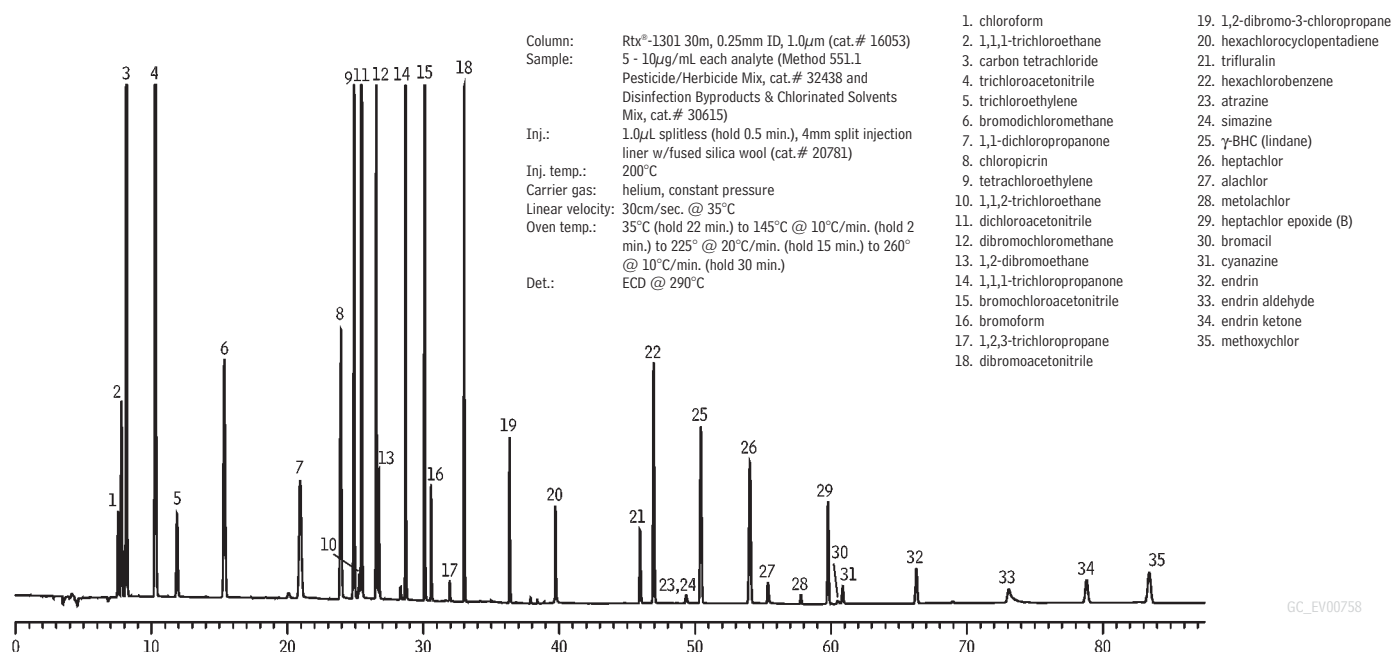
"some" promos / Products / Offers in the ADVNews have been since been progressively superseded / UPDATED OR Since Discontinued

CHECK THE latest Restek ADVantage Newsletter, Restek ESSENTIALS ... Or The Restek Catalog ... Or other Restek publications for updates

www.chromtech.net.au or NEW site 2015 > www.chromalytic.net.au



Australian Distributors
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Figure 1 Use an Rtx®-1301 column for optimal separation of disinfection byproducts.

Method 551.1 Pesticide/Herbicide Mix

alachlor	heptachlor epoxide (isomer B)
atrazine	hexachlorobenzene
bromacil	hexachlorocyclopentadiene
cyanazine (Bladex)	methoxychlor
endrin	metolachlor
endrin aldehyde	simazine
endrin ketone	trifluralin
g-BHC (Lindane)	
heptachlor	

1,000µg/mL each in acetone, 1mL/ampul
 cat. # 32438 (ea.)

Disinfection by-Product and Chlorinated Solvents Mix

bromochloroacetonitrile	1,2-dibromoethane[EDB]
bromodichloromethane	dichloroacetonitrile
bromoform	1,1-dichloro-2-propanone
carbon tetrachloride	tetrachloroethylene
chloroform	trichloroacetonitrile
chloropicrin	1,1,1-trichloroethane
dibromoacetonitrile	1,1,2-trichloroethane
dibromochloromethane	trichloroethylene
1,2-dibromo-3-chloropropane[DBCP]	1,2,3-trichloropropane
	1,1,1-trichloro-2-propanone

2000µg/mL each in acetone, 1mL/ampul
 cat. # 30615 (ea.)

Disinfection by-Product Mix

bromochloroacetonitrile	1,1-dichloro-2-propanone
chloropicrin	trichloroacetonitrile
dibromoacetonitrile	1,1,1-trichloro-2-propanone
dichloroacetonitrile	

2000µg/mL each in acetone, 1mL/ampul

Laboratory Performance Check Solution/ Pentane Extract

alachlor	83µg/mL	endrin	30
g-BHC	0.2	hexachlorocyclopentadiene	20
bromacil	83	trichloroethylene	30
bromodichloromethane	30		

In pentane, 1mL/ampul

cat. # 32442 (ea.)

Metribuzin

metribuzin

1,000µg/mL in acetone, 1mL/ampul

1,000 cat. # 32436 (ea.)

551.1 Internal Standard

1-bromo-4-fluorobenzene

1,000µg/mL in acetone, 1mL/ampul

cat. # 31854 (ea.)

551.1 Surrogate Standard

decafluorobiphenyl

1,000µg/mL in acetone, 1mL/ampul

cat. # 31855 (ea.)

Chloral Hydrate

chloral hydrate

1,000µg/mL in acetonitrile, 1mL/ampul

cat. # 30609 (ea.)

Rtx®-1 Column (fused silica)



Renzo Brun,
Restek France

Vive la France!

In addition to the traditional seasonal celebrations, 200-plus Restek employee-owners had something extra to commemorate this past December: Restek France has been meet-

ing chromatographers' needs for Restek products and Plus 1™ service for ten years! Félicitations to everyone at Restek France—and best wishes for many more achievements to come.

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.
- Fit column ODs from 0.33–0.74mm (Restek 0.1mm–0.53mm ID).

Me
Pe

alachlor
g-BHC
bromacil
bromodichloromethane

In r

—

GC/ECD Analysis of Chlorophenoxyacid Herbicides

Using Columns with Complementing Selectivity and New Reference Mixes

by John Lidgett, Analytical Reference Materials Technical Specialist

- Optimized analysis on two stationary phases.
- Complete set of reference mixes for US EPA Method 515.4.
- Acids / methyl esters calibration mixes are at concentrations designed for GC/ECD.

Chlorinated phenoxyacid acid herbicides used to control broadleaf weeds are very persistent contaminants in the environment, particularly in drinking water. These strongly polar compounds readily contribute to hydrogen bonds, making them poorly volatile and strongly adsorptive to chromatographic stationary phases. As a consequence, chlorophenoxyacid herbicides are difficult to analyze by GC. To make these compounds suitable for GC analysis they must be derivatized to methyl esters. The most common derivatization reagent is diazomethane. US Environmental Protection Agency Method 515.4

describes a derivatization procedure using diazomethane and an analysis of the methylated esters using GC with an electron capture detector (ECD). The target list of Method 515.4 phenoxyacid herbicides consists of carboxylic acids and phenols.

When monitoring these methylated esters by GC/ECD two columns are needed, to provide identification and confirmation. Further, it is important to select stationary phases that have low bleed and high thermal stability, because the columns should be heated between analyses

to drive off any retained materials. The primary column chosen for this analysis is a 30m, 0.32mm ID, 0.25 μ m Rtx®-CLPesticides2 column. The Rtx®-CLPesticides2 stationary phase is highly selective for electronegative compounds and so is very effective in analyses of chlorophenoxyacid herbicides (Figure 1). We selected our new, intermediate-polarity Rtx®-440 column as the confirmation column because it has unique selectivity for chlorinated pesticides and is thermally stable to 340°C. Figure 2 shows an analysis of methylated chlorophenoxyacid herbicides on a 30m, 0.32mm ID, 0.25 μ m Rtx®-440 column. Resolution is good, and the column exhibits very low bleed at 340°C. In combination, the two columns resolve all target compounds, and the reverse in elution order helps ensure correct identifications. Both columns provide fast analyses.

To design a chlorophenoxyacid herbicide reference material suitable for GC/ECD, detection limits should be determined for each compound in the mix. Because the ECD is highly sensitive, and exhibits a narrow range of linear detection, concentrations of the target compounds must be determined carefully. Additionally, chlorinated phenoxyacid herbicides are photosensitive and heat-labile, so the materials must be packaged in amber ampuls and kept away from heat. Restek now offers a complete set of reference materials for Method 515.4: a chlorinated acids calibration mix, a methylated chlorinated acids calibration mix, a surrogate standard (2,4-dichlorophenylacetic acid), and an internal standard (4,4'-dibromooctafluorobiphenyl). Note that the acids mix will degrade readily in the presence of alkaline compounds or strong oxidizers, and working solutions must be prepared in acidified glassware. The surrogate standard and internal standard are per recommendations in the EPA method. We selected the solvents for the surrogate standard and internal standard carefully, to ensure compatibility with the calibration mixes, and we prepare both standards at high concentrations, for more economical analysis.

Figure 1 Chlorophenoxyacid methyl esters are well separated on an Rtx®-CLPesticides2 column.

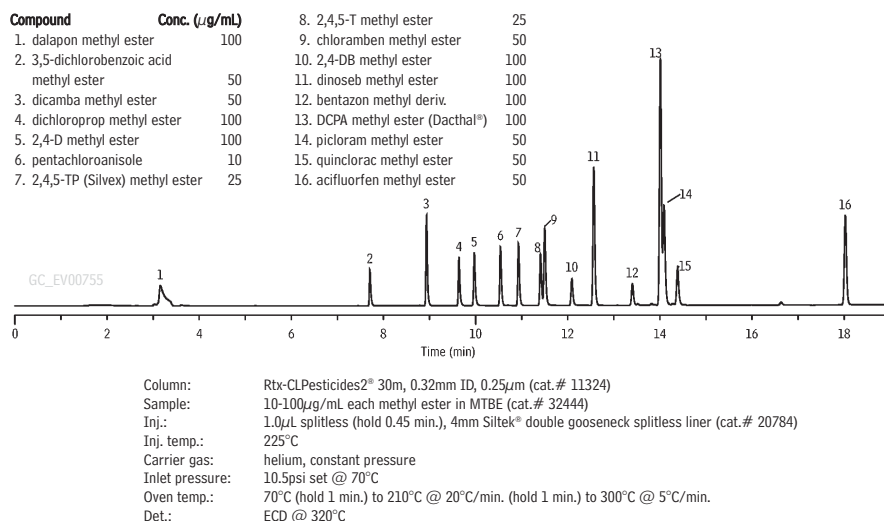
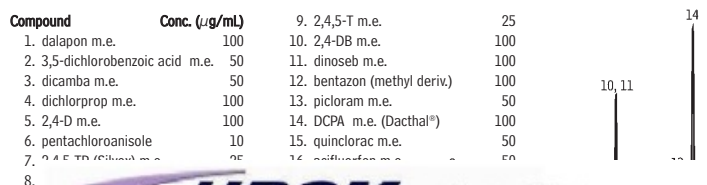


Figure 2 Good resolution of chlorophenoxyacid methyl esters on an Rtx®-440 column.

In combination, an Rtx®-CLPesticides2 column and an Rtx®-440 column resolve all target compounds and provide fast results.



If you are analyzing chlorophenoxyacid herbicides, and want fast analyses and reliable results, we highly recommend the combination of an Rtx®-CLPesticides2 column and an Rtx®-440 column, together with our complete set of reference materials.

11
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Oven temp.: 70°C (hold 1 min.) to 210°C @ 20°C/min. (hold 1 min.) to 300°C @ 5°C/min.
Det.: ECD @ 320°C

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Rtx®-440 Column (fused silica)

(proprietary intermediate-polarity Crossbond® phase)

Temp. limits: -60 to 310/330°C

ID	df (μm)	length	cat. #
0.32mm	0.25	30-Meter	12924

Rtx®-CLPesticides2 Column (fused silica)

Temp. limits: -60 to 320/340°C

ID	df (μm)	length	cat. #
0.32mm	0.25	30-Meter	11324



515.4 Calibration Mix

acifluorfen (Blazer®)	50μg/mL	3,5-dichlorobenzoic acid	50
bentazon	100	dichlorprop	100
chloramben	50	dinoseb	100
2,4-D	100	pentachlorophenol	10
dalapon	100	picloram	50
2,4-DB	100	quinclorac	50
D CPA diacid (tetrachloro-terephthalic acid)	50	2,4,5-T	25
dicamba	50	2,4,5-TP (Silvex)	25

In acetone, 1mL/ampul

cat. # 32443 (ea.)

515.4 Methylated Chlorinated Acids Mix

acifluorfen methyl ester	50μg/mL	3,5-dichlorobenzoic acid	50
bentazon methyl ester	100	dichlorprop methyl ester	100
chloramben methyl ester	50	dinoseb methyl ester	100
dalapon methyl ester	100	pentachloroanisole	10
2,4-D methyl ester	100	picloram methyl ester	50
2,4-DB methyl ester	100	quinclorac methyl ester	50
D CPA methyl ester (Dacthal®)	100	2,4,5-T methyl ester	25
dicamba methyl ester	50	2,4,5-TP (Silvex) methyl ester	25

In methyl *tert*-butyl ether, 1mL/ampul
cat. # 32444 (ea.)

515.4 Internal Standard

4,4-dibromooctafuorobiphenyl

2,000μg/mL in methyl *tert*-butyl ether, 1mL/ampul
2,000 cat. # 31856 (ea.)

515.4 Surrogate Mix

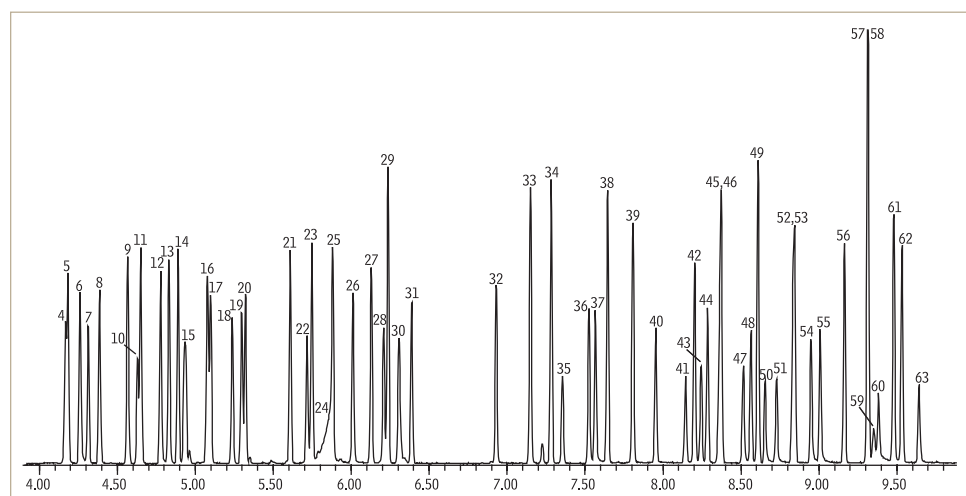
2,4-dichlorophenylacetic acid

1,000μg/mL in acetone, 1mL/ampul
1,000 cat. # 32439 (ea.)

Fast GC/MS Analysis of Semivolatile Organic Compounds

Using a 0.18mm ID / 0.36μm Film Rtx®-5Sil MS Column

(cont. from page 14)



Column: Rtx®-5Sil MS, 20m, 0.18mm ID, 0.36μm (cat.# 557810)
Sample: US EPA Method 8270D analytes, 10ppm each (10ng on column): 8270 MegaMix™ (cat.# 31850), Benzidine (cat.# 31441), Benzoic Acid (cat.# 31415), 2,4-Dinitrophenol (cat.# 31291), Acid Surrogate Mix (4/89 SOW) (cat.# 31063), B/N Surrogate Mix (4/89 SOW) (cat.# 31062)
Inj.: 1.0μL, splitless, 4mm ID gooseneck splitless inlet liner (cat.# 20798), splitless hold time 0.20 min., pressure pulse 0.15 min. @ 30psi
GC: Agilent 6890
Inj. temp.: 250°C
Carrier gas: helium, constant flow
Flow rate: 1.2mL/min.
Oven temp.: 50°C(hold 0.5 min.) to 330°C @ 18°C/min. (hold 3 min.)
Det.: Agilent 5973 GC/MS
Transfer line temp.: 280°C
Scan range: 35-550 amu
Solvent Delay: 1 min.
Tune: DFTPP
Ionization: EI

4. phenol-d6	21. isophorone	39. 2-chloronaphthalene	57. fluorene
5. phenol	22. 2-nitrophenol	40. 2-nitroaniline	58. 4-chlorophenyl phenyl ether
6. aniline	23. 2,4-dimethylphenol	41. 1,4-dinitrobenzene	59. 4-nitroaniline
7. bis(2-chloroethyl)ether	24. benzoic acid	42. dimethylphthalate	60. 4,6-dinitro-2-methylphenol
8. 2-chlorophenol	25. bis(2-chloroethoxy)methane	43. 1,3-dinitrobenzene	61. diphenylamine
9. 1,3-dichlorobenzene	26. 2,4-dichlorophenol	44. 2,6-dinitrotoluene	62. azobenzene



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Instrument Innovations!




Simplify Life in Your Laboratory

by Donna Lidgett, GC Accessories Product Marketing Manager

Whether you have an Agilent, PerkinElmer, Shimadzu, Thermo Finnigan, or Varian system, Restek consumables and parts will help you maintain optimum system performance, and give you the convenience and economy of one-stop shopping for all your GC needs.


Liners for ATAS injectors

new!







Liners for ATAS Injectors	Benefits/Uses:	ID/OD & Length (mm)	ea.	cat.# 5-pk.
	universal	3.0 5.0 x 80	22415	22416
ATAS Open Liner, 3mm				
	trace, active samples <1μL	1.0 5.0 x 80	22417	22418
ATAS Open Liner, 1mm				
	dirty samples	3.0 5.0 x 80	22419	22420
ATAS Fritted Gooseneck				

Liner for Varian 1177 GCs

new!

Liners for Varian 1177 GCs	Benefits/Uses:	ea.	cat.# 5-pk.
	trace samples <2μL, dirty samples	22421	22422
Low Pressure Drop Precision™ Liner (2.0mm ID, 6.3mm OD, 78.5mm length)			

Direct Injection Liners

DI Liners for Agilent 5890 & 6890 GCs (For 0.25/0.32/0.53mm ID Columns)	ID/OD & Length (mm)	ea.	cat.# 5-pk.
	4.0 ID 6.3 OD x 78.5	21054	21055
Drilled Uniliner® (hole on top)			
	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5
Siltek® Drilled Uniliner® (hole on top)			
	4.0 ID 6.3 OD x 78.5	20756*	20771
Drilled Uniliner® (hole on bottom)			
	4.0 ID 6.3 OD x 78.5	20508	20509
Double Gooseneck Drilled Uniliner® (hole on top)			
	4.0 ID 6.3 OD x 78.5	20954**	20989
Double Gooseneck Drilled Uniliner® (hole on bottom)			
	1.0 ID 6.3 OD x 78.5	21390-214.1	21391-214.5
Siltek® 1mm Drilled Uniliner® (hole on top)			

DI Liners for Shimadzu 17A & 2010 GCs (For 0.32/0.53mm ID Columns)	ID/OD & Length (mm)	ea.	cat.# 5-pk.
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All liners are

100%
deactivated

All liners are shipped intermediate polarity (IP) deactivated unless otherwise requested.

Drilled Uniliner® Inlet Liners

Hole makes direct injection possible with EPC-equipped Agilent 6890 GCs.

Allows injector to be operated in split/splitless mode.

Ideal for trace, active samples; high recovery and linearity.

Drilled Uniliner® inlet liners are available in two configurations.

Use hole on bottom configuration if analytes elute near the solvent peak.









Use hole on top configuration if analytes elute away from the solvent peak, or when the sample solvent is water.



free literature

Minimize Adsorption of Active Analytes, Using a Drilled Uniliner® GC Inlet Liner (lit. cat.# 59877)

DI Liners for PerkinElmer GCs (For 0.32/0.53mm ID Columns)	ID/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
	4.0 ID 6.2 OD x 92.1	20819	20822
Auto SYS Drilled Uniliner® (hole on top)			
	4.0 ID 6.2 OD x 92.1	21293	21294
Auto SYS Drilled Uniliner® (hole on bottom)			
	4.0 ID 5.0 OD x 92.1	21295	21296
Auto SYS Gooseneck Drilled Uniliner® (hole on top)			
	4.0 ID 6.2 OD x 92.1	21297	21298
Auto SYS Gooseneck Drilled Uniliner® (hole on bottom)			

DI Liners for Varian 1177 GCs (For 0.25/0.32/0.53mm ID Columns)	ID/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
	4.0 ID 6.3 OD x 78.5	21470	21471
Drilled Uniliner® (hole on top)			
	4.0 ID 6.3 OD x 78.5	21468	21469
Drilled Uniliner® (hole on bottom)			



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O-Rings and Liner Seals

For complete listings, refer to our catalog or website.

Viton® O-Rings for Agilent GCs

- Fit split (6.3mm OD) or splitless (6.5mm OD) liners.
- Max. temp.: 250°C
- Similar to Agilent part# 5180-4182



Description	qty.	cat.#
Viton® O-Rings for Agilent GCs	25-pk.	20377

Graphite O-Rings for Agilent and Varian 1177 GCs

- Max temp.: 450°C
- Cat.# 20296 similar to Agilent part# 5180-4168, cat.# 20298 similar to 5180-4173.



Description	Restek cat.#	
	10-pk.	50-pk.
Graphite O-rings for split liners (6.3mm ID)	20296	20297
Graphite O-rings for split-less liners (6.5mm ID)	20298	20299

Graphite Liner Seals for Varian 1078/1079 GCs

- Max temp.: 450°C.
- Similar to Varian part# 392611919 and 392534201.



Description	qty.	cat.#
Graphite Liner Seals for Varian 1078/1079 GCs (5mm)	10-pk.	22683

Viton® O-Rings for PerkinElmer Auto SYS™ GCs

- Max temp.: 250°C.
- Similar to PE part# N6101374.



Description	qty.	cat.#
Viton® O-Rings for PerkinElmer Auto SYS GCs	10-pk.	20262

Graphite O-Rings for PerkinElmer Auto SYS™ XL PSS

- Max temp.: 450°C.
- Similar to PE part# N6101751.



Description	qty.	cat.#
Graphite O-Rings for PerkinElmer Auto SYS XL PSS	10-pk.	21475
Graphite O-Rings for PerkinElmer Auto SYS XL PSS	25-pk.	21476

Viton® O-Rings for PerkinElmer PSS

- Max temp.: 250°C.
- Similar to PE part# N610-1747.



Description	qty.	cat.#
Viton® O-Rings for PerkinElmer PSS	10-pk.	20366

Graphite O-Rings for Shimadzu 17A and 2010 GCs

- Max. temp.: 450°C.



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17A and 2010 GCs	10-pk.	21477
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Injector and Detector Parts

new!

FID Collector Housing Kit for Agilent 5890 GCs

- Meets or exceeds OEM performance.
- Kit includes collector body, spanner nut, and silicone washer.



Description	Similar to Agilent part #	qty.	cat.#
FID Collector Housing Kit for Agilent 5890 GCs	19231-20920	kit	23037

FID Collector Mount for Agilent 5890 GCs

- Meets or exceeds OEM performance.



Description	Similar to Agilent part #	qty.	cat.#
FID Collector Mount for Agilent 5890 GCs	19231-20930	ea.	23036

FID Base Weldment for Agilent 5890 GCs

- Meets or exceeds OEM performance.
- Kit includes brass nut.



Description	Similar to Agilent part #	qty.	cat.#
FID Base Weldment for Agilent 5890 GCs	19231-80580	ea.	23041

FID Capillary Column Adaptor for PerkinElmer Auto SYS™ XL

- Made of high quality stainless steel.
- Meets or exceeds OEM performance.



Description	Similar to PE part #	qty.	cat.#
For use with PE style capillary nuts			
FID Capillary Column Adaptor for PerkinElmer Autosys XL	N6120020	ea.	22608
For use with 1/8" compression style nuts			
FID Capillary Column Adaptor for PerkinElmer Autosys XL	—	ea.	22609

Septum Cap for PerkinElmer Auto SYS™ XL

- Made of clear anodized aluminum and high-quality stainless steel.
- Meets or exceeds OEM performance.



Description	Similar to PE part #	qty.	cat.#
Septum Cap for PerkinElmer Autosys XL	N6100153	ea.	22322

Injector Adaptor for PerkinElmer Auto SYS™ XL

- Made of high quality stainless steel.
- Meets or exceeds OEM performance.
- Siltek®-treated version available for increased inertness.



Description	Similar to PE part #	qty.	cat.#
For use with PE style capillary nuts			
Injector Adaptor for PerkinElmer Auto SYS	N6100157	ea.	22318
Siltek®-Treated Injector Adaptor for PerkinElmer Auto SYS XL	—	ea.	22320
For use with 1/8" compression style nuts			
Injector Adaptor for PerkinElmer Auto SYS	—	ea.	22319
Siltek®-Treated Injector Adaptor for			



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Reliable Connections Made Simple

by Donna Lidgett, GC Accessories Product Marketing Manager

restek
innovation!

- Reliable seal integrity—will not unexpectedly disconnect during temperature-programmed analyses.
- Open design allows visual confirmation of the seals, for added confidence in the connections.
- Use standard Press-Tight® connectors.

SeCure™ “Y” Connectors

Connect two analytical columns to a transfer line or guard column.

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, 1 “Y” Universal Press-Tight® union, and 3 ferrules.

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

Graphite Ferrules for SeCure™ “Y” Connectors

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

Universal “Y” Press-Tight® Connectors

Description	ea.	3-pk.
Universal “Y” Press-Tight® Connector	20405	20406
Siltek®-treated Universal “Y” Press-Tight® Connector	20485	20486

Vu2 Union™ Connectors

Connect a guard column to an analytical column, a column to a transfer line, two columns in series, or 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	0.15–0.25mm	kit	21105
Vu2 Union™ Connector Kit	0.28/0.32mm	kit	21106
Vu2 Union™ Connector Kit	0.45/0.50 & 0.53mm	kit	21107
Knurled nut		2-pk.	21108

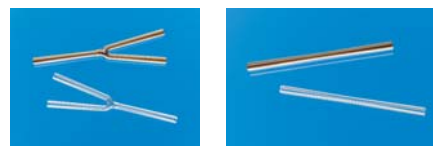
NOTE: Not recommended for GC column-to-MS connections—use the Vacuum Vu-Union® described in our catalog.

Graphite Ferrules for Vu2 Union™ Connectors

Ferrule ID	Fits Column ID	Graphite 2-pk.	Graphite 10-pk.
0.4mm	0.18–0.25mm	20280	20281
0.5mm	0.28/0.32mm	20282	20283
0.8mm	0.45/0.50 & 0.53mm	20284	20285



Make secure, reliable column-to-column connections with SeCure™ “Y” connectors. Secondary seals ensure a leak-tight connection.



Both SeCure™ “Y” and Vu2 Union™ Connectors use standard Press-Tight® connectors—no expensive, unique inserts to purchase.



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EZ No-Vent™ GC Column-MS Connector

Change Columns in Minutes Without Venting

by Donna Lidgett, GC Accessories Product Marketing Manager

new!

Now available for Varian 2000 Series MSs

- Save hours of downtime—100µm transfer line throttles vacuum and prevents MS pump-down.
- Easy to install and maintain—no special tools or plumbing required.
- Gold-plated body for inertness.
- Deactivated transfer line keeps analytes focused; high-temperature polyimide ferrules eliminate leaks at the problematic transfer line fitting.
- Lower cost than other “no-vent” fittings.
- Available for Agilent GCs with 5971/5972 or 5973 GC/MS, Varian Saturn 2000 Series MSs.

We designed the EZ No-Vent™ connector to be simple and easy to use. A critical orifice in the EZ No-Vent™ connector minimizes the amount of oxygen allowed into the MS source, eliminating the need for purge gas and enabling you to skip the lengthy vent and pump-down cycle otherwise required when you make a column change. This can save nearly a day of downtime with each column change. The EZ No-Vent™ connector easily attaches to the MS source without special tools or extra plumbing.

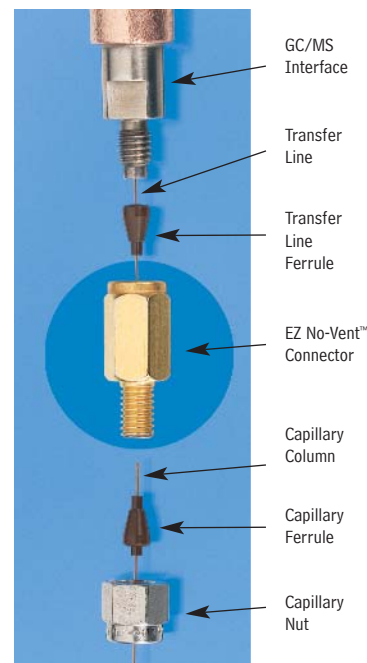
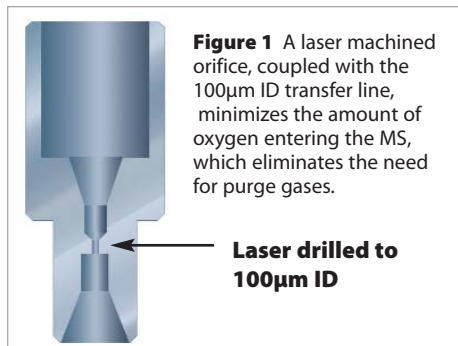
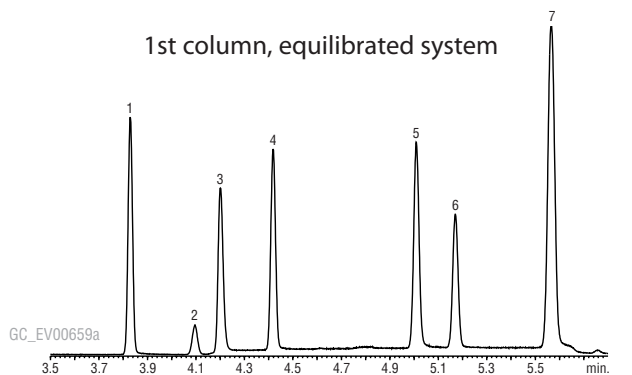
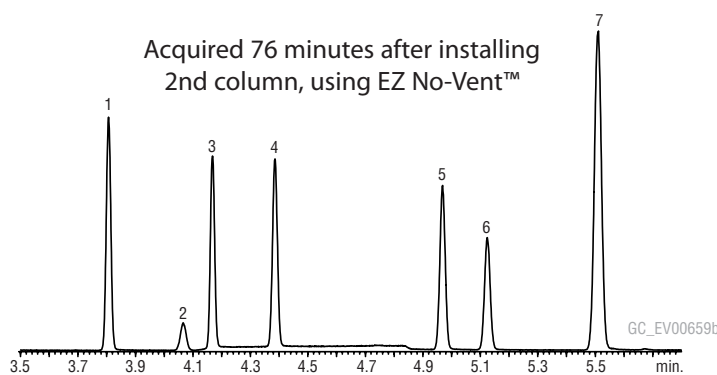


Figure 2 Sharp, symmetric peaks for gases show the EZ No-Vent™ connector does not add dead volume and allows rapid column changes.



502.2 Calibration Mix#1 (gases) cat# 30042

- | | |
|---|---------------------------|
| 1. dichlorodifluoromethane | 5. bromomethane |
| 2. 1,2-dichlorotetrafluoroethane (Freon® 114) | 6. chloroethane |
| 3. chloromethane | 7. trichlorofluoromethane |
| 4. vinyl chloride | |



Column: Rtx®-624 60m, 0.25mm ID, 1.4µm (cat# 10969)
Inj.: purge & trap
GC: Agilent 6890
Inj. temp.: 300°C
Carrier gas: helium, constant flow
Flow rate: 1.0mL/min.

Oven temp.: 60°C
Det: Agilent 5973 GC/MS
Transfer line temp.: 280°C
Scan range: 35-550 amu
Tune: BFB
Ionization: EI

Description

EZ No-Vent™ Connector Kit for Agilent 5971/5972 and 5973 GC/MS

qty.

cat.#

EZ No-Vent™ Connector Kit for Varian Saturn 2000 Series MSs

new!

kit

21323

Replacement ferrules for connecting capillary column to EZ No-Vent™:

0.4mm ID

2-pk.

21015

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Each kit includes: EZ No-Vent™

Connector, two 0.4mm ID ferrules for capillary column, two 0.4mm ID ferrules for transfer line, 100µm deactivated

It's Here! The 2005 Restek Catalog!

- 775+ pages / thousands of innovative products.
- Many new chromatograms.
- Helpful technical information.

Some of the new items in the 2005 Restek Catalog:

GC Columns

Rtx®-440 - Low-bleed, high-resolution, intermediate-polarity column for many applications.

Rtx®-5SII MS, 0.18mm ID - Monitor nanogram levels of semivolatile pollutants in 15 minutes.

Rtx®-Dioxin2 - Improved separation of dioxin or furan congeners, compared to diphenyl or high-cyano columns.

Rtx®-1PONA Column/Rtx®-5PONA Tuning Column - Performance enhanced, for 30% faster SIMDIS analysis, using helium.

Rtx®-XLB Column - Ideal for active, higher weight environmental analytes and other compounds.

Too new for our catalog! Read about the Rtx®-PCB column on page 13 of this *Advantage*.

HPLC Columns

Allure™ Aqueous C18 - Excellent retention and selectivity for polar analytes in highly aqueous mobile phases.

Ultra Quat - Monitor paraquat and diquat without ion pairing reagents.

HPLC Method Development Column Kits - Multiple stationary phases help to quickly optimize selectivity.

GC Accessories

Parker Balston FID-1000 Gas Station - Ultra-high purity hydrogen and zero grade air from a single unit.

EZ No-Vent™ Column/MS Connector for Varian Saturn 2000 Series MSs - Change column in minutes without venting.

Alumaseal™ aluminum ferrules - The sealing ease of graphite, the security and reliability of metal ferrules.

Vespel® ferrules - We give you more choices: Vespel®, Vespel®/graphite, or graphite.

HPLC Accessories

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