RESTEK ADVANTAGE Newsletter

2001-2005 ~ Quarterly

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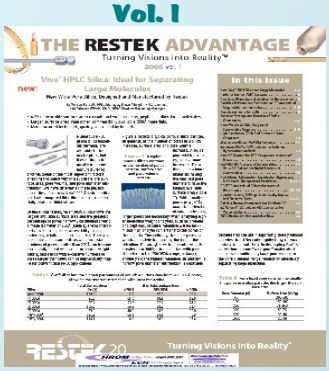
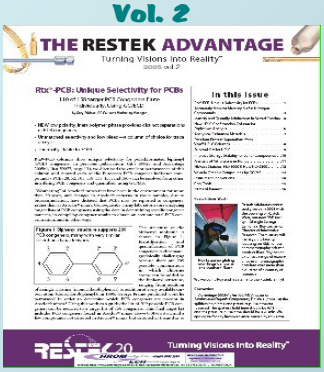
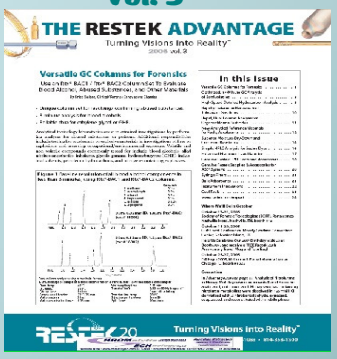
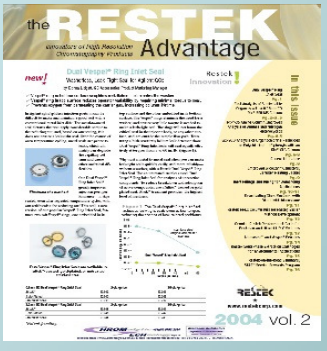
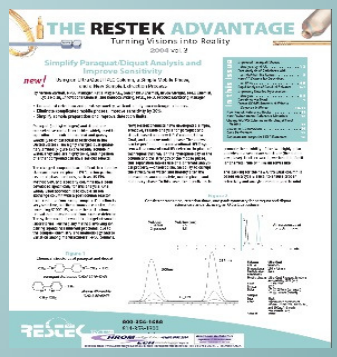
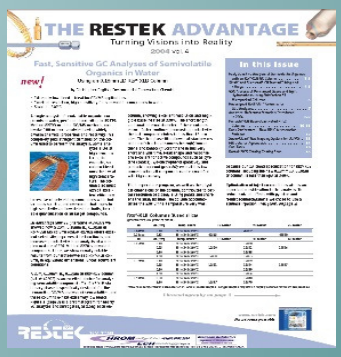
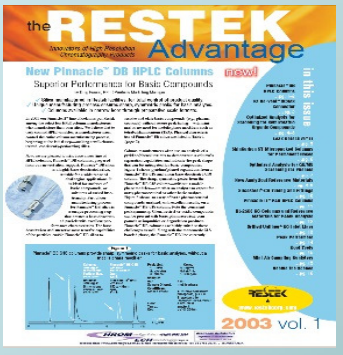
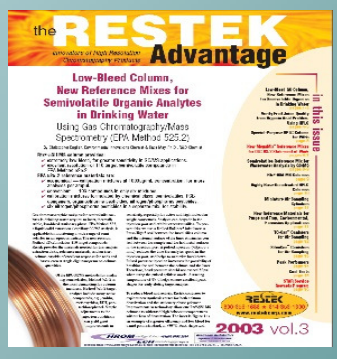
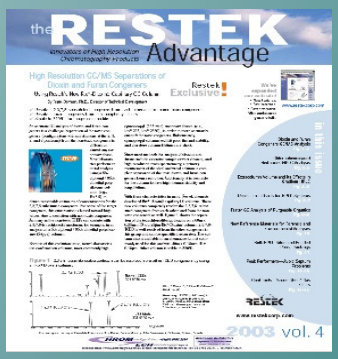
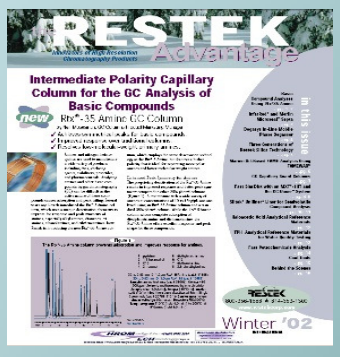
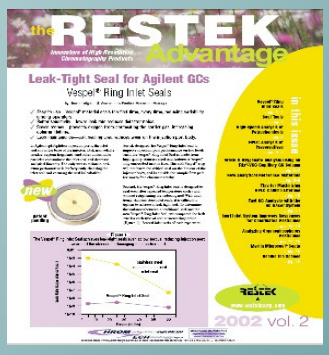
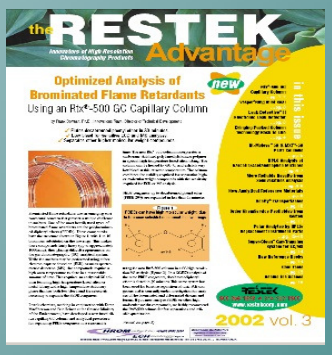
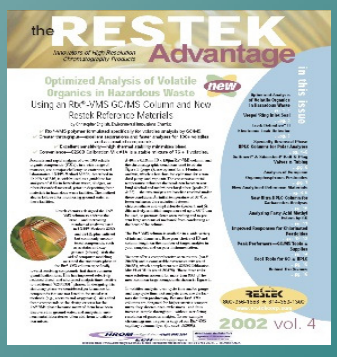
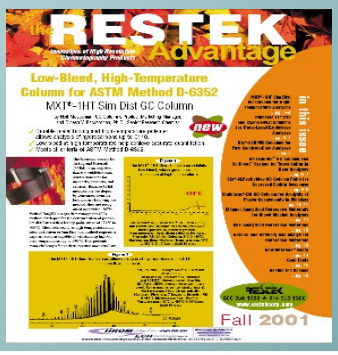
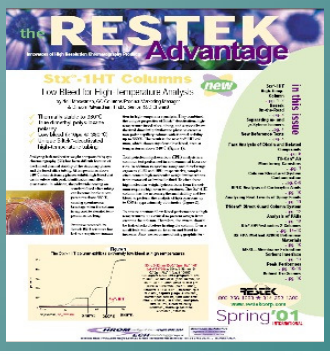
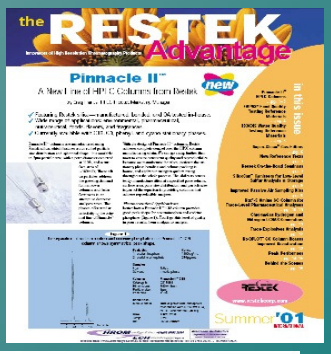
2005				
2004				
2003				
2002				
2001				

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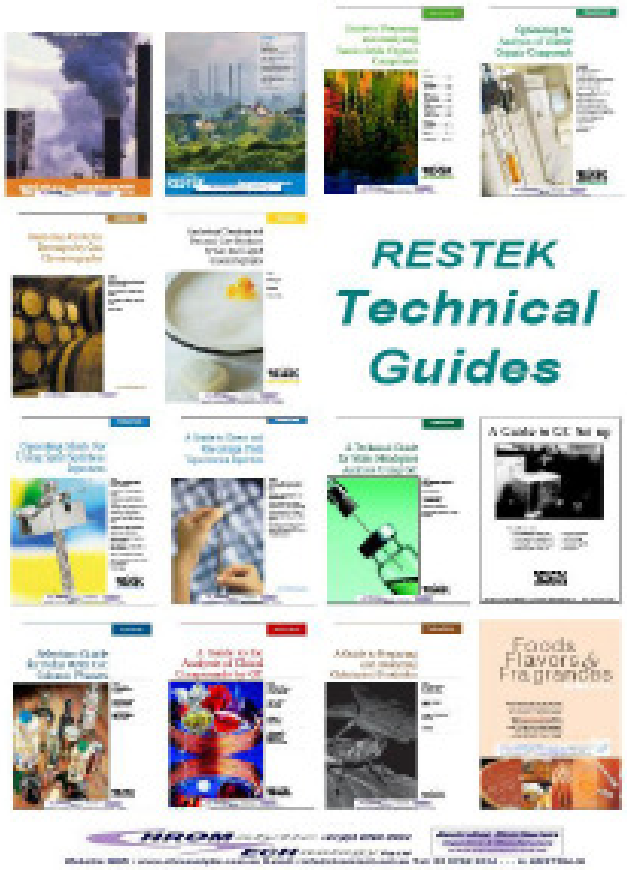


the RESTEK Advantage

Innovators of High Resolution Chromatography Products

new!

Dual Vespel® Ring Inlet Seal

Washerless, Leak-Tight Seal for Agilent GCs

by Donna Lidgett, GC Accessories Product Marketing Manager

Restek Innovation!

- ✔ Vespel® ring in bottom surface simplifies installation—eliminates the washer.
- ✔ Vespel® ring in top surface reduces operator variability by requiring minimal torque to seal.
- ✔ Prevents oxygen from permeating the carrier gas, increasing column lifetime.

In Agilent split/splitless injection ports, it can be difficult to make and maintain a good seal with a conventional metal inlet disk. The metal-to-metal seal dictates that you apply considerable torque to the reducing nut, and, based on our testing, this does not ensure a leak-tight seal. Over the course of oven temperature cycling, metal seals are prone to leaks, which ultimately can degrade the capillary column and cause other analytical difficulties.

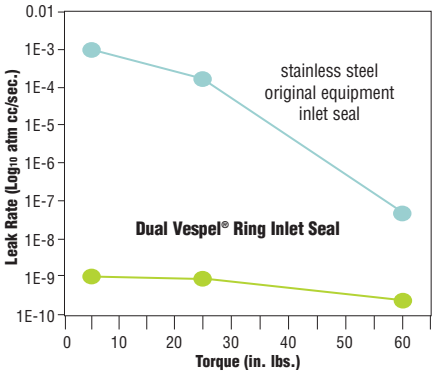
Eliminate the washer!

Our Dual Vespel® Ring Inlet Seal* greatly improves injection port performance—it stays sealed, even after repeated temperature cycles, without retightening the reducing nut! This seal, a new version of our popular Vespel® Ring Inlet Seal, features two soft Vespel® rings, one embedded in its top surface and the other embedded in its bottom surface. The Vespel® rings eliminate the need for a washer, and ensure very little torque is needed to make a leak-tight seal. The rings will not harm the critical seal in the injector body, or any other surface, and are outside the sample flow path. Tests using a high sensitivity helium leak detector show Dual Vespel® Ring Inlet Seals will seal equally effectively at torques from 5 to 60 in. lb. (Figure 1).

Why trust a metal-to-metal seal when you can make leak-tight seals quickly, easily, and more reliably—without a washer, with a Restek Dual Vespel® Ring Inlet Seal. Use an untreated stainless steel Dual Vespel® Ring Inlet Seal for analyses of unreactive compounds. To reduce breakdown and adsorption of active compounds, use a Siltek™-treated or gold-plated seal. Siltek™ treatment provides the highest level of inertness.

Dual Vespel® Ring Inlet Seals are available in Siltek™-treated, gold-plated, or untreated stainless steel.

Figure 1 The Dual Vespel® Ring Inlet Seal achieves leak-tight seals even at low torque, reducing the chance of leak-related problems.



0.8mm ID Dual Vespel® Ring Inlet Seal	2-pk./price	10-pk./price
Siltek™	21242	21243
Gold-Plated	21240	21241
Stainless Steel	21238	21239
1.2mm ID Dual Vespel® Ring Inlet Seal	**2-pk./price**	**10-pk./price**
Siltek™	21248	21249
Gold-Plated	21246	21247
Stainless Steel	21244	21245

Patent pending.

in this issue

RESTEK

www.restekcorp.com

2004 vol. 2

Fast Analysis of Semivolatile Organic Analytes

Using a 0.18mm ID Rtx®-5Sil MS Capillary GC Column

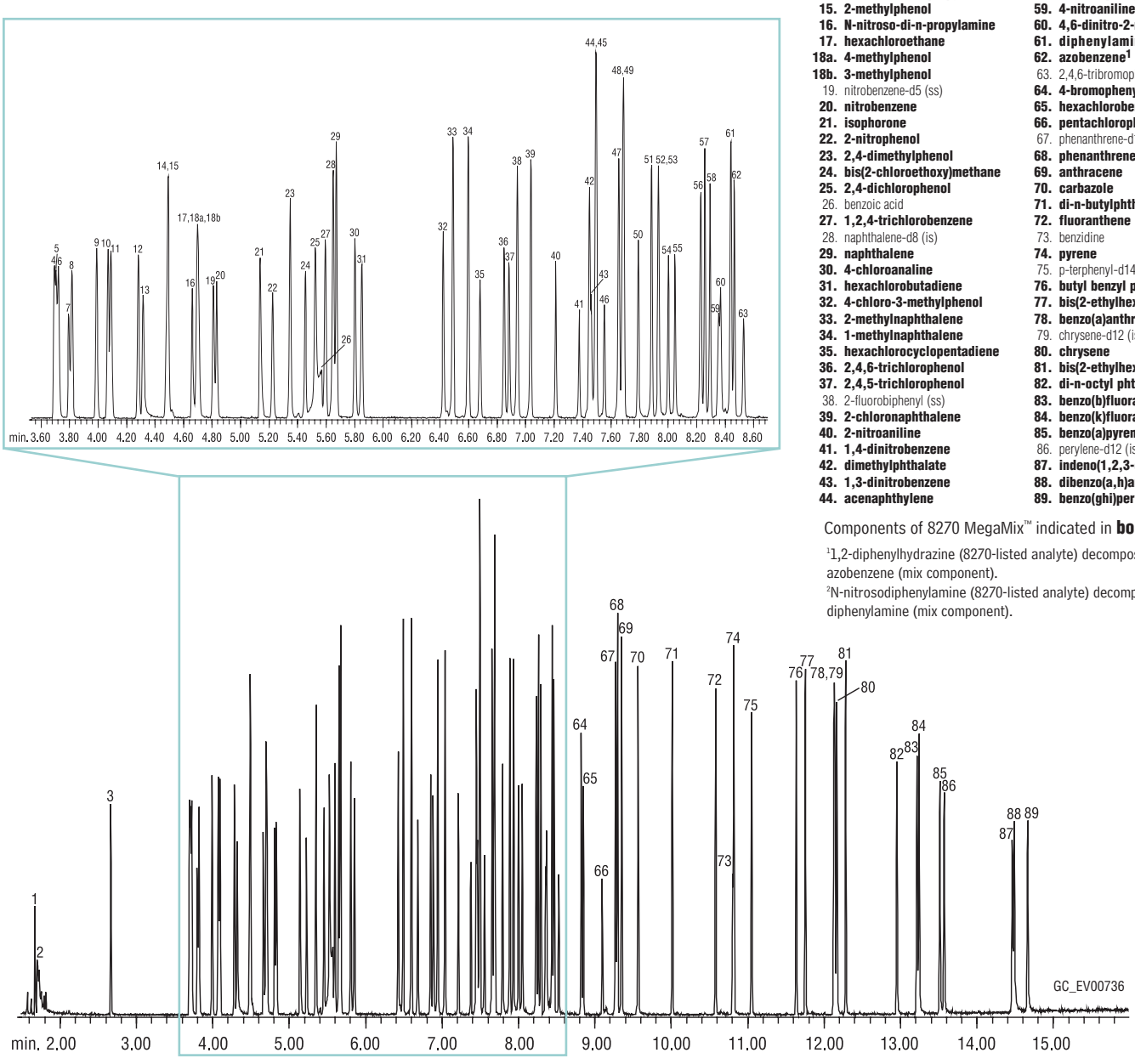
By Katia May, Ph.D., Senior R&D Chemist, and Christopher English, Environmental Innovations Chemist

- ✔ Improve efficiency by reducing analysis time for 90 compounds to less than 15 minutes.
- ✔ Low-bleed, high-resolution column is ideal for trace analyses.
- ✔ 8270 MegaMix™ reference mix includes 76 target compounds, has 18-month shelf life.

US EPA Method 8270D is one of the GC/MS methods followed to determine concentrations of semivolatile organic compounds in solid waste, soil, water, and air matrices. In a single analysis, environmental laboratories following Method 8270D typically evaluate 100 or more semivolatile organic compounds representing several classes of compounds of differing chemical properties and reactivity. This complexity puts stringent demands on the column used to perform the analysis. Some polycyclic aromatic hydrocarbons (PAHs) elute at high temperatures, for example, so the method requires a column with low bleed at high temperature. The column also must exhibit excellent efficiency, to ensure resolution of closely eluting compounds with similar mass spectra, including structural isomers. Additionally, calibration mixes of semivolatile compounds must be combined carefully, to prevent interactions that could compromise stability.

Rtx®-5Sil MS column performance allows improved detection limits and increased productivity, and the column performs exceptionally well in analyses of semivolatile compounds. Figure 1 shows an analysis of our 8270 MegaMix™ reference mix of 76 target

Figure 1 78 semivolatile pollutants, plus surrogates and internal standards, separated in less than 15 min. on a 0.18mm ID Rtx®-5Sil MS column.



1. **N-nitrosodimethylamine**
2. **pyridine**
3. 2-fluorophenol (ss)
4. **aniline**
5. phenol-d6 (ss)
6. **phenol**
7. **bis(2-chloroethyl)ether**
8. **2-chlorophenol**
9. **1,3-dichlorobenzene**
10. 1,4-dichlorobenzene-d4 (is)
11. **1,4-dichlorobenzene**
12. **1,2-dichlorobenzene**
13. **benzyl alcohol**
14. **bis(2-chloroisopropyl)ether**
15. **2-methylphenol**
16. **N-nitroso-di-n-propylamine**
17. **hexachloroethane**

18a. **4-methylphenol**

18b. **3-methylphenol**

19. nitrobenzene-d5 (ss)
20. **nitrobenzene**
21. **isophorone**
22. **2-nitrophenol**
23. **2,4-dimethylphenol**
24. **bis(2-chloroethoxy)methane**
25. **2,4-dichlorophenol**
26. benzoic acid
27. **1,2,4-trichlorobenzene**
28. naphthalene-d8 (is)
29. **naphthalene**
30. **4-chloroanaline**
31. **hexachlorobutadiene**
32. **4-chloro-3-methylphenol**
33. **2-methylnaphthalene**
34. **1-methylnaphthalene**
35. **hexachlorocyclopentadiene**
36. **2,4,6-trichlorophenol**
37. **2,4,5-trichlorophenol**
38. 2-fluorobiphenyl (ss)
39. **2-chloronaphthalene**
40. **2-nitroaniline**
41. **1,4-dinitrobenzene**
42. **dimethylphthalate**
43. **1,3-dinitrobenzene**
44. **acenaphthylene**
45. **2,6-dinitrotoluene**
46. **1,2-dinitrobenzene**
47. acenaphthene-d10 (is)
48. **3-nitroaniline**
49. **acenaphthene**
50. **2,4-dinitrophenol**
51. **dibenzofuran**
52. **4-nitrophenol**
53. **2,4-dinitrotoluene**
54. **2,3,4,6-tetrachlorophenol**
55. **2,3,5,6-tetrachlorophenol**
56. **diethyl phthalate**
57. **fluorene**
58. **4-chlorophenyl phenyl ether**
59. **4-nitroaniline**
60. **4,6-dinitro-2-methylphenol**
61. **diphenylamine[2]**
62. **azobenzene[1]**
63. 2,4,6-tribromophenol (ss)
64. **4-bromophenyl phenyl ether**
65. **hexachlorobenzene**
66. **pentachlorophenol**
67. phenanthrene-d10 (is)
68. **phenanthrene**
69. **anthracene**
70. **carbazole**
71. **di-n-butylphthalate**
72. **fluoranthene**
73. benzidine
74. **pyrene**
75. p-terphenyl-d14 (ss)
76. **butyl benzyl phthalate**
77. **bis(2-ethylhexyl)adipate**
78. **benzo(a)anthracene**
79. chrysene-d12 (is)
80. **chrysene**
81. **bis(2-ethylhexyl)phthalate**
82. **di-n-octyl phthalate**
83. **benzo(b)fluoranthene**
84. **benzo(k)fluoranthene**
85. **benzo(a)pyrene**
86. perylene-d12 (is)
87. **indeno(1,2,3-cd)pyrene**
88. **dibenzo(a,h)anthracene**
89. **benzo(ghi)perylene**

Components of 8270 MegaMix™ indicated in **bold.**

[1]1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).

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

compounds, plus benzoic acid, benzidine, and surrogate and internal standards, on our new 20m, 0.18mm ID, 0.18µm Rtx®-5Sil MS column (cat.# 42702). The Rtx®-5Sil MS stationary phase is based on a silarylene polymer specifically designed for the demanding GC/MS analysis of semivolatile compounds, and the column exhibits lower bleed than columns prepared from phenyl/methyl polymers. All target compounds can be quantified with greater sensitivity. The thin phase film in this column allows superior resolution of structural isomers benzo(b)fluoranthene and benzo(k)fluoranthene (peaks 83 and 84), while achieving a very short analysis time of less than15 minutes. Peak shape and response are excellent, even for active compounds such as 2,4-dinitrophenol and pentachlorophenol (peaks 50 and 66). Optimizing the temperature program, as well as the physical dimensions of the column, contributes to better resolution of closely eluting peaks and shortens the analysis time.

In order to achieve the separation shown in Figure 1, care must be taken to optimize injection conditions. To reduce solvent effects that could interfere with N-nitrosodimethylamine and pyridine (peaks 1 and 2), we chose a splitless inlet liner, rather than a direct injection liner (e.g., a Uniliner®). A cyclo double gooseneck design enables the sample to be completely volatilized in the injection port prior to condensing at the column inlet, and ensures more reproducible results, relative to a standard (straight) liner. The 2mm internal diameter provides the best results with 0.5µL injections. The splitless hold time also is very important: a change of only several seconds can reduce sensitivity by 50%. We discovered that a pulsed splitless analysis, using a 0.20 min. pulse 5psi higher than the column flow backpressure, dramatically improves sample transfer onto the column. Making the pulse 3 seconds (0.05 min.) longer than the splitless hold time (0.15 min.) allows excess solvent to be swept away quickly. The 270ºC injection port temperature vaporizes the sample with minimal analyte breakdown. GC conditions were adjusted to resolve analytes that coelute and share ions. Aniline and phenol (peaks 4 and 6), for example, were resolved by using an initial temperature ramp rate of 14ºC/min., and the key to resolving isomers benzo(b)fluoranthene and benzo(k)fluoranthene (peaks 83 and 84) is to be sure that they elute during the temperature ramp portion of the program. If the isomers elute during the final hold time they will not be well resolved. By using a 0.18mm ID Rtx®-5Sil MS column under these conditions, you will ensure a rapid and successful analysis of the 8270 compounds.

To meet the substantial demand for reference materials for Method 8270, we offer 8270 MegaMix™ reference mix (cat.# 31686)—a formulation of 76 target compounds in methylene chloride/benzene (75:25).

continued on page 5

Rtx®-5Sil MS 20m, 0.18mm ID, 0.18µm (cat.# 42702)

Sample:	US EPA Method 8270D mix: 8270 MegaMix™ (cat.# 31686), benzoic acid (cat.# 31415), benzidine (cat.# 31441), 2,4-dinitrophenol (cat.# 31291)*, Acid Surrogate Mix (4/89 SOW) (cat.# 31063), B/N Surrogate Mix (4/89 SOW) (cat.# 31062), SV Internal Standard Mix (cat.# 31206) *2,4-dinitrophenol also is a component of the MegaMix™.
Inj.:	0.5µL, 5ppm each component (2.5ng on column) (2,4-dinitrophenol at 10ppm/5ng on column; 3-methylphenol and 4-methylphenol at 2.5ppm/1.25ng on column), splitless (hold 0.15 min., pressure pulse 0.20 min. @ 30psi), 2mm cyclo double gooseneck inlet liner (cat.# 20907); Agilent 6890
Inj. temp.:	270°C
Carrier gas:	helium, constant flow
Flow rate:	1.2mL/min.
Oven temp.:	40°C (hold 0.5 min.) to 90°C @ 14°C/min., to 330°C @ 22°C/min. (hold 1 min.)
Det.:	Agilent 5973 GC/MS
Transfer line temp.:	280°C
Scan range:	35-550 amu
Solvent Delay:	1 min.
Tune:	DFTPP
Ionization:	EI

Use this new column for sub-15 minute analysis of 78 Method 8270D target compounds.

Rtx®-5Sil MS Columns (fused silica)

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

ID	df (µm)	temp. limits	20-Meter	
0.18mm	0.18	-60 to 330/350°C	42702	
ID	**df (µm)**	**temp. limits**	**15-Meter**	**30-Meter**
0.25mm	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
0.32mm	0.25	-60 to 330/350°C	12721	12724
	0.50	-60 to 330/350°C	12736	12739
	1.00	-60 to 325/350°C	12751	12754

Acid Surrogate Mix (4/89 SOW)

2-fluorophenol
2,4,6-tribromophenol
phenol-d6

Each	5-pk.	10-pk.
2,000µg/mL each in methanol, 1mL/ampul		
31025	31025-510	—
w/ data pack		
31025-500	31025-520	31125
10,000µg/mL each in methanol, 1mL/ampul		
31063	31063-510	—
w/ data pack		
31063-500	31063-520	31163
10,000µg/mL each in methanol, 5mL/ampul		
31087	31087-510	—
w/ data pack		
31087-500	31087-520	31187

B/N Surrogate Mix (4/89 SOW)

2-fluorobiphenyl
p-terphenyl-d14
nitrobenzene-d5

Each	5-pk.	10-pk.
1,000µg/mL each in methylene chloride, 1mL/ampul		
31024	31024-510	—
w/ data pack		
31024-500	31024-520	31124
5,000µg/mL each in methylene chloride, 1mL/ampul		
31062	31062-510	—
w/ data pack		
31062-500	31062-520	31162
5,000µg/mL each in methylene chloride, 5mL/ampul		
31086	31086-510	—
w/ data pack		
31086-500	31086-520	31186

8270 MegaMix™ (76 components)

Components listed in **bold** in Figure 1.

1,000µg/mL each (3-methylphenol and 4-methylphenol at 500µg/mL each) in methylene chloride:benzene (75:25), 1mL/ampul

Each	5-pk.	10-pk.
31686	31686-510	—
w/data pack		
31686-500	31686-520	31786

SV Internal Standard Mix

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

Each	5-pk.	10-pk.
2,000µg/mL each in methylene chloride, 1mL/ampul		
31206	31206-510	—
w/data pack		
31206-500	31206-520	31306
4,000µg/mL each in methylene chloride, 1mL/ampul		
31006	31006-510	—
w/ data pack		
31006-500	31006-520	31106

Benzidine

1,000µg/mL in methanol, 1mL/ampul

Each	5-pk.	10-pk.
31441	31441-510	—
w/data pack		
31441-500	31441-520	31541

Benzoic Acid

1,000µg/mL in methanol, 1mL/ampul

Each	5-pk.	10-pk.
31415	31415-510	—
w/data pack		
31415-500	31415-520	31515

2,4-Dinitrophenol

1,000µg/mL in methanol, 1mL/ampul

Each	5-pk.	10-pk.
31291	31291-510	—
w/data pack		
31291-500	31291-520	31391

Intermediate Polarity Capillary GC Column for Basic Compounds

Rtx®-35 Amine Column Improves Analysis of Amines and Nitrogen Heterocyclics

By Neil Mosesman, GC Columns Product Marketing Manager

- ✔ Improved responses compared to conventional columns.
- ✔ Symmetrical peaks for basic compounds.
- ✔ Resolve low molecular weight primary amines.

Amines and nitrogen heterocyclics are used to manufacture a wide variety of products, including dyes, chelating agents, stabilizers, pesticides, and pharmaceuticals. Gas chromatographic analysis of these and other basic compounds can be difficult, because the active compounds typically exhibit adsorption and peak tailing. An Rtx®-35 Amine column is ideal for analyses of these polar and low molecular weight amines.

The proprietary deactivation of the 35% phenyl Rtx®-35 Amine phase improves response and peak symmetry compared to conventional 35% phenyl columns. A test mixture of amines at concentrations of 10 to 15ng/µL was analyzed on an Rtx®-35 Amine column and a conventional 35% phenyl column. Ethanolamines are particularly difficult to analyze because in addition to their basic nature they have a hydroxyl group that can interact with silanol groups on the inner surface of the column. The conventional column completely adsorbed diethylenetriamine and diethanolamine, but the Rtx®-35 Amine column gave excellent responses and peak shapes for all compounds (Figure 1).

Primary amines often are analyzed on a Stabilwax®-DB column because it resolves these compounds well. However, the maximum operating temperature of this column, 220ºC, limits the molecular weight range of the analytes. Alternatively, an Rtx®-5 Amine column has a much higher maximum operating temperature, 315ºC, but does not adequately resolve primary amines. An Rtx®-35 Amine column combines the advantages of a Stabilwax®-DB column and an Rtx®-5 Amine column: it resolves primary amines and has high thermal stability. An Rtx®-35 Amine column offers excellent peak shape and high responses for these active compounds (Figure 2).

When analyzing basic drugs by GC, derivatization often is required to reduce peak tailing or improve response. These complex procedures can improve quantitative results, but they add time and cost to the analysis. An Rtx®-35 Amine column provides the selectivity to analyze a wide range of underivatized drug compounds. Many over-the-counter and prescribed medications for cold and flu relief contain amines and other basic compounds. The unique deactivation and selectivity of an Rtx®-35 Amine column, combined with its high thermal stability, simplifies the analysis for these components (Figure 3). Underivatized sympathomimetic amines also exhibit excellent peak shape and separation on an Rtx®-35 Amine column (request lit. cat.# 59380).

Because the Rtx®-35 Amine column offers excellent response and peak symmetry for amines and polar basic compounds, and has high thermal stability, analysts working with these analytes can improve the reliability and consistency of their data.

Figure 1 An Rtx®-35 Amine column minimizes adsorption and improves responses for amines.

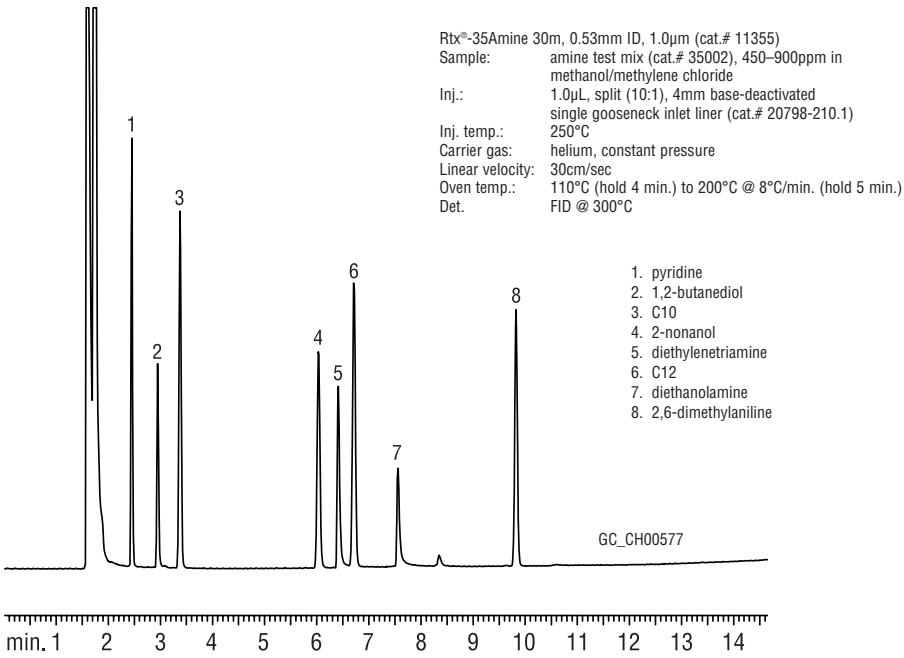
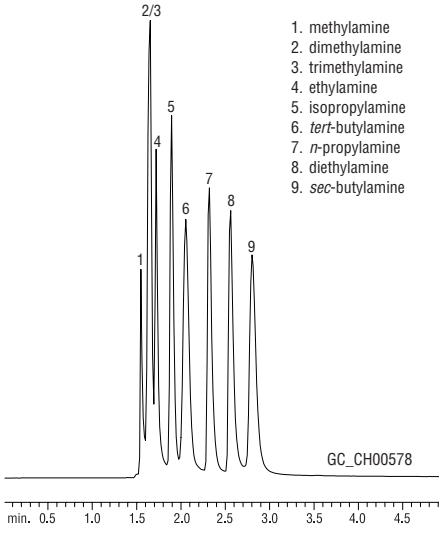


Figure 2 An Rtx®-35 Amine column offers good resolution of primary amines.



Rtx®-35 Amine 30m, 0.53mm ID, 1.0µm (cat.# 11355)
Sample: primary amines, 50ppm on-column conc. in water
Inj.: 1.0µL, split (10:1), 4mm base-deactivated single gooseneck inlet liner (cat.# 20798-210.1)
Injector: 250°C
Carrier gas: helium, constant pressure
Linear velocity: 35.7cm/sec
Oven temp.: 35°C (hold 5.0 min.)
Det.: FID @ 300°C

Base-Deactivated Inlet Liners

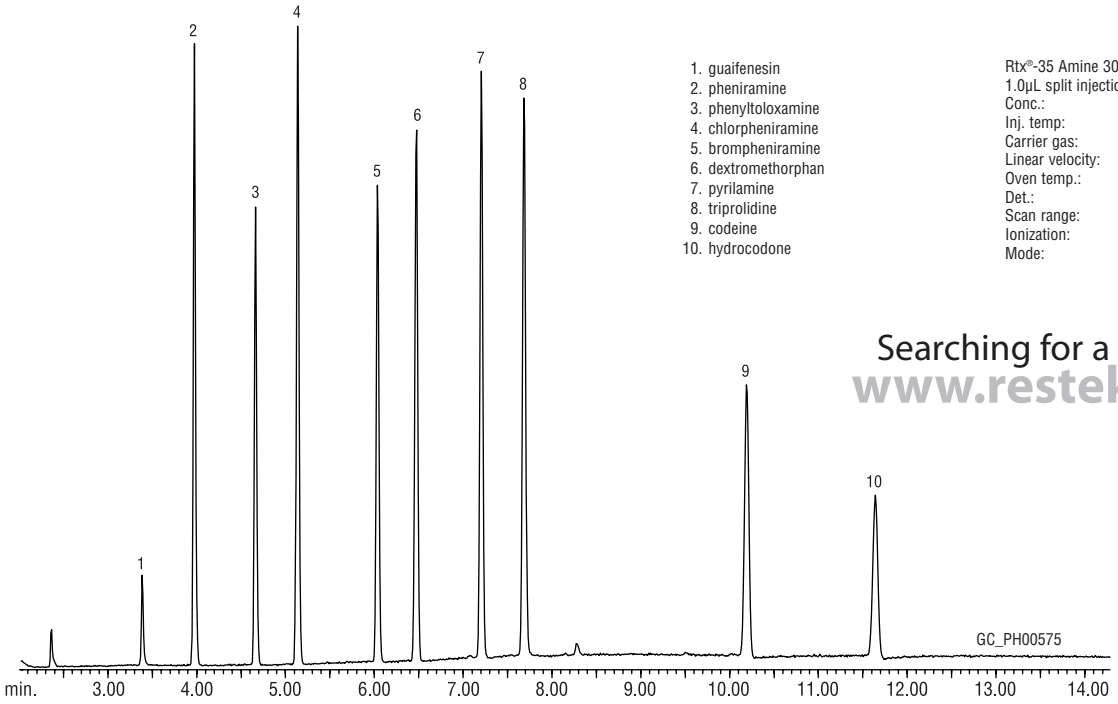
Add the corresponding suffix number to the liner catalog number. For inlet liners, see our 2004 *Chromatography Products Guide* (lit. cat.# 59854).

qty.	Base-Deactivated		Base-Deactivated w/ Base-Deactivated Wool	
each	-210.1	addl. cost	-211.1	addl. cost
5-pk.	-210.5	addl. cost	-211.5	addl. cost
25-pk.	-210.25	addl. cost	-211.25	addl. cost

For more info

For chromatograms of ethanolamines and amphetamines on Rtx®-35 Amine columns, request lit. cat.# 59380.

Figure 3 An Rtx®-35 Amine column simplifies GC/MS analysis of common cold and flu medications.



1. guaifenesin
2. pheniramine
3. phenyltoloxamine
4. chlorpheniramine
5. brompheniramine
6. dextromethorphan
7. pyrilamine
8. triprolidine
9. codeine
10. hydrocodone

Rtx®-35 Amine 30m, 0.25mm ID, 0.50µm (cat.# 11338)
1.0µL split injection of underivatized cold medicines

Conc.:	1000ng/µL
Inj. temp:	250°C
Carrier gas:	helium
Linear velocity:	30cm/sec.
Oven temp.:	250°C to 300°C @ 7°C/min. (hold 7 min.)
Det.:	MSD
Scan range:	40–450amu
Ionization:	EI
Mode:	scan

Rtx®-35 Amine Columns (fused silica)

(Crossbond® 35% diphenyl/65% dimethyl polysiloxane)

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.50	0 to 290/310°C	11335	11338
	1.00	0 to 280/300°C	11350	11353
0.32mm	1.00	0 to 280/300°C	11351	11354
	1.50	0 to 270/290°C	11366	11369
0.53mm	1.00	0 to 260/280°C	11352	11355
	3.00	0 to 240/260°C	11382	11385

Base-Deactivated Guard Columns

For analyzing basic compounds, use a base-deactivated guard column. For a 5m base-deactivated guard column, order cat. # 10000 ($45), 10001 ($50), or 10002 ($65) (0.25, 0.32, or 0.53mm ID, respectively). For more information on guard columns, see our 2004 *Chromatography Products Guide* (lit. cat.# 59854).

Fast Analysis of Semivolatile Organic Analytes

Using a 0.18mm ID Rtx®-5Sil MS Column, continued from page 3

For stability, two analytes in Figure 1, benzoic acid and benzidine, are introduced separately (cat.# 31415 and cat.# 31441, respectively). 2,4-Dinitrophenol, a component of the 8270 MegaMix™, is supplemented (cat.# 31291), to double the on-column concentration for this low-level calibration (<20ng on column).

For analysts who cannot use the MegaMix™, we offer six simpler calibration mixes of Method 8270 semivolatiles, formulated by chemical class (8270 Calibration Mix #1—8270 Calibration Mix #6, cat.#s 31618 –31623, described in the 2004 Restek catalog, page 359), and Organochlorine Pesticide Mix AB #3 (cat.# 32415, catalog page 358). EPA Appendix IX Mix #1 and Appendix IX Mix #2 (cat.#s 31625 and 31806, catalog page 358) complement this full set of mixes.

We developed each of these mixes, including the MegaMix™ mix, for maximum stability, through careful consideration of chemical properties of all potential components. Because 3-methylphenol and 4-methylphenol coelute, we include each in the 8270 MegaMix™ mix at half the concentration of the other components, to enable the user to calibrate at lower levels to quantify these compounds at the required limits. N-nitrosodiphenylamine, an amine target compound in Method 8270D, readily oxidizes to diphenylamine and nitric oxide, a highly reactive gas that can participate in many chemical reactions or act as a catalyst for other oxidation and reduction reactions in the mix. Consequently, we include diphenylamine, rather than N-nitrosodiphenylamine, in the 8270 MegaMix™ mix, to prevent degradation of other components of the mix. Another target compound, diphenylhydrazine, also oxidizes easily, forming azobenzene, so we include azobenzene in the 8270 MegaMix™ mix to assure stability. The stability of an unopened ampul of 8270 MegaMix™ mix is 18 months, as determined by real-time analysis.

In addition to the best choice for analytical column, and stable calibration mixtures, we also have available internal and surrogate standards and the tuning compound recommended in Method 8270D: SV Internal Standard Mix, Acid Surrogate Mix (4/89 SOW), and B/N Surrogate Mix (4/89 SOW), described here (see page 3), and PFTBA (MS Tuning Compound), cat.# 30482, described on catalog page 357.

If you are analyzing for semivolatile compounds by GC/MS, we suggest you evaluate an Rtx®-5Sil MS column and our 8270 MegaMix™ and other reference mixes. Rtx®-5Sil MS columns are available in all common dimensions, or you can use the short, thin-film 20m, 0.18mm ID, 0.18µm column for fastest analyses and highest productivity.

GC/ECD Analysis of Organochlorine Pesticides or Polychlorinated Biphenyls

Using a Low-Bleed Rtx®-XLB Column and Restek Reference Materials

by Greg France, Innovations Chemist, Gary Stidsen, Innovations Team Manager,
and Katia May, Ph.D., Senior R&D Chemist

- ✔ Rtx®-XLB column shows extremely low bleed and excellent inertness, improving sensitivity for active compounds.
- ✔ 20 common organochlorine pesticides in 3 convenient reference concentrations.
- ✔ 19 US EPA Method 8082A PCB congeners in one solution.

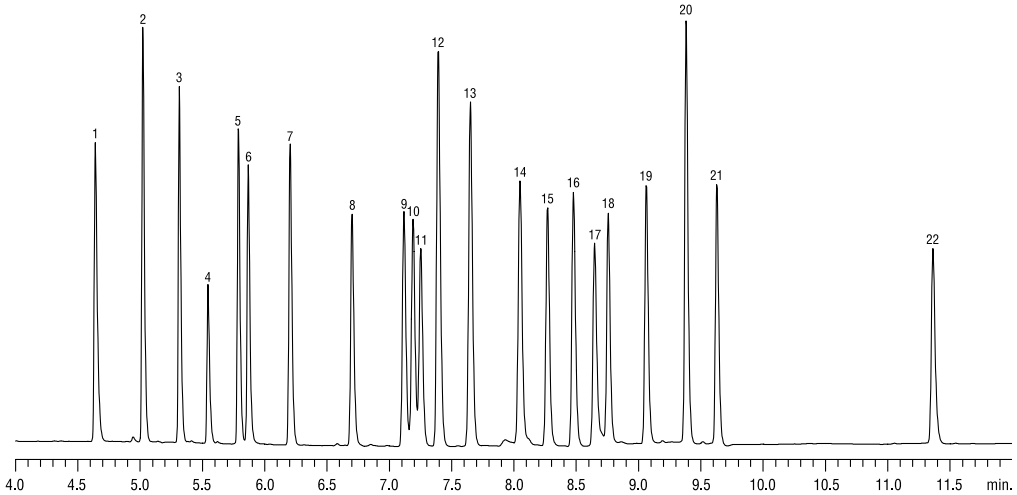
Various methods have provided guidelines for GC/electron capture detection (GC/ECD) analysis of organochlorine pesticides and PCBs in aqueous and soil matrices. Pesticides and PCB congeners now are analyzed by separate methods, to ensure more accurate PCB data and eliminate complications that arise in combined analysis. Analyses of individual PCB congeners greatly simplify quantitative studies, and improve data, relative to the difficult quantitative studies of PCBs as mixtures (e.g., Aroclor® mixtures)—especially with mixtures weathered by long exposure in the environment.

GC analysis of organochlorine pesticides and PCBs can be very challenging because of lengthy calibrations, linearity problems, and potential breakdown of some of the pesticides. In addition to adequate resolution of the target analytes, the column for this analysis must exhibit low bleed. A wide-bore (0.53mm ID) column is listed in US EPA Methods 8081A and 8082A for organochlorine pesticides, but a narrow-bore column may be used in single-column analyses. Our new 0.32mm ID, 0.5µm phase Rtx®-XLB column is ideal for analyses of active compounds, due to improvements in polymer synthesis and tubing deactivation. Figure 1, an analyses of 20 organochlorine pesticides (Organochlorine Pesticide Mix AB #2, cat.# 32292), demonstrates the superior efficiency and low bleed characteristic of the new column, even at 330ºC. The column, in combination with a high initial temperature, 120ºC, reduced analysis time to 11.5 minutes, with excellent separation. Very low bleed and high thermal stability ensure reliable detection at the 80/160/800 ppb level. The very low bleed also minimizes detector contamination, prolonging intervals between cleanings and thus increasing throughput over time. Note that to minimize breakdown of labile pesticides we minimized sample contact with metal surfaces by using a Drilled Uniliner® inlet liner to convey the sample directly onto the column.

Restek chemists carefully reviewed EPA Methods 8080 and 8081A, then developed three calibration mixes that include 20 most often monitored organochlorine pesticides. The mix used to obtain Figure 1 has varied concentrations of the target analytes, from 8 to 80µg/mL, because these pesticides exhibit significantly differing responses.* The other two mixes include the 20 analytes at a single concentration, 200µg/mL or 2000µg/mL. The 2000µg/mL concentration often is more practical than lower concentrations, especially if several mixes must be combined. We also offer all surrogates and internal standards currently required for these analyses.

PCBs are persistent in the environment, and accurately determining their presence and concentrations is very important. A common question is whether such analyses should be focused on mixtures of PCBs (e.g., Aroclor® mixes) or on individual congeners. Congener-specific analyses have important advantages over analyses of mixtures: generally, congener analyses offer lower detection limits and greater information content. In addition, compositions of weathered, degraded, and metabolized PCB mixtures can be measured and interpreted more easily. Also, it is easier to detect interferences caused by other chemicals, and quantification of individual congeners is more accurate. However, coelution of analytes is a problem in a PCB congener analysis, so a strong quality assurance program and reliable reference materials are needed by the analyst. To facilitate congener-specific analyses, we now make a reference mix of 19 PCB congeners at 100µg/mL each in isooctane, suitable for EPA Method 8082A. Depending on regulatory and project requirements,

Figure 1 Organochlorine pesticides separated in less than 12 minutes, using an Rtx®-XLB column.



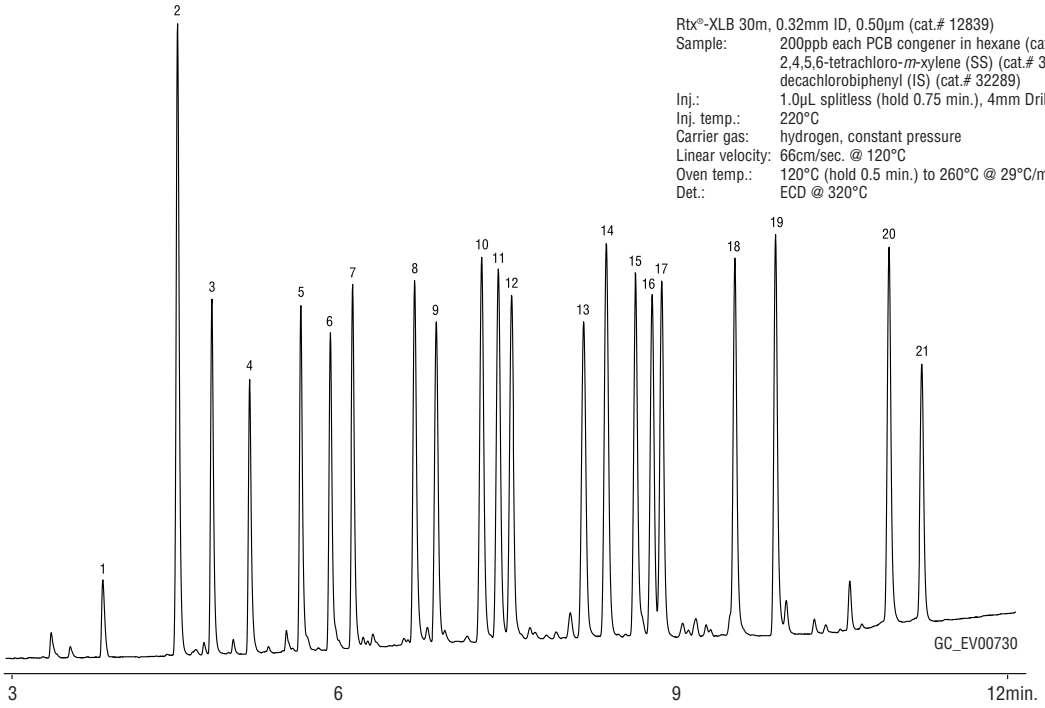
GC_EV00720

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

Rtx®-XLB 30m, 0.32 ID, 0.5µm (cat.# 12839)
Sample: Organochlorine Pesticide Mix AB (cat.# 32292) 80/160/800ppb in hexane
2,4,5,6-tetrachloro-*m*-xylene (cat.# 32027) surrogate, 80ppb
decachlorobiphenyl (cat.# 32029) surrogate, 160ppb
Inj.: 1.0µL splitless (0.75 min. hold), 4mm Drilled Uniliner® inlet liner (cat.# 21055)
Inj. temp.: 220°C
Carrier gas: hydrogen, constant pressure
Linear velocity: 60cm/sec. @ 120°C
Oven temp.: 120°C (hold 0.5 min.) to 260°C @ 29°C/min. (hold 2.5 min.), to 330°C @ 28°C/min. (hold 3 min.)
Det.: ECD @ 320°C

*For mix composition, see page 8 of this *Advantage*.

Figure 2 19 PCB congeners separated in less than 12 minutes, using an Rtx®-XLB column.



the mix can be used for reporting either PCB congener results or total PCBs. Decachlorobiphenyl and tetrachloro-*m*-xylene are appropriate as internal standard and surrogate standard, respectively. The PCB congener standard is a very useful addition to our group of Aroclor® reference mixes.

Figure 2 is a GC/ECD analysis of the 19 PCB congeners, with the internal and surrogate standards. To simplify the work of analysts who monitor both pesticides and PCBs, we used the same 30m, 0.32mm ID, 0.5µm Rtx®-XLB column and the same conditions in both analyses: the conditions used to obtain Figure 2 are optimal for very rapid analysis (11.5 min.) of the Method 8082A PCB congeners, as was true for the pesticides.

If you are performing analyses of organochlorine pesticides and/or PCBs, an Rtx-XLB® column and Restek reference materials will save time, help simplify your analysis, improve the quality of your data, and increase your productivity.

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	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.

Additional reference mixes listed on page 8.

For more **info** on the Rtx®-XLB column, request lit. cat.# 59957.

Suitable for ***European Methods or ASTM D-4059-96***

PCB Congener Mix, Method 8082A

2-chlorobiphenyl (BZ #1)
2,3-dichlorobiphenyl (BZ #5)
2,2',5-trichlorobiphenyl (BZ #18)
2,4',5-trichlorobiphenyl (BZ #31)
2,2',3,5'-tetrachlorobiphenyl (BZ #44)
2,2',5,5'-tetrachlorobiphenyl (BZ #52)
2,3',4,4'-tetrachlorobiphenyl (BZ #66)
2,2',3,4,5'-pentachlorobiphenyl (BZ #87)
2,2',4,5,5'-pentachlorobiphenyl (BZ #101)
2,3,3',4',6-pentachlorobiphenyl (BZ #110)
2,2',3,4,4',5'-hexachlorobiphenyl (BZ #138)
2,2',3,4,5,5'-hexachlorobiphenyl (BZ #141)
2,2',3,5,5',6-hexachlorobiphenyl (BZ #151)
2,2',4,4',5,5'-hexachlorobiphenyl (BZ #153)
2,2',3,3',4,4',5-heptachlorobiphenyl (BZ #170)
2,2',3,4,4',5,5'-heptachlorobiphenyl (BZ #180)
2,2',3,4,4',5',6-heptachlorobiphenyl (BZ #183)
2,2',3,4',5,5',6-heptachlorobiphenyl (BZ #187)
2,2',3,3',4,4',5,5',6-nonachlorobiphenyl (BZ #206)

100µg/mL each in isooctane, 1mL/ampul

Each	5-pk.	10-pk.
32416	32416-510	—
	w/data pack	
32416-500	32416-520	32516

PCB Congener Standard #1

2,4,4'-trichlorobiphenyl (BZ #28)
2,2',5,5'-tetrachlorobiphenyl (BZ #52)
2,2',4,5,5'-pentachlorobiphenyl (BZ #101)
2,2',3,4,4',5'-hexachlorobiphenyl (BZ #138)
2,2',4,4',5,5'-hexachlorobiphenyl (BZ #153)
2,2',3,4,4',5,5'-heptachlorobiphenyl (BZ #180)

10µg/mL each in isooctane, 1mL/ampul

Each	5-pk.	10-pk.
32290	32290-510	—
	w/data pack	
32290-500	32290-520	32390

PCB Congener Standard #2

BZ #28, BZ #52, BZ #101, BZ #138, BZ #153, BZ #180, plus 2,3',4,4',5-pentachlorobiphenyl (BZ #118)

10µg/mL each in isooctane, 1mL/ampul

Each	5-pk.	10-pk.
32294	32294-510	—
	w/data pack	
32294-500	32294-520	32394

GC/ECD Analysis of Organochlorine Pesticides or Polychlorinated Biphenyls

continued from page 7

8140/8141 Internal & Surrogate Standards

1,000µg/mL in acetone, 1mL/ampul

Each	5-pk.	10-pk.
Internal Standard: 1-bromo-2-nitrobenzene		
32279	32279-510	—
w/ data pack		
32279-500	32279-520	32379
Surrogate: 4-chloro-3-nitrobenzotrifluoride		
32282	32282-510	—
w/ data pack		
32282-500	32282-520	32382

2,4,5,6-Tetrachloro-*m*-xylene

Each	5-pk.	10-pk.
200µg/mL in acetone, 1mL/ampul		
32027	32027-510	—
w/ data pack		
32027-500	32027-520	32127
200µg/mL in acetone, 5mL/ampul		
32028	32028-510	—
w/ data pack		
32028-500	32028-520	32128

Decachlorobiphenyl (BZ #209)

Each	5-pk.	10-pk.
10µg/mL in isooctane, 1mL/ampul		
32289	32289-510	—
w/ data pack		
32289-500	32289-520	32389
200µg/mL in acetone, 1mL/ampul		
32029	32029-510	—
w/ data pack		
32029-500	32029-520	32129
200µg/mL in acetone, 5mL/ampul		
32030	32030-510	—
w/ data pack		
32030-500	32030-520	32130

508.1 Internal Standard

pentachloronitrobenzene

100µg/mL in ethyl acetate, 1mL/ampul

Each	5-pk.	10-pk.
32091	32091-510	—
w/data pack		
32091-500	32091-520	32191

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 (isomer B)	8
4,4'-DDT	16	methoxychlor	80

In hexane:toluene (1:1), 1mL/ampul

Each	5-pk.	10-pk.
32292	32292-510	—
w/data pack		
32292-500	32292-520	32392

Organochlorine Pesticide Mix AB #1

20 compounds listed for cat.# 32292 above

200µg/mL each in hexane:toluene (1:1), 1mL/ampul

Each	5-pk.	10-pk.
32291	32291-510	—
w/data pack		
32291-500	32291-520	32391

Organochlorine Pesticide Mix AB # 3

20 compounds listed for cat.# 32292 above

2,000µg/mL each in hexane:toluene (1:1), 1mL/ampul

Each	5-pk.	10-pk.
32415	32415-510	—
w/data pack		
32415-500	32415-520	32515

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Congratulations!

Charles Roberts of Chemtron Corporation won the digital camera in our "Are You Game?" give-away at PittCon® 2004. We hope you're enjoying your camera, Charles.

Many thanks to everyone who visited our booth, and we look forward to seeing you again next year in Orlando.

Improved SilcoCan™ and TO-Can™ Canisters

By Donna Lidgett, Air Monitoring Product Marketing Manager

Restek Innovation!

- ✔ Improved design: canister holder and valve bracket protect the canister, tube stub, and valve.
- ✔ Excellent long-term storage of polar and nonpolar volatile organics in ambient air.
- ✔ Eliminate adsorption of active compounds.

Optional gauge
- Quickly confirm vacuum or pressure inside canister.
- Monitor pressure changes.
- Fully protected by canister frame.
- Can be heated to 90°C during cleaning.

Newest coating technology

For high inertness, and to ensure sample stability, SilcoCan™ canisters are now deactivated with Restek's latest innovative surface treatment, which chemically bonds to the metal inner surface of the canister. This coating offers unsurpassed inertness for active compounds, including polar and sulfur-containing molecules, and will not crack, chip, or flake off, despite harsh handling in the field or during transport.

Enhanced valve and canister bracket

Canister holder and valve bracket protect canister, tube stub, and valve.

1/4" tube stub

Allows user to interchange valves.

Serial controlled

For quick, sure identification.

Improved SilcoCan™ Canisters (1/4" Valve)

	w/Non-Treated Valve			w/Silcosteel®-Treated Valve		
volume	qty.	cat.#	price	qty.	cat.#	price
1L	ea.	24180		ea.	24180-650	
3L	ea.	24181		ea.	24181-650	
6L	ea.	24182		ea.	24182-650	
15L	ea.	24183		ea.	24183-650	

	w/Gauge & Non-Treated Valve			w/Gauge & Silcosteel®-Treated Valve		
volume	qty.	cat.#	price	qty.	cat.#	price
1L	ea.	24140		ea.	24140-650	
3L	ea.	24141		ea.	24141-650	
6L	ea.	24142		ea.	24142-650	
15L	ea.	24143		ea.	24143-650	

Improved TO-Can™ Canisters (1/4" Valve)

volume	qty.	cat.#	price
1L	ea.	24172	
3L	ea.	24173	
6L	ea.	24174	
15L	ea.	24175	

Improved TO-Can™ Canisters (1/4" Valve, with Gauge)

volume	qty.	cat.#	price
1L	ea.	24176	
3L	ea.	24177	
6L	ea.	24178	
15L	ea.	24179	

1/4" Replacement Valves for Air Monitoring Canisters

	Non-Treated Valve			Silcosteel®-Treated Valve		
Description	qty.	cat.#	price	qty.	cat. #	price
1/4" Replacement Valve (2-port)	ea.	24145		ea.	24144	
1/4" Replacement Valve (3-port)	ea.	24147		ea.	24146	

Air Canister Heating Jacket

The ultimate in controlled heating, for reliably cleaning your air canisters!

new!

- Closely simulates oven environment—heats entire canister.
- Easily fits canister up to 6 liters.
- Prevents sample condensation, for accurate sub-sampling.
- Lightweight; comfortable to the touch when heated.
- Connect up to five Canister Heating Jackets to one 15 amp circuit.

Whether you made your canister cleaning system or purchased a commercial system, the new Restek Canister Heating Jacket will help you clean your canisters faster and more efficiently. The novel design ensures the entire canister, including the valve, is heated during the cleaning cycle, to remove contaminants most effectively. It also can be used to keep the sample heated during aliquot removal, which helps prevent condensation and assure accurate data for larger molecules. The Canister Heating Jacket incorporates two heat settings—low (75°C) and high (150°C)—to let you match the temperature to the volatility of your sample components. Connect up to five Canister Heating Jackets to one 15 amp circuit. If you try one in your system, we think you'll want more.

Description	qty.	cat.#	price
Air Canister Heating Jacket (110 volt)	ea.	24123	

Restek
Performance
Coatings

Inert, High-Quality Fittings and Tubing for Demanding Applications

by Gary Barone, Restek Performance Coatings Division

Siltek™/Sulfinert® and Silcosteel®-CR Treated Swagelok® Fittings

- ✔ Siltek™/Sulfinert® treatment* ensures ultimate inertness.
- ✔ Silcosteel®-CR treatment enhances acid resistance tenfold, or more.
- ✔ Restek treatments cannot chip, flake, or delaminate.
- ✔ Custom treatment available.

Swagelok® fittings are world-renowned for meeting demanding standards. Now, Restek is pleased to set the new standard for inert or corrosion-resistant tubing system components: Swagelok® products with Restek's unparalleled surface treatments—Siltek™ or Silcosteel®-CR treatment—available from stock.

Siltek™ treatment, which is equivalent to Sulfinert® treatment, is the ideal choice for ultimate inertness, intended specifically for systems used to collect, store, and transfer active compounds. The most reactive sample components can be stored and transferred via a Siltek™ treated system: even at parts-per-billion levels, sulfur-containing or other very active compounds exhibit virtually no adsorption. And, unlike coatings, Siltek™ and other Restek treatments produce a layer that is integral with the fitting surface—it will not chip, flake, or delaminate, even in the most stressful applications.

Silcosteel®-CR treatment is highly effective protection for stainless steel exposed to hydrochloric, nitric, or sulfuric acid, or to marine environments. In independent tests, Silcosteel®-CR treatment upgraded the corrosion resistance of 300-grade stainless steel samples by an order of magnitude (Table 1) and totally protected them against crevice corrosion (Figure 1).

If you need to construct a system for a demanding application, you will not find more suitable fittings than Restek treated Swagelok® fittings. Siltek™, Silcosteel®-CR, or other Restek surface treatments can be applied to other fittings or parts on request—contact our Technical Service chemists or your Restek representative.

Fitting Type	Size	Similar to Swagelok® #	Siltek™/Sulfinert® qty.	Siltek™/Sulfinert® cat.#	Siltek™/Sulfinert® price	Silcosteel®-CR qty.	Silcosteel®-CR cat.#	Silcosteel®-CR price
Union	1/16"	SS-100-6	ea.	22540		ea.	22575	
	1/8"	SS-200-6	ea.	22541		ea.	22576	
	1/4"	SS-400-6	ea.	22542		ea.	22577	
Tee	1/16"	SS-100-3	ea.	22543		ea.	22578	
	1/8"	SS-200-3	ea.	22544		ea.	22579	
	1/4"	SS-400-3	ea.	22545		ea.	22580	
Reducing Union	1/8" to 1/16"	SS-200-6-1	ea.	22546		ea.	22581	
	1/4" to 1/16"	SS-400-6-1	ea.	22547		ea.	22582	
	1/4" to 1/8"	SS-400-6-2	ea.	22548		ea.	22583	
Union Elbow	1/8"	SS-200-9	ea.	22549		ea.	22584	
	1/4"	SS-400-9	ea.	22550		ea.	22585	
Plug	1/16"	SS-100-P	ea.	22572		ea.	22619	
	1/8"	SS-200-P	ea.	22573		ea.	22620	
	1/4"	SS-400-P	ea.	22574		ea.	22597	
Cross	1/8"	SS-200-4	ea.	22551		ea.	22586	
	1/4"	SS-400-4	ea.	22552		ea.	22587	
Tube End Reducer	1/8" tube to 1/16"	SS-100-R-2	ea.	22553		ea.	22588	
	1/4" tube to 1/16"	SS-100-R-4	ea.	22554		ea.	22589	
	1/8" tube to 1/4"	SS-400-R-2	ea.	22555		ea.	22590	
	1/4" tube to 1/8"	SS-200-R-4	ea.	22556		ea.	22591	
Port Connector	1/8"	SS-201-PC	ea.	22557		ea.	22592	
	1/4"	SS-401-PC	ea.	22558		ea.	22593	
	1/8" tube to 1/4"	SS-401-PC-2	ea.	22559		ea.	22594	
Male Connector	1/8" to 1/8" NPT	SS-200-1-2	ea.	22561		ea.	22595	
	1/4" to 1/4" NPT	SS-400-1-4	ea.	22562		ea.	22596	
	1/16" to 1/8" NPT	SS-100-1-2	ea.	22563		ea.	22610	
	1/8" to 1/4" NPT	SS-200-1-4	ea.	22564		ea.	22611	
	1/4" to 1/8" NPT	SS-400-1-2	ea.	22565		ea.	22612	
Female Connector	1/8" to 1/8" NPT	SS-200-7-2	ea.	22566		ea.	22613	
	1/4" to 1/4" NPT	SS-400-7-4	ea.	22567		ea.	22614	
	1/4" to 1/8" NPT	SS-400-7-2	ea.	22568		ea.	22615	
	1/8" to 1/4" NPT	SS-200-7-4	ea.	22569		ea.	22616	
Bulkhead Union	1/8"	SS-200-61	ea.	22570		ea.	22617	
	1/4"	SS-400-61	ea.	22571		ea.	22618	

Table 1 Silcosteel®-CR treated stainless steel coupons show little weight loss after exposure to 6% w/w ferric chloride solution.

Sample	Weight Loss (g/m²)
Silcosteel®-CR 17	19
Silcosteel®-CR 28	25
Silcosteel®-CR 47	25
Bare Steel 27	231
Bare Steel 34	209
Bare Steel 37	228

Figure 1 Silcosteel®-CR treated 316L stainless steel coupons show no crevice corrosion and only slight pitting corrosion (top), while bare 316L stainless steel coupons exhibit severe crevice corrosion (bottom).

Siltek™ and Silcosteel®-CR Treated Electropolished Stainless Steel Tubing

- ✔ Exceptional inertness.
- ✔ Improved reliability and reproducibility; longer lifetime.
- ✔ Use with treated fittings for the most inert sample pathway available.

Restek also sets the highest standard in transfer tubing for analytical and process applications. The near-mirror finish of this electropolished tubing (surface roughness of only 5-7 micro-inches) creates a very small surface area that, in combination with unequalled Restek surface treatments, ensures superior inertness (Siltek™) or greatly enhanced corrosion resistance (Silcosteel®-CR). Further, we can provide continuous coils of 1/8" tubing up to 100 feet (30.5m) or 1/4" tubing up to 300 feet (91.4m)—a first for electropolished tubing.

The extremely inert Siltek™ surface is ideal in sulfurs or automotive exhaust testing, stack gas sampling, process monitoring, or any other application in which a representative sample must be transferred without loss.

In systems used to transfer hydrochloric, nitric, sulfuric, or other acids, or seawater, Silcosteel®-CR treated electropolished stainless steel tubing will last longer and require less maintenance. Silcosteel®-CR treated samples were very well protected from pitting and crevice corrosion, compared to bare steel samples (Table 1 and Figure 1, p. 10).

For maximum inertness, we recommend a sample transfer system constructed from Restek treated electropolished stainless steel tubing and Restek treated Swagelok® fittings. To find out how Restek treated components will improve your system's performance, contact our Technical Service Group (ext. 4), or your Restek representative, and ask to speak with our coatings experts.

Siltek™/Sulfinert® Treated Electropolished Stainless Steel Tubing

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

Silcosteel®-CR Treated Electropolished Stainless Steel Tubing

			Price-per-foot			
ID	OD	cat.#	5-24 ft.	25-99 ft.	100-299 ft.	>300 ft.
0.085"	1/8"	22536				
0.180"	1/4"	22537				

Restek Innovation!

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.

Deactivating Glass Surfaces with Dimethyldichlorosilane (DMDCS)

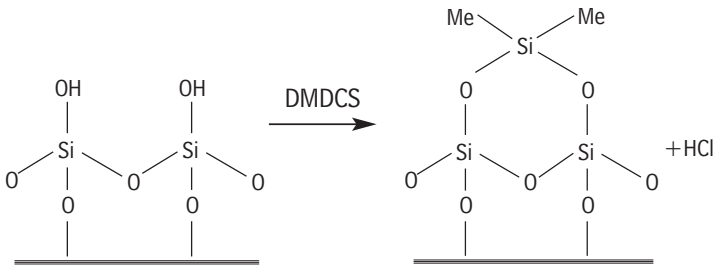
by Jack Crissman, Ph.D., Analytical Reference Materials Product Marketing Manager

- ✔ Convenient 20mL ampuls.
- ✔ Unopened ampuls have long shelf life.
- ✔ Detailed deactivation procedure available on request.

Although glass is widely thought of as an "inert" material, glass surfaces are, in fact, slightly acidic and highly adsorptive, due to the presence of silanol groups (SiOH). These reactive groups interact via hydrogen bonding with amine (-NH), carboxylic acid (-COOH), hydroxyl (-OH), or thiol (-SH) functional groups, and compounds containing these groups adsorb to untreated glass surfaces. To minimize adsorption in sample preparation glassware and in the GC sample pathway, and prevent chromatographic tailing or loss of sensitivity at low sample concentrations, it is important to eliminate or mask the reactive silanol groups.

One popular way to deactivate glass surfaces is to chemically bond a non-adsorptive molecule to the active silanol groups (Figure 1). This typically is accomplished using dimethyldichlorosilane—DMDCS. The procedure is suitable for most analyses that involve concentrated samples and non-active matrices. It can be followed to clean and deactivate glass GC inlet liners, derivatization vials, and all glassware used for preparing analytical reference materials. Restek now offers DMDCS in 20mL ampuls, for analysts who wish to deactivate their glassware themselves.

Figure 1 Dimethyldichlorosilane deactivates silanol groups on a glass surface.



Dimethyldichlorosilane (DMDCS)

Neat, 20mL/ampul

Each	5-pk.
31840	31840-510

Alternative Surface Treatments

An alternative procedure, polymeric deactivation, provides maximum coverage of glass surfaces and should be used to treat inlet liners for critical analyses involving very low concentrations of highly active compounds (e.g., endrin, DDT, drugs). All liners supplied by Restek undergo polymeric deactivation.

For **ultimate inertness**, and most accurate data for trace levels of reactive analytes, we recommend Siltek™ deactivation. Siltek™ deactivated guard columns, inlet sleeves, and other glassware are listed in the Restek catalog. For other items, ask our Technical Service chemists or your Restek representative about deactivation.

Searching for the Perfect Solution?

Let Restek create the perfect reference mixture—to your exact specifications. Contact the Technical Service Team or visit us online at **www.restekcorp.com/solutions**

Restek HPLC Column Kits for Faster, Easier Method Development

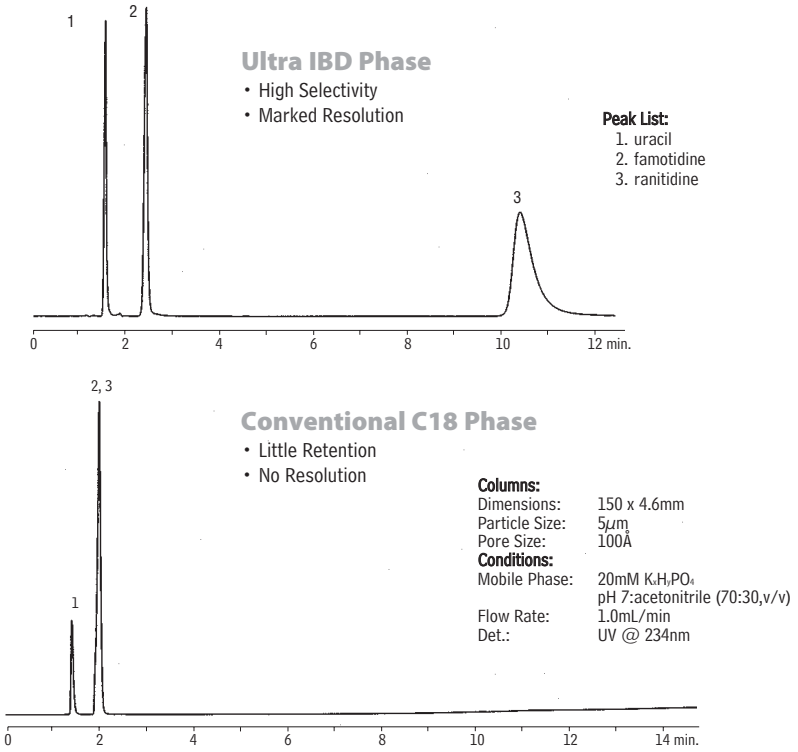
by Rebecca Wittrig, Ph.D., HPLC Product Marketing Manager

- ✔ Multiple stationary phases, for quick optimization of selectivity.
- ✔ Specific kits for MS or UV detection—both include columns specifically for basic analytes.
- ✔ Fast LC kits use economical cartridge design.

When developing a new HPLC assay, method development chemists often start with a C18 or C8 stationary phase, because these phases have proven useful for analyzing a wide range of organic compounds. Many analysts have learned, however, that a C18 or C8 stationary phase is not the best choice for every separation. A cyano-, pentafluorophenyl, or amino-containing stationary phase, a phase with embedded polar groups, or a phase designed for compatibility with highly aqueous mobile phases might provide superior resolution of target compounds (Figure 1).

To help analysts efficiently select the optimum stationary phase, we have assembled kits of columns for use in HPLC method development. The four columns in each kit incorporate a range of stationary phase types, are configured for rapid analyses, and are optimized for the detector type: 50 x 4.6mm ID columns containing 5µm packings for use with UV detection, 30 x 2.1mm ID columns containing 3µm packings for LC/MS. The Fast LC method development kits include four 30 x 2.1mm ID or four 30 x 4.0mm ID cartridges containing 3µm packings, and a cartridge holder.

Figure 1 Better chromatography for basic molecules, using an Ultra IBD column. The base-deactivated column has a unique blend of hydrophobic and polar character for better resolution of closely related compounds.



HPLC and LC/MS Column Kits

Description	qty.	cat.#	price
LC/MS (1 Allure™ C18 (USP L1), 1 Allure™ PFP Propyl, 1 Allure™ Basix (USP L10), 1 Ultra Aqueous C18 (USP L1))	four 30 x 2.1mm, 3µm columns	25136	
LC/UV (1 Pinnacle™ DB C18 (USP L1), 1 Allure™ PFP Propyl, 1 Ultra Aqueous C18 (USP L1), 1 Ultra IBD)	four 50 x 4.6mm, 5µm columns	25137	

Fast LC Method Development Kit

Each kit includes four Fast LC Cartridges (Ultra C18 (USP L1), Ultra Aqueous C18 (USP L1), Ultra Cyano (USP L10), Ultra PFP (USP L43)) and a Fast LC cartridge holder.

Description	qty.	cat.#	price
Fast LC Development Kit	four 30 x 2.1mm columns	25296	
Fast LC Development Kit	four 30 x 4.0mm columns	25297	

If the columns in the kits listed below don't appear to meet your needs, please contact our Technical Service group or your Restek distributor for information about custom kits.

Bulk Silica Available!

High Performance Silica Products
(lit. cat.# 59901)
Tight production specifications ensure highly consistent lot-to-lot performance from Restek's proprietary silica materials. This 6-page publication characterizes Pinnacle II™ and Pinnacle™ DB stock bulk products—bare silica and silica with popular bonded phases—and illustrates typical results achieved by using them. Custom materials can be designed from a broad range of particle sizes, pore sizes, and phases.

HPLC Method Development Course

Many analysts anticipate developing a new HPLC method with apprehension. To help de-mystify this process, Restek, in cooperation with ChromVision, offers a dynamic, thorough two-day course on the subject, presented in a logical and systematic manner by an expert on the effects of adsorbent chemistry and structure on HPLC retention. It provides the knowledge and tools necessary for understanding why a particular stationary phase would be chosen for separating various analytes, and how to improve selectivity by choosing the proper eluent. Eight lectures cover all aspects of method development and are complemented and reinforced by four workshops. The course is especially useful to analysts working with pharmaceutical or biological/biochemical analytes. For more information about this and other Restek seminars, visit our seminars web page: **www.restekcorp.com/seminar**

Genuine Restek Replacement Parts for Beckman and Hitachi HPLC Systems

Keep Your System Running Smoothly

by Rebecca Wittrig, Ph.D., HPLC Product Marketing Manager

- ✔ Designed to meet or exceed original equipment performance.
- ✔ Simplify ordering—a single source for columns, tools, parts, reference mixes.
- ✔ Renowned Plus 1™ service.

Restek Replacement Parts for Beckman HPLC Systems

Description	Model #	Similar to Beckman part #	qty.	cat.#	price
Inlet Check Valve Assembly	110A&B, 112, 114M, 116, 118, 125, 126, 127, 128	240720	ea.	25439	
Outlet Check Valve Assembly	110A&B, 112, 114M, 116, 118, 125, 126, 127, 128	240721	ea.	25440	
Inlet Check Valve Cartridge for Check Valve Assembly	110A&B, 112, 114M, 116, 118, 125, 126, 127, 128	240620	ea.	25441	
Outlet Check Valve Cartridge for Check Valve Assembly	110A&B, 112, 114M, 116, 118, 125, 126, 127, 128	240621	ea.	25442	
Graphite Guide (Bushing)	Pumps	243714	ea.	25443	
Guide Sleeve for Graphite Plunger Guide	110 Series	243713	ea.	25444	
Outlet Check Filter Frit Assembly	Pumps	240619	ea.	25445	
Piston Guide Assembly	Pumps	243045	ea.	25446	
Plunger	110 Series and 112 Pumps	243053	ea.	25447	
Plunger Seal	110 Series	887138	ea.	25448	
Plunger Seal	112 Pumps	236797	ea.	25449	
Sapphire Plunger	114M, 116, 118, 125, 126, 127, 128 Pumps	240714	ea.	25450	
Plunger Seal	114M, 116, 118, 125, 126, 127, 128 Pumps	241037	ea.	25451	
Pump Seal, Gold	114, 116, 125, 126, 127, 128 Pumps	241037	ea.	25452	
Plunger Wash Seal	Pumps	238627	ea.	25453	
Deuterium Lamp	DU60, 62, 64, 65	596791	ea.	25454	

Restek Replacement Parts for Hitachi HPLC Systems

Description	Model #	Similar to Hitachi part #	qty.	cat.#	price
SS Check Valve Cartridges ($^1/_{16}$" Ruby Ball)	655/6000/6200	AN0-085	2-pk.	25455	
Inlet Check Valve Assembly	655/6000/6200	AN0-0833	ea.	25456	
Outlet Check Valve Assembly	655/6000/6200	AN0-0834	ea.	25457	
Plunger Assembly	655, 6000, 6200, 7100	810-1033	ea.	25458	
Plunger Assembly	L655A, 6000, 6200, 7100	655-1080	ea.	25459	
Plunger Assembly, Gold	655, 6000, 6200, 7100	655-1080	ea.	25460	
Inlet Check Valve Assembly	L-7100	AN0-0836	ea.	25461	
Outlet Check Valve Assembly	L-7100	AN0-0837	ea.	25462	
Rotor Assembly for Dilutor Valve	AS-7200 Autosampler	810-3085	ea.	25463	
Rotor Seal Kit	AS-7200, AS-7250 Injection Valve	AN0-0818	ea.	25464	
Deuterium Lamp, Prealigned	L4000, L4200, L4250, L7400	885-3570	ea.	25465	

Sonic Debubbler

- ✔ Fast and neat—no breaking connections.
- ✔ Easy to use.
- ✔ Less solvent waste; less clean-up.

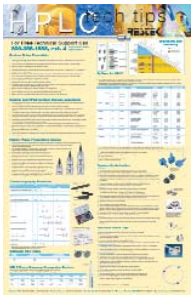
COOL TOOL!

Just touch the Sonic Debubbler to the inlet line or check valve — sonic vibrations will quickly dislodge or redissolve trapped air bubbles. Reduces downtime or conversion time from one mobile phase to another.

Description	qty.	cat.#	price
Sonic Debubbler	ea.	20444	
Sonic Debubbler (220V)	ea.	25098	

Genuine Restek Replacement Parts for HPLC Systems
(lit. cat.# 59012)
A listing of Restek parts for Agilent, Beckman, Hitachi, PerkinElmer, Shimadzu, and Waters instruments.

HPLC Column Selection Guide
(lit. cat.# 59454A)
A useful chart to keep with your workbooks, or post on a wall. Quickly scan important characteristics of Restek HPLC columns. Includes cross-references to similar phases.



HPLC Tech Tips Wall Chart
(lit. cat.# 59894A)
Almost everything you need to remember about HPLC, condensed into 3 feet by 2 feet: mobile phase basics, buffers (types, pK_as, pH ranges, formula masses, more), miscibility and solubility chart (invaluable!), system setup and optimization, detector tips, pressure conversion factors, most-used chromatographic equations, column storage essentials. Post near your instrument to save time; perhaps save a column.

Free! Call our literature hotline: 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative.

PEAK PERFORMERS

Chromatography Accessories and Tools You Can Rely On!

new!

Alumaseal™ Ferrules Eliminate Sealing Compromises

Tired of leaking fittings? Want a ferrule that seals every time?
These new aluminum ferrules combine the sealing ease of graphite with the security and reliability of metal ferrules.

by Brad Rightnour, Instrument Innovations Manager

- ✔ Aluminum construction, will not crack or fragment.
- ✔ Eliminates out-gassing, makes leak-tight seals, for less detector noise.
- ✔ No retightening after temperature cycles—excellent for GC/MS.
- ✔ Unique two-piece design permanently locks on fused silica tubing without causing breakage.
- ✔ Will not stick in fittings, like Vespel® or graphite.
- ✔ Use with any 1/16" compression-type fitting.

Alumaseal™ ferrules* combine the advantages of graphite and Vespel®/graphite ferrules, and eliminate the disadvantages, for enhanced performance in any application. The unique front and back ferrule design seals easily and surely, the ferrules will not fragment or off-gas, and they are stable to 550ºC. Alumaseal™ ferrules will not allow oxygen to permeate into the system, and do not require retightening after temperature cycles, making them ideal for GC/MS applications. They can be used with any standard 1/16" compression-type fitting, with fused silica columns.

For leak-tight seals, we highly recommend you try Alumaseal™ ferrules.

Ferrule ID	Fits Column ID	qty.	cat.#	price
0.4mm	0.25mm	10-pk.	21472	
0.5mm	0.32mm	10-pk.	21473	
0.8mm	0.53mm	10-pk.	21474	

**Patent pending.*

new!

New Vespel® Ferrules

Now—A Complete Range of Ferrule Choices from Restek

by Donna Lidgett, GC Accessories Product Marketing Manager

- ✔ 100% high-temperature Vespel® polyimide.
- ✔ Stable to 350ºC.
- ✔ Durable and leak-tight.

Fitting Size	Ferrule ID	qty.	cat.# / price
Capillary Ferrules for 1/16-inch compression-type fittings			
1/16"	0.3mm	10-pk.	22213
1/16"	0.4mm	10-pk.	22214
1/16"	0.5mm	10-pk.	22215
1/16"	0.8mm	10-pk.	22216
1/16"	1.0mm	10-pk.	22217
1/16"	1.2mm	10-pk.	22218
Standard Ferrules for 1/16-, 1/8-, and 1/4-inch fittings			
1/16"	1/16"	10-pk.	22210
1/8"	1/8"	10-pk.	22211
1/4"	1/4"	10-pk.	22212
1/4"	1/8"	10-pk.	22219

Encapsulated Ferrules

For 1/16-Inch Compression Fittings

- ✔ Will not deform and stick in fittings.
- ✔ Reusable.
- ✔ Less torque needed to seal ferrule.
- ✔ Restek's unique blend of graphite minimizes fragmentation and outgassing.

Ferrule ID	Fits Column ID	cat.#	price/10-pk.
0.4mm	0.25mm	21036	
0.5mm	0.32mm	21037	
0.8mm	0.53mm	21038	

Please note

We offer a wide selection of ferrules. In addition to new Alumaseal™ and Vespel® ferrules,we have Vespel®/graphite, graphite, and Teflon® ferrules. To review these other choices, visit our website or refer to our 2004 catalog.

Restek Solid Phase Extraction Cartridges

For Cleanup of Environmental Samples

by Lydia Nolan, Instrument Support Chemist

- ✔ Special purpose SPE cartridges for specific methods.
- ✔ General purpose cartridges with normal, reversed, or ion exchange phases.
- ✔ Extraction manifolds and pressure/vacuum pump available.

Solid phase extraction is one of the most widely used forms of sample preparation. Ease of use, safety, conservative solvent usage, and cost effectiveness all contribute to its popularity.

We offer a range of SPE cartridges to meet demanding cleanup procedures. Our standard products include Florisil® sorbent, ion exchange resins, normal phase materials, and bonded reversed phase materials. In addition, we offer specialty products, including Florisil® in ultraclean glass tubes, mixed bed sorbents, and cartridges for specific applications. Our SPE manifolds and pressure/vacuum pump, described in our chromatography supplies catalog, will keep your system running efficiently.

Massachusetts TPH Cartridges

- Reduced background extractables.
- Easier quantification.
- Reproducible fractionation patterns, tube after tube, lot after lot.
- Lower cost than glass tubes.
- Efficient processing on vacuum manifolds.

If you are seeing aromatic hydrocarbons in your aliphatics fraction, use our Massachusetts TPH silica gel cartridges. We designed them specifically for separating aliphatic and aromatic hydrocarbons, to meet the requirements of the Massachusetts methodology, which has been adopted by other states as well. Capillary GC columns and reference materials for the Massachusetts methodology are described in Application Essentials 59744, available free on request.

Florisil® Cartridges

Florisil® adsorbent is an excellent material for cleanup of samples containing pesticides and/or PCBs. In addition, it can be used to separate mixtures of aliphatic and aromatic hydrocarbons. An effective cleanup procedure is summarized in Application Note 59562A, available free on request.

Specialty Cartridges

Specifically designed to provide consistent and reproducible results for the listed method or application. Polypropylene tubes with polyethylene frits.

Description	Applications	Tube Volume, Bed Weight	qty.	cat.#	price
Massachusetts TPH	Extraction of hexane-extractable petroleum hydrocarbons from soil and waste samples. Specially treated to reduce contaminants and increase capacity. Silica.	20mL, 5g	20-pk.	26065	
EPA Method 548.1	Extraction of endothal from aqueous samples. Weak anion exchange resin.	6mL	30-pk.	26063	
EPA Method 552.1	Extraction of haloacetic acids from aqueous samples. Strong anion exchange resin.	1mL	100-pk.	26064	
Organo Tin	High-capacity cleanup of butyl and phenyl tin compounds from soil, water, and biota. Mixed bed.	60mL	16-pk.	24049	
RDX	Extraction of explosive compounds (EPA Method 8330) from water samples.	6mL, 500mg	30-pk.	26093	

Normal Phase Cartridges

Hydrophilic (polar) adsorbents used to extract hydrophilic analytes from nonpolar matrices, such as organic solvents (e.g., polar contaminants from sample extracts). Polypropylene tubes with polyethylene frits, except as indicated otherwise.

	3mL/200mg (50-pk.)	3mL/500mg (50-pk.)	6mL/500mg (30-pk.)	6mL/1000mg (30-pk.)
Florisil®	—	24031	—	24034
(EPA SW 846 methods and CLP protocols)	—	24032*	26086**	26085**
Silica (EPA SW 846 methods)	—	24035	—	24038
	—	24036*	—	—

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

Resprep™ 12- or 24-Port Tube Manifolds

- Use with any standard male luer-end SPE tube or cartridge.
- Inert, Teflon® sample guides reduce cross-contamination and carryover.
- Flexible sample collection rack will accommodate a variety of receiving vessels.
- Quick vacuum-release valve for better control.
- Individual vacuum control for each tube—improves reproducibility.

Complete manifold includes glass basin with built-in vacuum regulator, polypropylene top plate with 12 or 24 individual control valves, 12- or 24-position collection rack, and 12 or 24 Teflon® sample guides.

Description	qty.	cat.#	price
Resprep™ 12-Port Manifold	kit	26077	
Resprep™ 24-Port Manifold	kit	26080	

Massachusetts EPH/VPH/APH Methods

(lit. cat.# 59744)

Massachusetts' gas chromatographic methods for volatile (VPH), extractable (EPH), and air phase (APH) fractions of gasoline and other petroleum products have been adopted by other states, and in Canada. This 4-page publication lists many Restek products that can help a laboratory meet the requirements of the Massachusetts methods, including capillary columns (Restek columns are specified in each method), extraction cartridges, analytical reference materials, and air sampling canisters.

Free! Call our literature hotline: 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative.

Restek Seminars for 2004

We're on the Road Again!

by Rick Parmely, Director of Technical Training

Do you question which GC injection technique is best for your sample? Are you uncomfortable when formulating or adjusting HPLC mobile phases?

A comprehensive seminar with the experienced Restek chromatographers will help you reduce guesswork, improve throughput, and obtain more reliable data, without straining your schedule or travel budget. You'll appreciate your investment as you spend less time experimenting with analytical conditions or troubleshooting problems.

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Comprehensive Capillary GC

Date	Location	cat.#
May 25	Nashville, TN	65392
Sept. 13	Boulder, CO	65375
Sept. 13	Blue Ash, OH	65387
Sept. 15	Columbus, OH	65376
Sept. 16	Salt Lake City, UT	65377
Sept. 17	Buffalo, NY	65384
Oct. 14	RTP, NC	65379
Oct. 21	Buena Park, CA	65378

GC/MS

Date	Location	cat.#
Nov. 1	Pleasanton, CA	65390
Nov. 3	Seattle, WA	65391

Comprehensive HPLC

Date	Location	cat.#
Aug. 3	Indianapolis, IN	65381
Oct. 5	Rockville, MD	65374
Oct. 6	Princeton, NJ	65370
Oct. 8	Plymouth Meeting / King of Prussia, PA	65371
Oct. 18	La Jolla / San Diego, CA	65389

HPLC Method Development (two days)

Date	Location	cat.#
June 24/25	Downers Grove, IL	65360
July 15/16	King of Prussia, PA	65361
Sept. 23/24	Indianapolis, IN	65362
Oct. 21/22	Foster City, CA	65363

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By Doug Elliott, STAR™ Service Rewards Coordinator

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THE RESTEK ADVANTAGE

Turning Visions into Reality

2004 vol. 3

Simplify Paraquat/Diquat Analysis and Improve Sensitivity

new!

Using an Ultra Quat HPLC Column, a Simple Mobile Phase, and a New Sample Extraction Process

by Vernon Bartlett, HPLC Manager, Katia May, Ph.D., Senior R&D Chemist, Bruce Albright, HPLC Chemist, Lydia Nolan, Innovations Chemist, and Rebecca Wittrig, Ph.D., HPLC Product Marketing Manager

- Consistent retention and peak symmetry, without costly ion exchange columns.
- Eliminate complicated mobile phases, improve sensitivity by 30%.
- Simplify sample preparation and improve detection limits.

Paraquat (methyl viologen) and diquat are non-selective contact herbicides widely used in agriculture to control broadleaf and grassy weeds (use of paraquat is restricted in the United States). The highly charged dual quaternary amines (Figure 1) are readily soluble in water. They also are highly toxic, and ingestion of either compound can have serious effects.

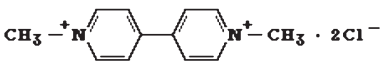
The charged compounds are difficult to retain by standard reversed phase HPLC, so ion pairing reversed phase methods, such as US EPA Method 549, and specialty columns have been developed specifically for this analysis. One widely used approach is to couple an ion exchange column with a post-column reactor that creates a fluorescing complex. Detection is very sensitive, but the columns are costly, often exceeding $1000 US, as are the post-column derivatization system and fluorescence detector. The system can be beyond the budget of smaller laboratories. Further, any method involving ion pairing agents has inherent problems, due to the complex chemistry and methodology and to variation among manufacturers' HPLC columns.

Now, Restek chemists have developed a simple, effective, reliable analysis for paraquat and diquat, based on a new HPLC column, Ultra Quat, and a unique mobile phase. The analysis can be performed on a conventional HPLC system with a conventional UV detector. In place of techniques that rely on the hydrophobicity of the column and the strength of the mobile phase, this separation makes use of a different analytical property—chaotropism: an ability to disrupt the structure of water and thereby alter the interactions among analyte, mobile phase, and stationary phase. In this case, the objective is to promote the solubility of the two highly polar analytes in a secondary substrate (the stationary phase). In other words, we bend the familiar chemical rule of "like dissolves like".

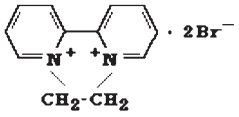
The packing for the new Ultra Quat column is based on a type B silica, to ensure proper selectivity and analyte retention, and to mini-

Figure 1

Chemical structures of paraquat and diquat.



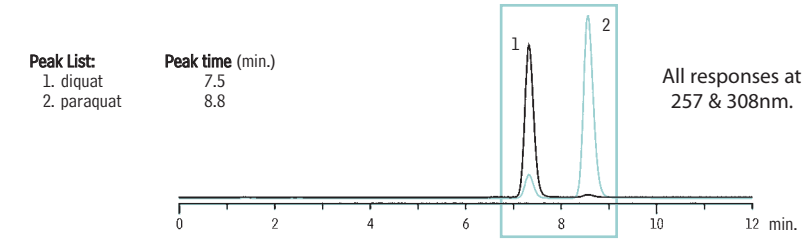
paraquat dichloride [CAS# 1910-42-5]

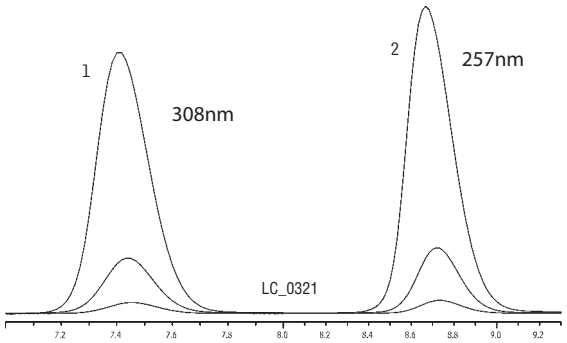


diquat dibromide [CAS # 85-00-7]

Figure 2

Consistent resolution, retention times, and peak symmetry for paraquat and diquat reference standards, using an Ultra Quat column.





Column: Ultra Quat
Cat. #: 9181565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Conditions:
Mobile phase: Ultra Quat Reagent Solution: acetonitrile, 95:5 (v/v)
Flow: 1.0 mL/min.
Temp.: 27°C
Det.: UV @ 257nm (paraquat)
UV @ 308nm (diquat)
Sample:
Inj.: 20µL
Conc.: 20ppm each component (above); 20µg/mL, 40µg/mL, and 100µg/mL each component (left)
Solvent: water

in this issue

mize residual silanols and metal ions on the packing particles, which could interact with the analytes and cause tailing and unwanted (and sometimes unpredictable) retention.

The reagent solution we use in the mobile phase, Ultra Quat Reagent Solution (cat.# 32441), alters the chemical nature of the analytes as perceived by the column and mobile phase. It reduces the ability of water to solvate the analytes and hydrogen bond with them, forcing the charged complexes into the stationary phase and improving retention.

Unlike ion pairing techniques, our new approach requires only water, Ultra Quat Reagent Solution, and acetonitrile (which cannot form hydrogen bonds) to accomplish the separation. For highest sensitivity, we monitor for paraquat at 257nm and for diquat at 308nm. Using the new column, mobile phase, and conditions, the detection limit for either herbicide is 6ppb in the final sample extract—a detectable amount of 0.12 nanograms on column. Data are summarized in Table 1. Using the solid phase extraction procedure in Table 2, which concentrates samples 200-fold (1L to 5mL), the detection limit is 0.03ppb—a significant improvement over current methodology. Analyte concentrations can be increased by modifying the solid phase extraction procedure or by increasing the injection volume, to improve quantification and detection limits.

Figure 2 overlays chromatograms of paraquat and diquat reference standards at a range of concentrations (20μg/mL–100μg/mL); resolution, retention times, and peak symmetry are highly consistent. Concentrations up to 100μg/mL are consistent with linear detector responses.

Note that glassware used to prepare and analyze samples and reference materials for this analysis must be deactivated (e.g., with dimethyldichlorosilane—DMDCS, cat.# 31840). EPA Method 549.2 requires retesting of all samples if the response for the reference standards changes by more than 20% over the time of the analysis. We found all reference standards showed degradation after only 1 hour in untreated glassware, with the lowest concentrations being the most affected. 30% losses in response were not uncommon; a diquat reference standard of 6ppb in water became undetectable.

When you perform the challenging paraquat/diquat analysis, our new Ultra Quat column, Ultra Quat Reagent Solution and Paraquat/Diquat Calibration Mix, and extraction procedure will give you the edge you need to obtain the most accurate and consistent information.

In Summary

Highly polar paraquat and diquat can't be separated on a reversed phase HPLC column without adding ion pair modifier to the mobile phase, but the ion pair reagent in current methodology does not provide optimum resolution and does not permit detection below 0.7μg/mL. We have developed a column and a mobile phase modifier for rapid, complete resolution of paraquat and diquat, with detection to concentrations as low as 0.5μg/mL—an improvement of 30%.

Table 1

Approximate detection/quantification limits for paraquat and diquat, using an Ultra Quat column.

On column limit of detection (LOD): 0.12ng
On column limit of quantification (LOQ): 1.2ng

Sample Volume (mL)	Injection Volume (μL)	Limit of Detection (ppb)	Limit of Quantification (ppb)
1	20	6	20
100	20	0.06	0.2
250	20	0.024	0.08
1000	20	0.006	0.02
1	100	1.2	4
100	100	0.012	0.04
250	100	0.0048	0.016
1000	100	0.0012	0.004
1	200	0.6	2
100	200	0.006	0.02
250	200	0.0024	0.008
1000	200	0.0006	0.002

Table 2

Solid phase extraction of diquat and paraquat from aqueous samples.

Sample Extraction

SPE Tubes:	Restek WCX, weak cation exchanger, 3mL/500mg, cat.# 26062.
Samples:	1 liter deionized water containing 50μg each of diquat and paraquat. Samples spiked with 20μL 549.2 Calibration Mix, cat.# 32437, diluted with HPLC grade water.
Conditioning:	3mL acetonitrile, then 3mL deionized water, applied sequentially. Do not allow adsorbent bed to dry before applying sample.
Extraction:	Pass 1 liter water samples through SPE tubes at a rate of 5-10mL/min. Arrange 5mL collection vessels under extraction tubes. Place 1mL acidic elution solution* in each tube, draw into bed, allow to stand for up to 1 min. Pass solution at a slow (drop-wise) rate through SPE tubes into collection vessels. Repeat with 2 x 2mL acidic elution solution. Correct final volume in collection vessels to 5mL with acidic elution solution.
Analysis:	Neutralize eluates with approximately 20μL concentrated ammonium hydroxide, then analyze by HPLC. Adjust amount of ammonium hydroxide used to assure each sample is neutral (test with pH indicating paper).

*1mL 85% H_3PO_4 diluted to 1 liter with deionized HPLC grade water (0.1%).

Results

Analyte	Recovery (%)	RSD (%)
diquat	99.0	0.89 (n=5)
paraquat	96.3	1.59 (n=5)

Extracted samples stored and analyzed in Silcote™ CL7 deactivated autosampler vials (cat.# 24671). Polypropylene vials and inserts (e.g., cat.# 24651) also may be used.

Ultra Quat HPLC Column

Physical Characteristics:
particle size: 5μm, spherical
pH range: 2.5 to 7.5
temperature limit: 80°C

5μm Column, 4.6mm ID	cat.#
150mm	9181565

Ultra Quat Guard Cartridges

	4.0mm ID
Length	cat.#
10mm	918150210
20mm	918150220

Ultra Quat Reagent Solution

Each	10-pk.
In water, 20mL/ampul	
32441	32541

Paraquat & Diquat Calibration Mix

diquat dibromide paraquat dichloride

Each
1,000μg/mL each in water, 1mL/ampul
32437
w/data pack
32437-500

Dimethyldichlorosilane (DMDCS)

Each	5-pk.
Neat, 20mL/ampul	
31840	31840-510

WCX Solid Phase Extraction Tubes

3mL/500mg, 50-pk., cat.# 26062,

GC/MS Analysis of Phthalate and Adipate Esters in Drinking Water

Using New Restek Reference Mixes and a Low-Bleed Column

by Katia May, Ph.D., Senior R&D Chemist, and Christopher English, Environmental Innovations Chemist

- New calibration and quality control check mixes save preparation time and effort.
- Stable baseline with Rtx®-5Sil MS column—no interference with sensitive detection.
- Rapid analysis, excellent resolution.

Phthalate esters are of considerable interest because their extensive use in consumer products, mainly as plasticizers, leads to widespread human exposure and potential for environmental contamination. In the United States, the Environmental Protection Agency (EPA) established strict drinking water standards for two of these semivolatile compounds, bis(2-ethylhexyl)phthalate and bis(2-ethylhexyl)adipate, as potential carcinogenic agents. Because even trace amounts of these esters can have a harmful effect on drinking water quality, the goal is to extract the compounds efficiently and identify them accurately. EPA Method 506 offers a procedure for extracting, identifying, and quantifying seven phthalate and adipate esters in drinking water, using liquid/liquid extraction (methylene chloride / hexane) or liquid/solid extraction (octadecyl (C18) disk, e.g., Restek cat.# 24004), extract concentration to 1mL, and analysis by gas chromatography/mass spectrometry.

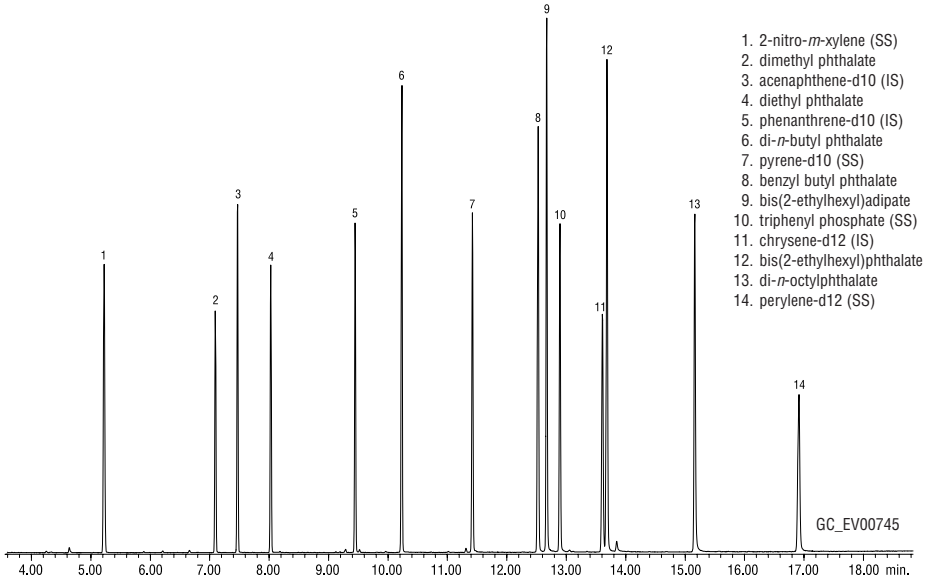
We have developed two new reference materials for analyses of the phthalate and adipate esters targeted by Method 506. We prepare 506 Calibration Mix in isooctane at 1000µg/mL, per method recommendation, and 506 Laboratory Performance Check Mix in purge-and-trap grade methanol at x10^5 the method detection limit (MDL) for each analyte.

In our quest for superior chromatography and improved detection limits for this and other analyses, we have developed a series of low bleed polymeric stationary phases, using Crossbond® bonding technology. By providing stable baselines at higher temperatures, these phases allow higher signal-to-noise ratios, and thus greater sensitivity.

EPA Method 506 suggests low levels of phthalate and adipate esters be evaluated using a photoionization detector. The method allows other approaches for detection, however, if equivalent performance can be demonstrated. Figure 1 shows a GC/MS analysis of the phthalates and adipates, using an Rtx®-5Sil MS column. Column bleed is low, even at the 310ºC oven temperature needed to elute the phthalate esters with the highest boiling points. At this temperature, column bleed from an unstable column could have a significant effect on detection limits. The 80ºC starting temperature and 18ºC/min. temperature program ensure a fast analysis, without inhibiting resolution. US EPA 525.2 internal standards and surrogates were used since the method does not list specific monitoring compounds.

In analyses for phthalate and adipate esters, a low-bleed Rtx®-5Sil MS column can extend detection limits and help ensure you of reliable data from your samples.

Figure 1 Rapid analysis of phthalates, with excellent resolution, using an Rtx®-5Sil MS column.



Rtx®-5Sil MS Column (fused silica)

(Selectivity equivalent to Crossbond® 5% diphenyl / 95% dimethyl polysiloxane) (temp. limits -60°C to 330°C)

30-Meter, 0.25mm ID, 0.25µm df
cat.# 12723

new!

Resprep™-C18 & Resprep™-C8 SPE Disks

- 47mm glass fiber disks embedded with C18 or C8 bonded silica.
- Extract semivolatile organic compounds.
- Deep-pore design reduces clogging and allows faster flow rates.
- Meet requirements for US EPA Methods 525.1, 506, 550.1, 549.1.
- Lower cost than Teflon® disks.

Description	qty.	cat.#
Resprep™-C18 47mm SPE Disks	20-pk.	24004
Resprep™-C8 47mm SPE Disks	24-pk.	24048

506 Laboratory Performance Check Mix

benzyl butyl phthalate	250µg/mL	di-*n*-octyl phthalate	650
bis(2-ethylhexyl)adipate	1200	diethyl phthalate	100
bis(2-ethylhexyl)phthalate	250	dimethyl phthalate	100
di-*n*-butyl phthalate	100		

Each	5-pk.	10-pk.
In P&T methanol, 1mL/ampul		
31844	31844-510	—
	w/data pack	
31844-500	31844-520	31944

506 Calibration Mix

benzyl butyl phthalate	di-*n*-octyl phthalate
bis(2-ethylhexyl)adipate	diethyl phthalate
bis(2-ethylhexyl)phthalate	dimethyl phthalate
di-*n*-butyl phthalate	

Each	5-pk.	10-pk.
1,000µg/mL each in isooctane, 1mL/ampul		
31845	31845-510	—
	w/data pack	
31845-500	31845-520	31945

Column:	Rtx®-5Sil MS, 30m, 0.25mm ID, 0.25µm (cat.# 12723)
Sample:	506 Calibration Mix, 1000µg/mL each analyte (cat.# 31845) Method 525.2 Internal Standard Mix (cat.# 31825) Method 525.2 Surrogate Standard Mix (cat.# 31826)
Inj.:	1.0µL, 20ppm each analyte using a 4mm splitless single gooseneck inlet liner (cat.# 20799) splitless hold time 0.40 min., 0.45 min. pressure pulse @ 50psi
GC:	Agilent 6890
Inj. temp.:	270°C
Carrier gas:	helium, constant flow
Flow rate:	1.0mL/min.
Oven temp.:	80°C (hold 0.5 min.) to 260°C @ 18°C/min., to 310°C @ 6°C/min. (hold 1 min.)
Det.:	Agilent 5973 GC/MS
Transfer line temp.:	280°C
Scan range:	35–550 amu
Solvent delay:	3 min.
Tune:	DFTPP

Fast Dual-Column Analysis of Pesticides or PAHs

Using an Rtx®-440 Capillary GC Column

new!

By Greg France, Innovations Chemist, and Gary Stidsen, Innovations Team Manager

- Analyze 20 organochlorine pesticides in less than 9 minutes.
- Analyze 16 PAHs in 22 minutes.
- New low-bleed, high-resolution column is ideal for dual-column analyses.

Assessments for organochlorine pesticides or polynuclear aromatic hydrocarbons (PAHs) are critical, frequently performed GC analyses—and they often are among the most challenging. Issues that can arise include analyte breakdown and poor linearity, and calibration times can be long. In addition to the problems inherent to the analysis, analysts must be concerned with column reactivity and bleed, which affect sensitivity and reproducibility. In analyses of PAHs, there are critical pairs to resolve and, because the samples often include interfering hydrocarbons, a confirmation column typically is required. Compounding these problems is constant pressure to process more samples in less time.

Figure 1 Separate 20 organochlorine pesticides in 9 minutes, using an Rtx®-440 column.

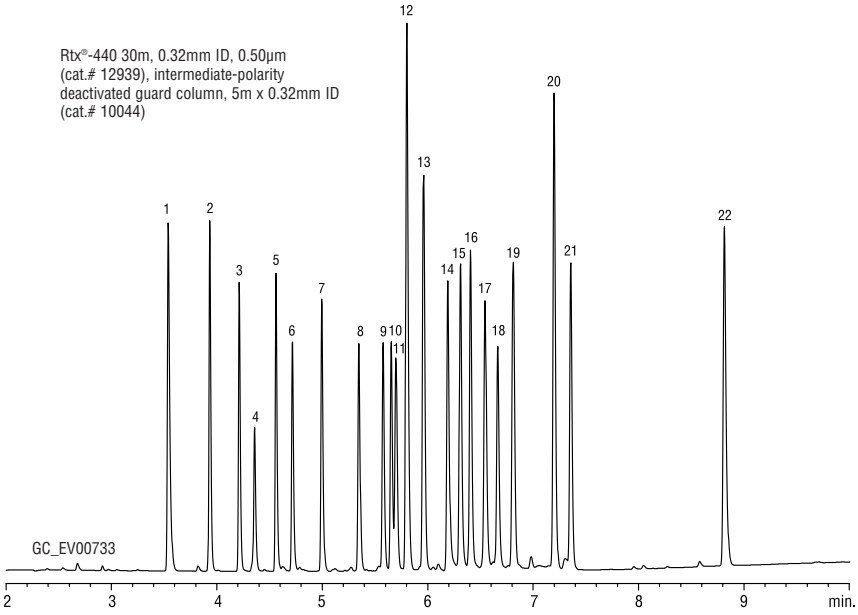
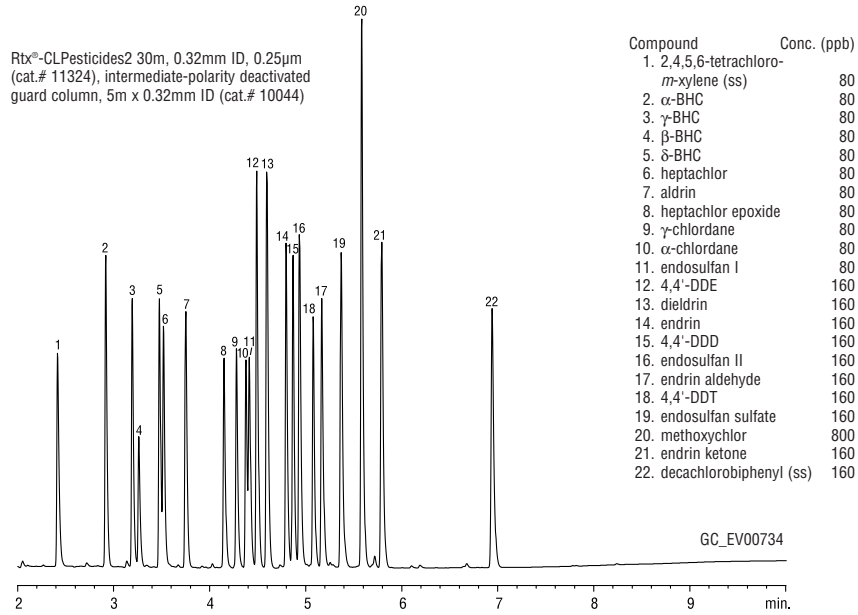


Figure 2 An Rtx®-CLPesticides2 column complements the Rtx®-440 column in dual-column analysis of organochlorine pesticides.



Compound	Conc. (ppb)
1. 2,4,5,6-tetrachloro-*m*-xylene (ss)	80
2. α-BHC	80
3. γ-BHC	80
4. β-BHC	80
5. δ-BHC	80
6. heptachlor	80
7. aldrin	80
8. heptachlor epoxide	80
9. γ-chlordane	80
10. α-chlordane	80
11. endosulfan I	80
12. 4,4'-DDE	160
13. dieldrin	160
14. endrin	160
15. 4,4'-DDD	160
16. endosulfan II	160
17. endrin aldehyde	160
18. 4,4'-DDT	160
19. endosulfan sulfate	160
20. methoxychlor	800
21. endrin ketone	160
22. decachlorobiphenyl (ss)	160

With the new Rtx®-440 column, Restek makes available an excellent choice for both of these demanding applications.

Organochlorine Pesticides: Sub-10-Minute Analyses

Figure 1 shows a separation of 20 commonly analyzed organochlorine pesticides, obtained in less than 10 minutes by using an Rtx®-440 column. Only α-chlordane and endosulfan I (peaks 10 & 11) are not separated to the baseline. The column's excellent thermal stability is indicated by a virtually flat baseline between the initial temperature and the maximum temperature of the program, 330ºC. In a dual-column approach to this application, an Rtx®-440 column can be paired with an Rtx®-CLPesticides2 column. The latter column will provide an equally fast separation (Figure 2) and near-equivalent resolution, with a reverse in elution order for endrin aldehyde and 4,4'-DDT (peaks 17 & 18). By connecting the two columns to a "Y" connector and making the sample injection onto a 5-meter intermediate-polarity deactivated guard column, the two analyses can be conducted simultaneously.

Polynuclear Aromatic Hydrocarbons: Baseline Resolution of Critical Pairs

In Figure 3, 16 commonly encountered PAHs have eluted from an Rtx®-440 column in less than 18 minutes. Two critical pairs, phenanthrene/anthracene (peaks 5 and 6) and benzo(a)anthracene/chrysene (peaks 9 and 10), are resolved to baseline, and benzo(b)fluoranthene and benzo(k)fluoranthene (peaks 11 and 12) and indeno(1,2,3-cd)pyrene and dibenzo(a,h)anthracene (peaks 14 and 15) are almost completely separated. Also notice the excellent thermal stability—baseline rise is negligible even at 320ºC. Similar results can be obtained by using an Rtx®-5Sil MS column or an Rtx®-CLPesticides2 column and constant flow, as shown in the Applications section of our general catalog. An Rtx®-440 column can be paired with either of these other columns, for a rapid, dual-column/FID analysis of commonly encountered PAHs.

Conditions for Figures 1 and 2

Sample:	Organochlorine Pesticide Mix AB #2 (cat.# 32292), 2,4,5,6-tetrachloro-*m*-xylene (ss) (cat.# 32027), decachlorobiphenyl (ss) (cat.# 32029), diluted in hexane, on-column amounts listed on figure
Inj.:	1.0µL splitless (hold 0.75 min.), 4mm Drilled Uniliner® inlet liner (cat.# 21055)
Inj. temp.:	225°C
Carrier gas:	hydrogen, constant pressure
Linear velocity:	73cm/sec. (Rtx®-440) or 77cm/sec. @ 140°C (Rtx®-CLPesticides2)
Oven temp.:	140°C (hold 0.5 min.) to 268°C @ 30°C/min., to 290°C @ 11°C/min., to 330°C @ 25°C/min. (hold 5 min.)
Det.:	ECD @ 320°C

Conclusion

The new Rtx®-440 column is an excellent addition to the selection of innovative columns available from Restek. The column exhibits high thermal stability and, for organochlorine pesticides, a selectivity alternative to the Rtx®-CLPesticides2 column. An Rtx®-440 column can be paired with an Rtx®-CLPesticides2 column to ensure sub-10-minute analysis times for organochlorine pesticides, or can be used as a confirmation column, with an Rtx®-5Sil MS or an Rtx®-CLPesticides2 column, for GC/FID analysis of PAHs.

Organochlorine Pesticide Mix AB #2

	8µg/mL		
aldrin	8	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

Each	5-pk.	10-pk.
In hexane:toluene (1:1), 1mL/ampul		
32292	32292-510	—
	w/data pack	
32292-500	32292-520	32392

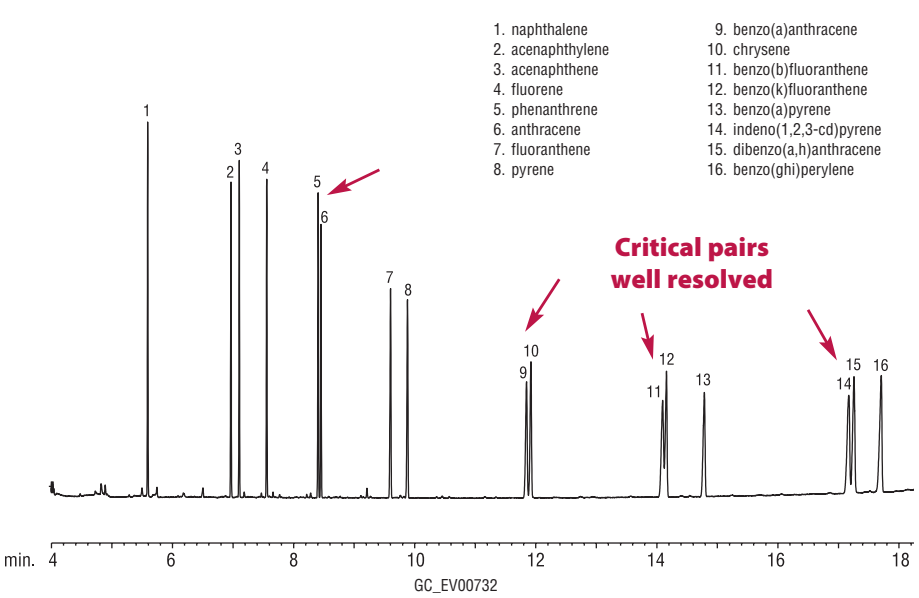
2,4,5,6-Tetrachloro-*m*-xylene

Each	5-pk.	10-pk.
200µg/mL in acetone, 1mL/ampul		
32027	32027-510	—
	w/data pack	
32027-500	32027-520	32127
200µg/mL in acetone, 5mL/ampul		
32028	32028-510	—
	w/data pack	
32028-500	32028-520	32128

Decachlorobiphenyl (BZ #209)

Each	5-pk.	10-pk.
10µg/mL in isooctane, 1L/ampul		
32289	32289-510	—
	w/data pack	
32289-500	32289-520	32389
200µg/mL in acetone, 1mL/ampul		
32029	32029-510	—
	w/data pack	
32029-500	32029-520	32129
200µg/mL in acetone, 5mL/ampul		
32030	32030-510	—
	w/data pack	
32030-500	32030-520	32130

Figure 3 Analyze 16 PAHs in 22 minutes, and resolve critical pairs, with an Rtx®-440 column.



Column:	Rtx®-440 30m, 0.25mm ID, 0.25µm (cat.# 12923)
Sample:	610 PAH Mix (cat.# 31011) diluted to 20ppm each compound in methylene chloride
Inj.:	1.0µL 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

Searching for a chromatogram ? www.restek.com

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

Each	5-pk.	10-pk.
2,000µg/mL each in methylene chloride, 1mL/ampul		
31011	31011-510	—
	w/data pack	
31011-500	31011-520	31111

SeCure™ "Y" Connectors

- Use standard "Y" Press-Tight® connectors and 1/16" graphite ferrules.
- Reliable seal integrity, will not unexpectedly disconnect during temperature-programmed analyses.
- Open design allows visual confirmation of the seal for added confidence in the connection.

Restek Innovation!

Kits include: SeCure™ "Y" connector body, 3 knurled nuts, 1 "Y" Universal Press-Tight® union, 3 ferrules

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

Rtx®-440 Columns (fused silica)

ID	df (µm)	temp. limits	30-Meter
0.25mm	0.25	20°C to 320/340°C	12923
	0.50	20°C to 320/340°C	12938
0.32mm	0.25	20°C to 320/340°C	12924
	0.50	20°C to 320/340°C	12939
0.53mm	0.50	20°C to 320/340°C	12940
	1.00	20°C to 320/340°C	12955

Rtx®-CLPesticides2 Columns (fused silica)

ID	df (µm)	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter	60-Meter
0.10mm	0.10	-60 to 310/330°C	43301		43302		
0.18mm	0.14	-60 to 310/330°C	42301		42302		
0.25mm	0.20	-60 to 320/340°C		11320		11323	11326
0.32mm	0.25	-60 to 320/340°C		11321		11324	
0.53mm	0.42	-60 to 300/320°C		11337		11340	

Rapid Analysis of Residual Solvents in Pharmaceuticals

Restek Innovation!

Using Static Headspace Sampling and Stop-Flow GC

by Christopher English, Environmental Innovations Chemist, Rebecca Wittrig, Ph.D., HPLC Product Marketing Manager, and Frank Dorman, Ph.D., Director of Technical Development

- Resolve 35 residual solvents in 18 minutes.
- Simplify inventory—use one pair of chromatography columns and one set of conditions for all ICH Class I and Class II solvents.
- Complete, easy to install system.

Kit is easily attached to Agilent 6890 GC!

The International Conference on Harmonization (ICH) makes recommendations concerning amounts of residual solvents considered safe in pharmaceutical finished goods. The ICH has published guidelines and daily exposure limits for 61 solvents, classified in three groups, according to their toxicity. Class I solvents are known carcinogens or environmental hazards, to be avoided if at all possible. Class II solvents are less toxic, but their use should be limited. Class III solvents have low toxicity or no health-related exposure limit.[1] All pharmaceutical products must be analyzed for residual solvents, regardless of the matrix, and an enormous number of methods potentially can be required to address the total list of solvents. The complexity and high cost of compliance are major hurdles in drug manufacture.

In February 2004, Teledyne Tekmar developed a universal analytical method for extracting and determining 32 ICH Class II and Class III residual solvents, using static headspace sampling.[2] Simultaneously, Restek chemists were developing an approach for resolving the Class I and Class II solvents, using a new technology known as Stop-Flow GC, but lacked a sample preparation method suitable for achieving the detection limits required by the ICH.[3] By using a Teledyne Tekmar 7000HT headspace autosampler unit in conjunction with Stop-Flow GC technology, it is possible to achieve resolution, sensitivity, and rapid sample turn-around times for the Class I and Class II residual solvents. In Stop-Flow GC the solvents are separated by passing the sample through a two-column ensemble consisting of a Stabilwax® column and an Rtx®-200 column coupled in series. Carrier gas flow through the second (Rtx®-200) column is interrupted briefly (stop-flow pulses) to tune the separation at the outlet of the column ensemble.

In an analysis on two GC columns in series there are four possible outcomes for two sample components: 1) the two compounds are resolved at the column junction and remain resolved at the end of the ensemble; 2) the two compounds coelute at the junction, but are resolved on the second column; 3) the two compounds are resolved at the junction, but coelute at the end of the column ensemble; 4) the two compounds coelute at the column junction and at the end of the ensemble. For 1) and 2) no adjustment is necessary. For 4) other stationary phase combinations should be investigated to ensure separation on at least one of the two columns. For 3) Stop-Flow GC is appropriate. Carrier gas flow into the second column is interrupted briefly, immediately after one of the two compounds has crossed the junction, but while the other compound is still in the first column. The timing and duration of the stop-flow pulse are set to ensure that the two components remain separated when they reach the end of the column ensemble. The key to choosing a column ensemble for a specific application is to make separate analyses on each column, to ensure that no two compounds coelute on both stationary phases.

Figure 1 is the product of applying three stop-flow pulses at the junction point of the column ensemble, to pull apart three analytes: trichloroethene, acetonitrile, and chloroform. The other analytes are resolved by adjusting the carrier gas flow and temperature program, and do not require pulses. The chromatogram includes all ICH Class I and Class II solvents, except ethylene glycol (which was not detected at 200ppm), at 200ppm each in 5mL of 1,3-dimethyl-2-imidazolidinone (DMI) solvent. By resolving closely eluting component pairs, Stop-Flow GC enables pharmaceutical laboratories to monitor all ICH Class I and Class II solvents with one pair of chromatography columns and a single set of conditions.

This analysis for 35 residual Class I and Class II solvents is rapid, sensitive, and reliable. If you are required to monitor solvents in pharmaceutical products, we welcome the opportunity to discuss Stop-Flow GC with you.

References

1. *ICH Guidance for Industry, Q3A Impurities: Residual Solvents* US Dept. of Health and Human Services, Food and Drug Administration, Center for Drug Evaluation and Research, Center for Biologics Evaluation and Research (CBER). International Conference on Harmonization, Dec. 1997.
2. Wallace, B. and J. Kancler. *One Universal Method for Residual Solvents in Pharmaceuticals Using a High Temperature Static Headspace Sample Introduction System* Application Note 7000-021b.doc, Teledyne Tekmar Instruments, Feb. 2004.
3. Wittrig, R.E.; F.L. Dorman, C.M. English, R.D. Sachs, *J.Chromatogr.* A 1027: 75-82 (2004).

Acknowledgement

Special thanks to Brian Wallace of Teledyne Tekmar for the use of the 7000HT headspace autosampler.

Stop-Flow GC for Agilent 6890 GCs

Description	qty.	cat.#
Stop-Flow System for use with Cool On-Column EPC (includes: Stop-Flow enclosure, top mounting plate, 1-line weldment, and interface cable)	kit	21168
Stop-Flow System for use with Split/Splitless EPC (includes: Stop-Flow enclosure, top mounting plate, 2-line weldment, and interface cable)	kit	21169

Stabilwax® Column

15-Meter, 0.25mm, ID 0.5μm df, cat.# 10635

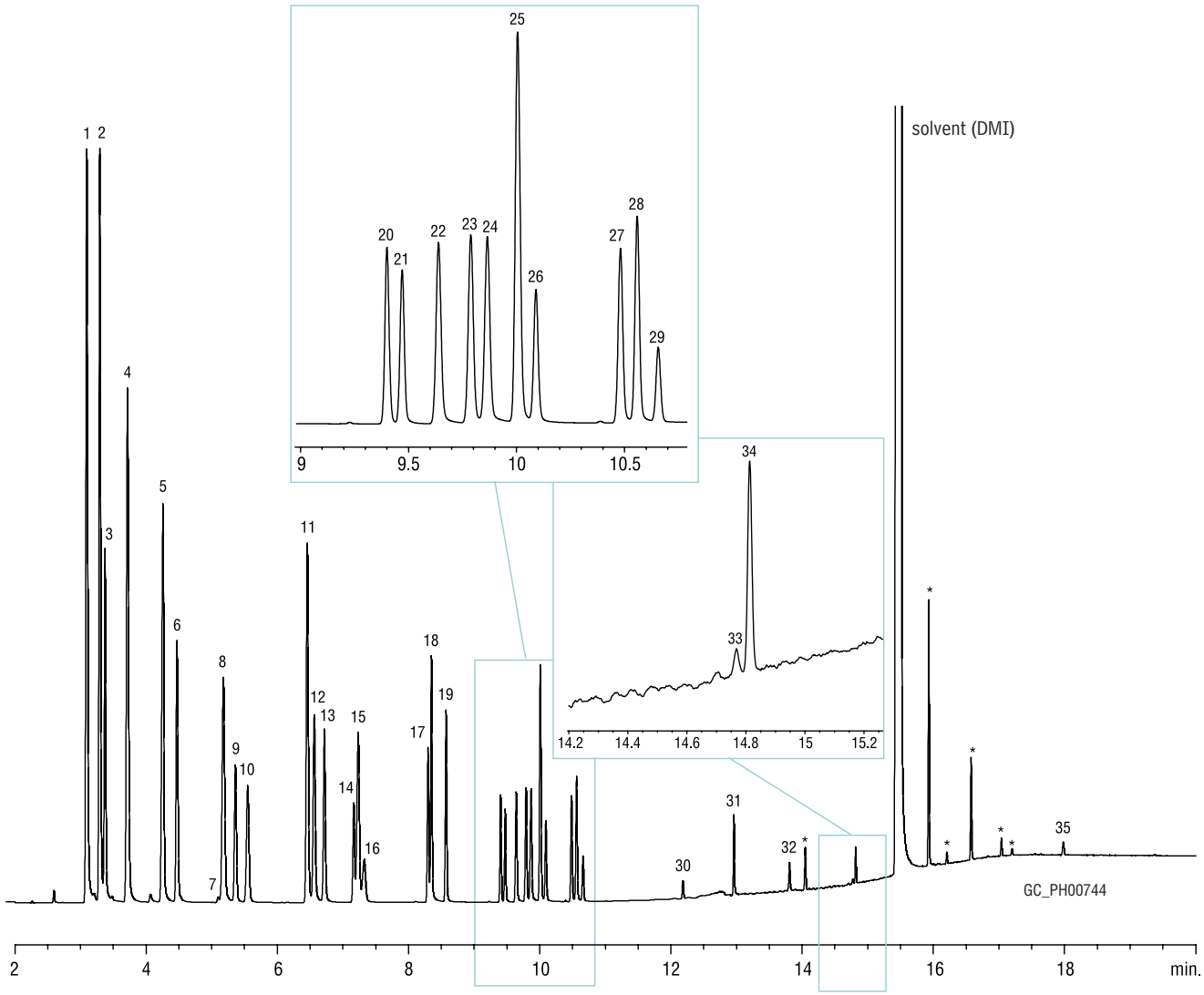
Rtx®-200 Column

30-Meter, 0.25mm ID, 1.0μm df, cat.# 15053

Did you know?

We offer many reference mixes of residual solvents for EP and USP methods.
For descriptions, please refer to our chromatography supplies catalog, or visit our website.

Figure 1 Stop-Flow GC provides a rapid, sensitive analysis for ICH Class I and Class II residual solvents.



1. 2-methylpentane
2. hexane
3. 1,1-dichloroethene
4. methyl cyclopentane
5. methanol
6. *trans*-1,2-dichloroethene
7. carbon tetrachloride
8. methyl cyclohexane
9. methylene chloride
10. 1,1,1-trichloroethane
11. benzene
12. 1,2-dimethoxyethane
13. *cis*-1,2-dichloroethene
14. trichloroethene
15. acetonitrile
16. chloroform
17. 1,2-dichloroethane
18. toluene
19. 1,4-dioxane
20. nitromethane
21. 2-methoxyethanol
22. 2-hexanone (MBK)
23. *p*-xylene
24. *m*-xylene
25. pyridine
26. 2-ethoxyethanol
27. *o*-xylene
28. chlorobenzene
29. 1,1,2-trichloroethane
30. dimethyl formamide (DMF)
31. N,N-dimethylacetamide
32. 1,2,3,4-tetrahydronaphthalene
33. formamide
34. 1-methyl-2-pyrrolidinone
35. sulfolane
* impurities in solvent

Headspace Conditions

Instrument:	Teledyne Tekmar 7000HT high temperature static headspace unit
Platen temp.:	140°C
Sample equilibration:	5 min.
Mixing time:	10 min.
Mixing power:	2
Mixture stabilization:	1 min.
Pressure time:	0.2 min.
Pressure equilibration:	0.3 min.
Vial vol.:	22mL (high temperature vials)
Sample loop vol.:	1mL (standard size, Silcosteel® treated)
Loop/line temp.:	250°C
Loop fill time:	0.1 min.
Loop equilibration:	0.05 min.
Inj. time:	1.0 min.
Static vial press.:	3.5psi helium
Vial press.:	8psi helium
Variable inj. press. (VIPR):	5psi helium
Interface:	plumbed through injection port, 1:20 split

GC Conditions

Column #1:	Stabilwax®, 15m x 0.25mm x 0.5µm (cat. # 10635)
Column #2:	Rtx®-200, 30m x 0.25mm x 1.0µm (cat. # 15053)
Sample:	200ppm each component in 1,3-dimethyl-2-imidazolidinone (DMI)
Instrument:	Agilent 6890
Inj. port temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1.9mL/min., 25.6psi @ 40°C
Oven temp.:	40°C (hold 2 min.) to 55°C @ 4°C/min., to 110°C @ 25°C/min. (hold 2 min.) to 250°C @ 25°C/min. (hold 5 min.)
Det.:	FID #1 at column junction, FID #2 at sample outlet (equal settings)
Det. temp.:	250°C
Reaction gas:	hydrogen, 40mL/min.
Air flow:	400mL/min.
Makeup:	helium, 40mL/min.
Data collection rate:	100Hz

Stop-Flow Conditions

Instrument:	Restek Stop-Flow System for Agilent 6890 GC with cool on-column EPC (cat. #21168)
Inj. port connection:	cool on-column injector
Pressure:	31.0psi, constant pressure
Pulses:	valve opened 3.00 - 3.15 min., 4.65 - 5.02 min., 5.10 - 5.40 min.
Total analysis time:	20.55 min.

Improving Detailed Hydrocarbon Analysis

Using an Rtx®-1PONA Capillary GC Column

by Barry Burger, Petroleum Applications Chemist,
and Neil Mosesman, GC Columns Product Marketing Manager

- Column meets or exceeds all ASTM D-6730-01 and Canadian General Standards Board method requirements.
- 30% faster analysis (C13 retention = 97 minutes), using helium.
- Excellent responses and peak symmetry for polar oxygenates.
- Guaranteed column-to-column reproducibility for retention, efficiency, selectivity, peak skewness, resolution, low bleed.

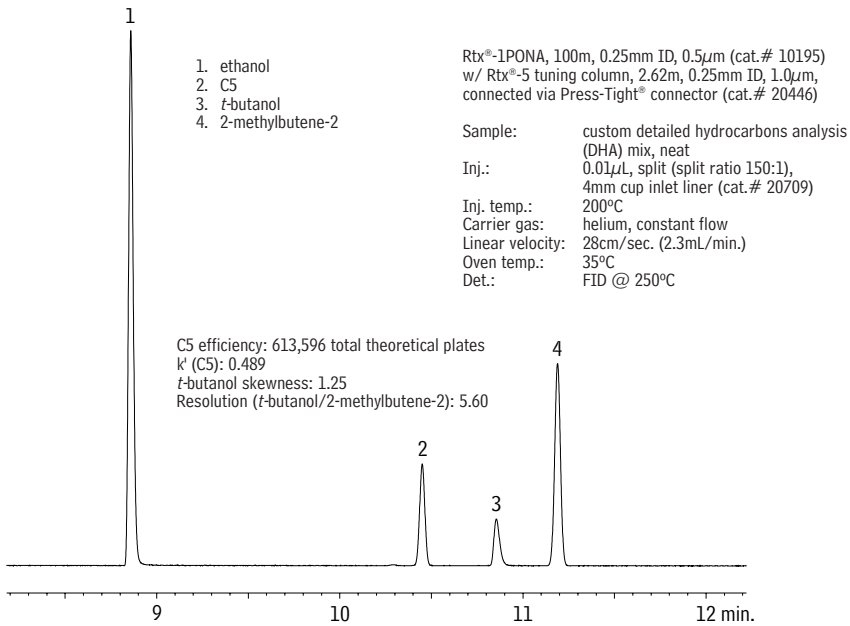
Gasolines are complex mixtures of hundreds of compounds. Information about concentrations of the individual components is important for evaluating raw materials and controlling refinery processes. A high-resolution GC method for detailed hydrocarbon analysis (DHA) of gasolines is outlined in American Society of Testing and Materials (ASTM) Method D-6730-01—often referred to as the PONA (paraffins, olefins, naphthenes, aromatics) or PIANO (paraffins, isoparaffins, aromatics, naphthenes, olefins) analysis.* ASTM D-6730-01 is specific for the analysis of these hydrocarbon components, plus oxygenated additives such as methanol, ethanol, *tert*-butanol, methyl *tert*-butyl ether (MTBE), and *tert*-amyl methyl ether (TAME) in spark-ignition engine fuels.

To maximize resolution of these complex mixtures, the ASTM method recommends a 100-meter x 0.25mm ID capillary column coated with 0.5µm of 100% dimethyl polysiloxane stationary phase, and sets minimum resolution criteria for several critical pairs of closely eluting compounds. To retain the aromatics, and accomplish the separations, a short tuning column, approximately 2-3 meters long, coated with 5% diphenyl/95% dimethyl polysiloxane polymer, is connected to the inlet of the 100-meter analytical column. Through a series of trial analyses, the length of the tuning column is adjusted to ensure the critical resolutions are achieved.

Analytical columns used for this application must exhibit high efficiency and exceptional inertness, especially for polar oxygenates in gasoline. Figure 1 illustrates a column efficiency of 613,596 total theoretical plates, measured on C5, and shows excellent peak symmetry for the oxygenated additives, including ethanol and *t*-butanol (*t*-butanol skewness = 1.25). We test every Rtx®-1PONA column for retention (k), efficiency (n), stationary phase selectivity (RI), and bleed, and guarantee reproducible column-to-column performance.

An Rtx®-1PONA column meets all ASTM D-6730-01 requirements for critical pair resolution, as demonstrated by Figure 2. A 2.6-meter tuning column was used to achieve the highlighted resolutions, based on retention of the aromatics (e.g., resolution for 1-methylcyclopentene / benzene = 1.28.).

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



*In alternate terminology: paraffins & isoparaffins = alkanes; naphthenes = cyclic alkanes; olefins = alkenes.

In addition to qualifying for the ASTM D-6730-01 analysis, Rtx®-1PONA columns meet the similarly stringent requirements of Canadian General Standards Board (CGSB) methodology. For additional detailed hydrocarbon analysis chromatograms and more information about these high-performance columns, please request a free copy of Applications Note 59568, or review the applications note and chromatography on our website.

Rtx®-1 PONA Column (fused silica)

(Crossbond® 100% dimethyl polysiloxane phase optimized for hydrocarbon analysis) (temp. limits.: -60 to 300/340°C)
100m, 0.25mm ID, 0.50µm df, cat.# 10195,

Rtx®-5 PONA Tuning Column

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane phase)
5m, 0.25mm ID, 1.0µm df, cat.# 554206,

Press-Tight® Connectors

- Made from inert fused silica.
- Fit column ODs from 0.33–0.74mm (Restek 0.1mm–0.53mm ID).
- Angled connector reduces strain on connection.

Description	5-pk.
Universal Press-Tight® Connectors	20400
Siltek™-treated Universal Press-Tight® Connectors	20480
Universal Angled Press-Tight® Connectors	20446
Siltek™-treated Universal Angled Press-Tight® Connectors	20482

Vu2 Union™ Connector

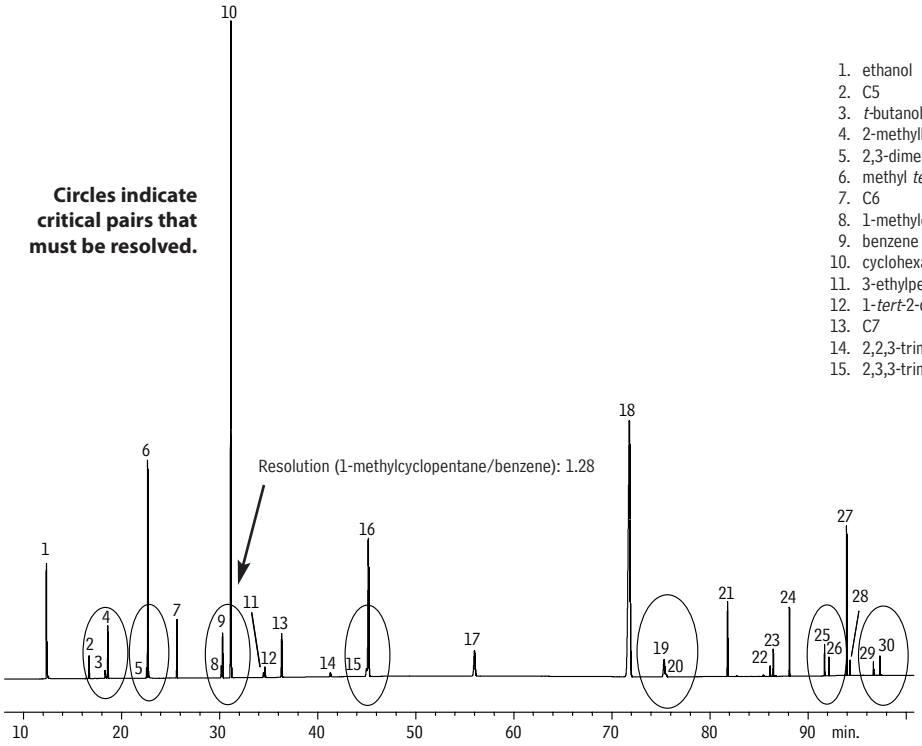
A Vu2 Union™ connector combines the simplicity of a Press-Tight® union with the strength of a metal union. The columns cannot unexpectedly disconnect, even at temperatures as high as 400°C.

Secure, reliable column-to-column connections!

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

Connector Kit (Ferrules Fit Restek Column ID)	cat.#
Vu2 Union™ Connector Kit (0.15–0.25mm)	21105
Vu2 Union™ Connector Kit (0.28/0.32mm)	21106
Vu2 Union™ Connector Kit (0.45/0.50 & 0.53mm)	21107

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



1. ethanol
2. C5
3. *t*-butanol
4. 2-methylbutene-2
5. 2,3-dimethylbutane
6. methyl *tert*-butyl ether (MTBE)
7. C6
8. 1-methylcyclopentene
9. benzene
10. cyclohexane
11. 3-ethylpentane
12. 1-*tert*-2-dimethylcyclopentane
13. C7
14. 2,2,3-trimethylpentane
15. 2,3,3-trimethylpentane
16. toluene
17. C8
18. ethylbenzene
19. *p*-xylene
20. 2,3-dimethylheptane
21. C9
22. 5-methylnonane
23. 1,2-methylethylbenzene
24. C10
25. C11 (undecane)
26. 1,2,3,5-tetramethylbenzene
27. naphthalene
28. C12 (dodecane)
29. 1-methylnaphthalene
30. C13 (tridecane)

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 hydrocarbons analysis (DHA) mix, neat
Inj.:	0.01µL, split (split ratio 150:1), 4mm cup inlet liner (cat.# 20709)
Inj. temp.:	200ºC
Carrier gas:	helium, constant flow
Linear velocity:	28cm/sec. (2.3mL/min.)
Oven temp.:	5ºC (hold 15 min.) to 50ºC @ 5ºC/min. (hold 50 min.) to 200ºC @ 8ºC/min. (hold 10 min.)
Det.:	FID @ 250ºC

Restek on the Road

Autumn Seminars in Europe and the US

by Rick Parmely, Technical Training & Education Manager

Our GC and HPLC seminars are well known and well received across the USA and around the world. Our autumn seminars include those listed here. For details about these seminars, please contact the Restek representative listed. For the most current schedule of US seminars, please visit our website (www.restek.com/seminar). If you're interested in a seminar in your city, or country, please call and talk with us.

US Seminars

Comprehensive Capillary GC

Date	Location
Sept. 13	Boulder, CO
Sept. 13	Blue Ash, OH
Sept. 15	Columbus, OH
Sept. 16	Salt Lake City, UT
Sept. 17	Buffalo, NY
Oct. 14	RTP, NC
Oct. 21	Buena Park, CA

GC/MS

Nov. 1	Pleasanton, CA
Nov. 3	Seattle, WA

Comprehensive HPLC

Oct. 5	Rockville, MD
Oct. 6	Princeton, NJ
Oct. 8	Plymouth Meeting/ King of Prussia, PA
Oct. 18	La Jolla/San Diego, CA

HPLC Method Development (2 days)

Sept. 23/24	Indianapolis, IN
Oct. 21/22	Foster City, CA

International Seminars

GC/MS Seminar

Date	Location	Contact
September 14	Lisbon, Portugal	Dias De Sousa (phone: +351 21 953 31 20 or e-mail: DS@dias-de-sousa.pt)
September 15	Barcelona, Spain	Teknokroma (phone: +34 936 748 800 or e-mail: comercial@teknokroma.es)
September 17	Milan, Italy	CPS Analytics (phone: +39 (0)2 8954201 or e-mail: cps@cps.it)
September 20	Stockholm, Sweden	Coricon AB (phone: +46 18 34 90 34 or email: info@coricon.se)
September 21	Oslo, Norway	Instrument Teknikk (phone: (+47) 67 16 41 00 or e-mail: firmapost@instrument-teknikk.no)
October 11	Cork, Ireland	Restek Ireland (phone: +44-28-90-814576 or e-mail: restekeurope@aol.com)
October 12	Dublin, Ireland	Restek Ireland (phone: +44-28-90-814576 or e-mail: restekeurope@aol.com)
October 14	Cork, Ireland	Restek Ireland (phone: +44-28-90-814576 or e-mail: restekeurope@aol.com)
October 20	Moscow, Russia	Laverna Group (phone: +7-095-482-2001 or e-mail: rodchenkov@lab.comcor.ru)

Comprehensive GC Seminar

Date	Location	Contact
October 18	Budapest, Hungary	Lab Comp (phone: +36 1 347 6090 or e-mail: labcomp@lab-comp.hu)

Convenient Calibration, Faster GC/MS Analysis for Volatile Organics in Water

Using a New Restek Calibration Mix and an Rtx®-VMS Column

by Katia May, Ph.D., Senior R&D Chemist, and Jack Crissman, Ph.D., Analytical Reference Materials Marketing Manager

- 60-component MegaMix™ includes six target gases—eliminates mixing errors.
- 0.18mm Rtx®-VMS column offers fast cycles, excellent resolution of gases.
- Monitor drinking water - wastewater - hazardous waste.

Volatile organic analytes (VOAs) are a common source of environmental pollution, and are among the most difficult and expensive contaminants to monitor in water. Analysis and quantification of VOAs in drinking water are detailed in US EPA methods 502 and 524, and in many other methods worldwide.

Until now, Restek has offered two complex calibration mixes of volatile compounds for drinking water analysis: a mix containing 54 target compounds (502.2 MegaMix™, cat.# 30432), and one containing 73 compounds (Drinking Water VOA MegaMix™ 524.2 Rev. 4.2, cat.# 30601). The only target compounds in the EPA methods that we do not include in these mixes are the highly volatile gases, and, for Method 524.2, the reactive ketones. To prevent acetal formation, we offer the five ketones as a separate mix (cat.# 30602). We also offer the six gases separately, as 502.2 Calibration Mix #1 (cat.# 30042 or cat.# 30439). Analysts monitoring samples for the gases combine the gases mix with either the 54-component mix or the 73-component mix—this takes time and can introduce variation or mixing errors.

For the convenience of our customers, we have developed a new 60-component calibration mix (Volatiles MegaMix™ with Gases, cat.# 30603) that contains the 54 target compounds in 502.2 MegaMix™ mix, plus the six gases in 502.2 Calibration Mix #1, at 200 ppm each in purge and trap methanol. The new mix is suitable for Method 502, Method 524, or other methods followed in monitoring these compounds.
The new mix brings a choice. The 60-component mix is very convenient to use, and eliminates both variation and the potential for errors (associated with measuring and mixing from multiple ampuls). An unopened ampul of this mix has a 24-month shelf life, but once the ampul is opened, the gases can begin to escape from the solution, and opened ampuls of the new mix should be replaced more frequently than ampuls of the 54-component mix. (This also is true of the 6-component gas mix.) Analysts choosing to work with the 54-component mix and the six gases mix must contend with the potential for mixing errors, but can see longer lifetimes from ampuls of the opened 54-component mix—if they are stored properly. We recommend storing all VOAs reference mixes in a freezer, especially those containing the gases.

Chemists monitoring VOAs in water require fast and accurate analyses. A chromatography column with a cyanopropylphenyl/dimethyl polysiloxane stationary phase (e.g., a "624" column) or a diphenyl/dimethyl polysiloxane phase (e.g., a "502.2" column) can provide a fast analysis, but some compounds are likely to coelute, creating quantification problems. In contrast, Rtx®-VMS columns are designed specifically for analyses of volatiles by GC/MS, and circumvent such problems. Analysis on a 20m, 0.18mm ID, 1.0µm Rtx®-VMS column (cat.# 49914), using a 45ºC initial oven temperature, will provide good resolution of the early eluting gases and ensure faster oven cycles. Under optimized analytical conditions and using a dual purge and trap system, as shown in Reference 1, Figure 47, the narrow bore column can reduce the analysis time to approximately 10 minutes, without sacrificing resolution.

If you are testing for volatiles in drinking water, wastewater, or hazardous waste, an Rtx®-VMS column and our new 60-component volatiles MegaMix™ with gases will help you meet the requirements for most analytical methods.

Reference

1. *Optimizing the Analysis of Volatile Organic Compounds* Restek technical guide, lit. cat.# 59887A, free on request. Also available on our website.

Rtx®-VMS Columns (fused silica)

(temp. limits -40°C to 240/260°C)
0.18mm ID, 1.00µm df
20-Meter, cat.# 49914,
40-Meter, cat.# 49915,

Volatiles MegaMix™ with Gases (60 Components)

benzene
bromobenzene
bromochloromethane
bromodichloromethane
bromoform
bromomethane (methyl bromide)
n-butylbenzene
sec-butylbenzene
tert-butylbenzene
carbon tetrachloride
chlorobenzene
chloroethane (ethyl chloride)
chloroform
chloromethane (methyl chloride)
2-chlorotoluene
4-chlorotoluene
dibromochloromethane
1,2-dibromo-3-chloropropane
1,2-dibromoethane (EDB)
dibromomethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
dichlorodifluoromethane (CFC-12)
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethylene
cis-1,2-dichloroethylene
trans-1,2-dichloroethylene
1,2-dichloropropane
1,3-dichloropropane
2,2-dichloropropane
1,1-dichloropropene
trans-1,3-dichloropropene
cis-1,3-dichloropropylene
ethylbenzene
hexachloro-1,3-butadiene (hexachlorobutadiene)
isopropylbenzene (cumene)
4-isopropyltoluene (*p*-cymene)
methylene chloride (dichloromethane)
naphthalene
n-propylbenzene
styrene
toluene
1,1,1,2-tetrachloroethane
1,1,2,2-tetrachloroethane
tetrachloroethylene
1,2,4-trichlorobenzene
1,2,3-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethylene
trichlorofluoromethane (CFC-11)
1,2,3-trichloropropane
1,3,5-trimethylbenzene
1,2,4-trimethylbenzene
vinyl chloride
m-xylene
o-xylene
p-xylene

Each	5-pk.	10-pk.
200µg/mL each in P&T methanol, 1mL/ampul		
30603	30603-510	—
	w/data pack	
30603-500	30603-520	30703

502.2 MegaMix™ (54 Components)

benzene
bromobenzene
bromochloromethane
bromodichloromethane
bromoform
n-butylbenzene
sec-butylbenzene
tert-butylbenzene
carbon tetrachloride
chlorobenzene
chloroform
2-chlorotoluene
4-chlorotoluene
dibromochloromethane
1,2-dibromo-3-chloropropane
1,2-dibromoethane
dibromomethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
cis-1,2-dichloroethene
trans-1,2-dichloroethene
1,2-dichloropropane
1,3-dichloropropane
2,2-dichloropropane
1,1-dichloropropene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
ethylbenzene
hexachlorobutadiene
isopropylbenzene
p-isopropyltoluene
methylene chloride
naphthalene
n-propylbenzene
styrene
1,1,1,2-tetrachloroethane
1,1,2,2-tetrachloroethane
tetrachloroethene
toluene
1,2,3-trichlorobenzene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethene
1,2,3-trichloropropane
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
m-xylene
o-xylene
p-xylene

Each	5-pk.	10-pk.
200µg/mL each in P&T methanol, 1mL/ampul		
30432	30432-510	—
	w/data pack	
30432-500	30432-520	30532
2,000µg/mL each in P&T methanol, 1mL/ampul		
30431	30431-510	—
	w/data pack	
30431-500	30431-520	30531

502.2 Calibration Mix #1 (gases)

bromomethane
chloroethane
chloromethane
dichlorodifluoromethane
trichlorofluoromethane
vinyl chloride

Each	5-pk.	10-pk.
200µg/mL each in P&T methanol, 1mL/ampul		
30439	30439-510	—
	w/data pack	
30439-500	30439-520	30539
2,000µg/mL each in P&T methanol, 1mL/ampul		
30042	30042-510	—
	w/data pack	
30042-500	30042-520	30142

Additional Reference Materials for GC/MS Analysis of Volatile Organics in Water

Drinking Water VOA MegaMix™, 524.2 Rev. 4.1 (73 Components)

acrylonitrile
allyl chloride
benzene
bromobenzene
bromochloromethane
bromodichloromethane
bromoform
n-butylbenzene
sec-butylbenzene
tert-butylbenzene
carbon disulfide
carbon tetrachloride
chloroacetonitrile
chlorobenzene
1-chlorobutane
chlorodibromomethane (dibromochloromethane)
chloroform
2-chlorotoluene
4-chlorotoluene
1,2-dibromo-3-chloropropane (DBCP)
1,2-dibromoethane (ethylene dibromide)
dibromomethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
trans-1,4-dichloro-2-butene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
cis-1,2-dichloroethene
trans-1,2-dichloroethene
1,2-dichloropropane
1,3-dichloropropane
2,2-dichloropropane
1,1-dichloropropene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
diethyl ether (ethyl ether)
ethylbenzene
ethyl methacrylate
hexachlorobutadiene
hexachloroethane
iodomethane (methyl iodide)
isopropylbenzene (cumene)
4-isopropyltoluene (*p*-cymene)
methacrylonitrile
methyl acrylate
methylene chloride (dichloromethane)
methyl methacrylate
methyl *tert*-butyl ether (MTBE)
naphthalene
nitrobenzene
2-nitropropane
pentachloroethane
propionitrile (ethylcyanide)
n-propylbenzene
styrene
1,1,1,2-tetrachloroethane
1,1,2,2-tetrachloroethane
tetrachloroethene
tetrahydrofuran
1,2,3-trichlorobenzene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethene
1,2,3-trichloropropane
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
toluene
m-xylene
o-xylene
p-xylene

Each	5-pk.	10-pk.
2,000µg/mL each in P&T methanol, 1mL/ampul		
30601	30601-510	—
	w/data pack	
30601-500	30601-520	30701

Ketones Mix, 524.2 Rev. 4.1

acetone
2-butanone (MEK)
1,1-dichloro-2-propanone
2-hexanone
4-methyl-2-pentanone (MIBK)

Each	5-pk.	10-pk.
5,000µg/mL each in 90% P&T methanol:10% water, 1mL/ampul		
30602	30602-510	—
	w/data pack	
30602-500	30602-520	30702

Antifoam Agent for Purge & Trap Samples

- Efficiently controls foam over a wide pH range.
- Effective at less than 0.1% of sample volume.
- Will not conflict with chromatography of target analytes.

Foam generated when purge gas passes through a sample can enter the analytical trap, and possibly into the GC column. Our non-hazardous silica-containing antifoam agent is of great help in volatile organics analyses.

Each	5-pk.
Neat, 1mL/ampul	
31822	31822-510

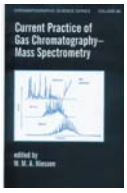
Food for Thought:

New Selections from the Restek Bookshelf

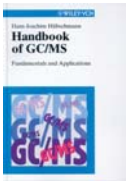
by Jack Crissman, Educational Products Marketing Manager

Many other titles available - see our website or catalog.

Chiral Separations by Liquid Chromatography and Related Technologies
Types, structures, and properties of chiral stationary phases and their preparation, application, and future. Technologies include sub- and super-critical fluid chromatography, capillary electrochromatography, and thin layer chromatography.
H.Y. Aboul-Enein and I. Ali, Marcel Dekker, Inc., 2003, 400pp., ISBN 0-8247-4014-9
cat.# 21449 (ea.) $165



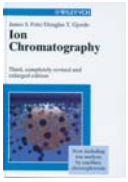
Current Practice of Gas Chromatography-Mass Spectrometry
Principles, instrumentation, and a wide range of applications. 40 contributors, worldwide; more than 1200 references, equations, tables, and graphics. A superb reference for operators, managers, and students.
W.M.A. Niessen, Marcel Dekker, Inc., 2001, 528pp., ISBN 0-8247-0473-8
cat.# 21489 (ea.) $175



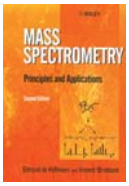
Handbook of GC/MS. Fundamentals and Applications
Sample preparation through data evaluation, including MS library searches and a substance index. Applications include environmental, food, pharmaceutical, and clinical analysis.
Hans-Joachim Hübschmann, Wiley-VCH, 2001, 608pp., ISBN 3-527-30170-4
cat.# 21490 (ea.) $230

Handbook of Size Exclusion Chromatography and Related Techniques. 2nd Ed.
High-speed SEC, SEC of low molecular weight materials, and the extended family of techniques from two-dimensional liquid chromatography to high osmotic pressure chromatography.
C. Wu, Marcel Dekker, Inc., 2003, 716pp., ISBN 0-8247-4710-0
cat.# 21448 (ea.) $195

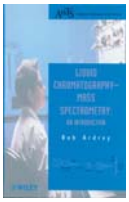
The HPLC Solvent Guide. 2nd Ed.
Even experienced analysts tend to select from three familiar solvents. This guide describes many solvents suitable for HPLC separations.
P.C. Sadek, John Wiley & Sons, Inc., 2002, 664pp., ISBN 0-471-41138-8
cat.# 21979 (ea.) $105



Ion Chromatography. 3rd Ed.
Materials, principles, and methods, including capillary electrophoresis and chemical speciation. Excellent introduction for novices or guide for experienced analysts.
J.S. Fritz and D.T. Gjerde, Wiley-VCH, 2000, 267pp., ISBN 3-527-29914-9
cat.# 21789 (ea.) $135



Mass Spectrometry. Principles and Applications. 2nd Ed.
Principles, theories, and key applications, focused on recent developments. Expanded coverage of ESI and MALDI, and of biological and pharmaceutical applications. For students, and for researchers reviewing the latest techniques and developments.
E. de Hoffmann and V. Stroobant, John Wiley & Sons, Inc., 2001, 420pp., ISBN 0-471-48566-7 cat.# 21978 (ea.) $65



Liquid Chromatography-Mass Spectrometry: An Introduction (softcover)
An indispensable reference for anyone wishing to use this increasingly important tandem technique.
R.E. Ardrey, John Wiley & Sons Ltd., 2003, 296pp., ISBN 0-471-49801-7
cat.# 20176 (ea.) $50

New Reference Materials for Environmental Analyses

by Katia May, Ph.D., Senior R&D Chemist

US EPA Method 8270 Mixes in 100% Methylene Chloride

- Better peak shape for early eluting semivolatiles, compared to methylene chloride/benzene solvent.
- Methanol-free methylene chloride enhances stability.
- Calibration mix and matrix spike mix.

new!

Most complex mixes for US EPA Method 8270 are prepared in combinations of methylene chloride/benzene solvent. The primary reason for using benzene, a high boiling solvent, is the belief that polyaromatic hydrocarbons are not readily soluble in methylene chloride. Benzene, however, can contribute to poor peak shape and low responses for early eluting compounds such as pyridine, N-nitrosomethylamine, N-nitrosomethylethylamine, 1,4-dioxane, and 2-picoline. Restek chemists have studied the solubility of polyaromatic hydrocarbons and determined that our unique method of preparing our Method 8270 MegaMix™ calibration mix (cat.# 31850) allows us to exclude benzene as a solvent.

Methylene chloride alone is an effective solvent for these analytes; methanol-free methylene chloride enhances the stability of the product. Our new 8270 MegaMix™ in methylene chloride is a direct replacement for the older mix, but allows better chromatography.

Similarly, we offer new 8270 Matrix Spike Mix (cat.# 31851), 8270 Benzidines Mix (cat.# 31852), and 1,4-Dioxane (cat.# 31853) in 100% methylene chloride. Equivalents of these three mixes are available in methanol/methylene chloride/benzene (cat.# 31687) or methanol (cat.# 31688, cat.# 30287), respectively.

8270 MegaMix™ (76 components)

acenaphthene	2,4-dinitrophenol
acenaphthylene	2,4-dinitrotoluene
aniline	2,6-dinitrotoluene
anthracene	di-*n*-butyl phthalate
azobenzene[1]	di-*n*-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-cd)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-*n*-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

Each	5-pk.	10-pk.
1,000µg/mL each in methylene chloride, 1mL/ampul*		
31850	31850-510	—
	w/data pack	
31850-500	31850-520	31950

*3-methylphenol and 4-methylphenol at 500µg/mL.
[1]1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).
[2]N-nitrosodiphenylamine (8270-listed analyte) decomposes to diphenylamine (mix component).

8270 Matrix Spike Mix (76 components)

Same components as 8270 MegaMix™, but at lower concentration for spiking.

Each	5-pk.	10-pk.
200µg/mL each in methylene chloride, 5mL/ampul*		
31851	31851-510	—
	w/data pack	
31851-500	31851-520	31951

*3-methylphenol and 4-methylphenol at 100µg/mL.

8270 Benzidines Mix

benzidine 3,3'-dimethylbenzidine
3,3'-dichlorobenzidine

Each	5-pk.	10-pk.
2,000µg/mL in methylene chloride, 1mL/ampul		
31852	31852-510	—
	w/data pack	
31852-500	31852-520	31952

1,4-Dioxane

Each	5-pk.	10-pk.
2,000µg/mL in methylene chloride, 1mL/ampul		
31853	31853-510	—
	w/data pack	
31853-500	31853-520	31953

US EPA Method 524 Surrogates Standard

- Separate mixes for surrogates and internal standard.
- Fortification solution combines surrogates and internal standard.
- Calibration mixes and all other quality control mixes also available.

new!

US EPA Method 524 requires a surrogates standard, an internal standard, and the surrogates and internal standard combined in a fortification solution. We have offered the fortification solution (cat.# 30201) and the internal standard (cat.# 30030); we now offer the surrogates mix, described here. Use the new mix to monitor method performance by combining it with the sample before extraction. Together with Drinking Water VOA MegaMix™ 524.2 Rev. 4.2 calibration mix (cat.# 30601, see page 11), Ketones Mix 524.2 Rev. 4.2 (cat.# 30602, see page 11), and additional calibration and quality control mixes listed in our catalog, the new mix completes our set of reference materials for Method 524.

524.2 Surrogate Standard

1-bromo-4-fluorobenzene 1,2-dichlorobenzene-d4

Each	5-pk.	10-pk.
2,000µg/mL each in P&T methanol, 1mL/ampul		
30607	30607-510	—
	w/data pack	
30607-500	30607-520	30707

Drinking Water Odor Standard

- New reference mix of the two most common odor-causing compounds.
- Convenient concentration for purge and trap analysis: 100µg/mL in methanol.

new!

Unpleasant odor in drinking water is associated with the growth and decay of microorganisms. Blue-green algae, green algae, diatoms, and flagellates are the four groups responsible for most common odor problems. Geosmin, produced by blue-green algae, has an earthy, musty smell. Actimycetes, mold-like bacteria also present in surface water, produce another common odor compound: 2-methylisoborneol.

The threshold value for these compounds is low (10ppt) and purge and trap analyses usually are used to quantify them. To help monitor the quality of drinking water, Restek's researchers have developed this convenient new reference mix.

Drinking Water Odor Standard

(+/-)-geosmin 2-methylisoborneol

Each	5-pk.
100µg/mL each in P&T methanol, 1mL/ampul	
30608	30608-510

Searching for the Perfect Solution?

Let Restek create the perfect reference mixture—to your exact specifications. Contact the Technical Service Team, or your Restek representative, or visit us online at **www.restek.com/solutions**

Make GC/MS Column Changes in Minutes Without Venting

EZ No-Vent™ Connector Allows Fast Re-equilibration with No Loss of Data

by Brad Rightnour, Instrument Innovations Manager,
and Christopher English, Environmental Innovations Chemist

- Cut downtime by 50% or more when changing columns.
- No special tools - no extra plumbing - low cost.
- Gold plated for inertness.
- 100μm ID transfer line keeps analytes focused.

Restek Innovation!

A laser machined critical orifice in the EZ No-Vent™ connector minimizes the amount of air admitted into the MS source, eliminating the need for purge gas and for the lengthy vent and pump-down cycle otherwise required with a column change. This can save hours of downtime with each column change. The EZ No-Vent™ connector easily attaches to the MS source without special tools or extra plumbing: Figure 1 shows the connector installed and ready for use.

We tested the EZ No-Vent™ connector for dead volume, using highly volatile gases that are very susceptible to tailing. For reference, we operated the system as a purge-and-trap GC/MS system, with a split at the injection port and with the column inserted directly into the MS interface. When we installed the EZ No-Vent™ connector in the MS interface, we anticipated that any dead volume in the connector would be revealed by tailing and broader peaks, relative to direct connection. Peak shape was excellent.

After establishing the connector had no effect on peak shape, we evaluated the ability of the MS to stabilize after a column change without venting. Again we used a purge-and-trap system, and halogenated volatiles as the analytes. We acquired Figure 2a, changed the column, and acquired Figure 2b 76 minutes later. Note the peak shapes and responses are unchanged. In the interval between the two analyses we verified MSD tuning, and the system passed bromofluorobenzene (BFB) criteria. Subsequent detailed investigations confirmed the EZ No-Vent™ connector will allow several column changes in a single day, with no harm to the MS or loss of data.

If you're tired of waiting for your MS to stabilize after column changes, we highly recommend an EZ No-Vent™ connector. It will greatly reduce downtime and increase sample throughput.

Figure 1 MS transfer line installed in an EZ No-Vent™ connector. Connection can be made quickly, without special tools.

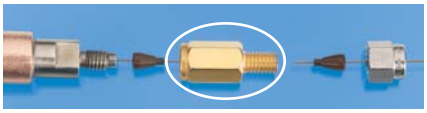
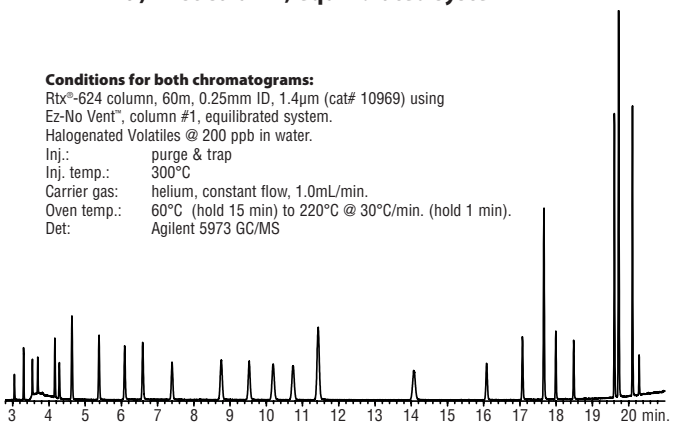


Figure 2 Sample acquired within an hour and a half after changing columns, using an EZ No-Vent™ connector.

a) First column, equilibrated system

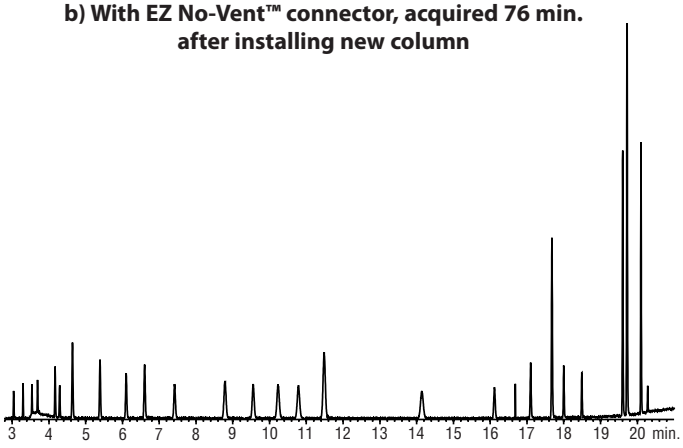
Conditions for both chromatograms:
Rtx®-624 column, 60m, 0.25mm ID, 1.4µm (cat# 10969) using
Ez-No Vent™, column #1, equilibrated system.
Halogenated Volatiles @ 200 ppb in water.

Inj.:	purge & trap
Inj. temp.:	300°C
Carrier gas:	helium, constant flow, 1.0mL/min.
Oven temp.:	60°C (hold 15 min) to 220°C @ 30°C/min. (hold 1 min).
Det:	Agilent 5973 GC/MS



GC_EV00659c

b) With EZ No-Vent™ connector, acquired 76 min. after installing new column



GC_EV00659d

EZ No-Vent™ GC Column-Mass Spectrometer Connector

Description	qty.	cat.#
EZ No-Vent™ Connector Kit for Agilent 5971/5972 and 5973 GC/MS Kit includes: EZ No-Vent™ Connector, two 0.4mm ID ferrules for capillary column, two 0.4mm ID ferrules for transfer line, 100μm deactivated transfer line (3 ft.), column plug, column nut.	kit	21323
Replacement ferrules for connecting capillary column to EZ No-Vent™: 0.4mm ID	2-pk.	21015
0.5mm ID	2-pk.	21016
Replacement ferrules for connecting transfer line to EZ No-Vent™: 0.4mm ID	2-pk.	21043
Replacement 100μm deactivated transfer line	3 ft.	21018
Replacement EZ No-Vent™ Column Nut	5-pk.	21900
Replacement EZ No-Vent™ Plug	2-pk.	21915
Open-End Wrenches ($^{1}/_{4}$" x $^{5}/_{16}$")	2-pk.	20110

Did you know?

Restek offers supplies and innovative tools for your MS. Refer to our general catalog.

Instrument Innovations!

Simplify Life in Your Laboratory

by Donna Lidgett, GC Accessories Product Marketing Manager

Restek's Instrument Innovations Team has been busy introducing new products, to maintain our reputation of supplying superior-quality manufactured parts, accessories, and operating supplies for gas chromatography. We do whatever it takes to provide you with the best—from original equipment manufacturer's replacement parts to our own innovative improvements and special designs.

We are Your #1 Source for Consumables and Supplies!

Injector Wrench for Shimadzu 17A and 2010 GCs

new!

- Designed specifically for removing Shimadzu injection port weldments.
- High-quality stainless steel construction.

Description	Similar to Shimadzu part #	qty.	cat.#
Injector Wrench for Shimadzu GCs	221-46977-00	ea.	21159

Septum Holder Kit for TRACE™ 2000 GCs

new!

- Includes septum support and holder.
- Made from high quality stainless steel.

Description	Similar to TF part #	qty.	cat.#
Septum Holder Kit for TRACE™ 2000 GCs	23303015 350054335	ea.	21299

Silver PTV Seals for Agilent 6890 GCs

new!

Description	Similar to Agilent part #	qty.	cat.#
Silver PTV Seals for Agilent 6890 GCs	5182-9763	5-pk.	21409

Injector Mounting Posts for Agilent Autosamplers

new!

- Performance equivalent to OEM parts.

Description	Similar to Agilent part #	qty.	cat.#
A) Injector Mounting Post for Agilent 7673A & B Autosamplers	18597-60805	ea.	21236
B) Injector Mounting Post for Agilent 7683A Autosampler	07673-21140	ea.	21237
C) Injector Mounting Post for Agilent 7683N Autosampler	G2613-20500	ea.	21172

Splitless Liners for PerkinElmer GCs

new!

Splitless Liners for PerkinElmer GCs	Benefits/Uses:	ID*/OD & Length (mm)	cat.# ea.	5-pk.	25-pk.
 Auto SYS Splitless	headspace & purge & trap	1.0 ID 6.2 OD x 92.1	21272	21273	21274

*Nominal ID at syringe needle expulsion point.

Dual Vespel® Ring Inlet Seals

- Vespel® ring embedded in bottom surface eliminates need for washer.
- Vespel® ring embedded in top surface reduces operator variability by requiring minimal torque to seal.
- Prevents oxygen from permeating the carrier gas, increasing column lifetime.

Eliminate the washer!

Available in Siltek™-treated, gold-plated, or untreated stainless steel.

Our Dual Vespel® Ring Inlet Seal* greatly improves injection port performance—it stays sealed, even after repeated temperature cycles, without retightening the reducing nut! This new version of our popular Vespel® Ring Inlet Seal features two soft Vespel® rings, one embedded in its top surface and the other embedded in its bottom surface. The Vespel® rings eliminate the need for a washer, and ensure very little torque is needed to make a leak-tight seal. The rings will not harm the critical seal in the injector body, and are outside the sample flow path. Tests show Dual Vespel® Ring Inlet Seals will seal equally effectively at torques from 5 to 60 in. lb.

Why trust a metal-to-metal seal when you can make leak-tight seals quickly, easily, and more reliably—without a washer, with a Restek Dual Vespel® Ring Inlet Seal. Use an untreated stainless steel seal for analyses of unreactive compounds. To reduce breakdown and adsorption of active compounds, use a Siltek™-treated or gold-plated seal. Siltek™ treatment provides the highest level of inertness.

0.8mm ID Dual Vespel® Ring Inlet Seal	2-pk.	10-pk.
Siltek™	21242	21243
Gold-Plated	21240	21241
Stainless Steel	21238	21239

1.2mm ID Dual Vespel® Ring Inlet Seal	2-pk.	10-pk.
Siltek™	21248	21249
Gold-Plated	21246	21247
Stainless Steel	21244	21245

*Patent pending.

FID-1000 Gas Station

Convenient, Safe Source of Zero Air and Pure Hydrogen

- Single unit produces UHP zero air from house compressed air and 99.9995% pure hydrogen from deionized water.
- Ideal for supplying 1 - 2 FIDs, FTDs, or FPDs.
- Eliminates inconvenient, dangerous cylinders.
- Silent operation, minimal operator attention required.

new!

Parker Balston's FID-1000 Gas Station provides both UHP grade hydrogen fuel gas and zero grade air (<0.1ppm THC) for flame ionization detectors on gas chromatographs. The system is specifically designed to supply fuel gas to either 1 or 2 FIDs and to support flame thermionic and flame photometric detectors.

The gas station produces up to 1000cc/min. of zero air by purifying compressed air to a total hydrocarbon concentration of less than 0.1ppm (measured as methane).

The hydrogen generator produces hydrogen gas from deionized water, using the principle of electrolytic dissociation of water and hydrogen proton conduction through a proton exchange membrane cell. Hydrogen is supplied at 90cc/min. at pressures up to 60 psig.

Built to International Standards

Produced and supported by an ISO 9001 registered organization, Parker Balston's hydrogen generators are built to meet the toughest laboratory standards - CSA, UL, CE, and IEC 1010.

When ordering an FID-100 Gas Station for use in countries other than the United States, add the appropriate international power cord suffix (see table at right) to the Gas Station cat.#.

Specifications: FID-1000 Gas Station

	Hydrogen	Zero Air
Product Purity	99.9995%	<0.1ppm total hydrocarbons
Flow Rate	90cc/min.	1000cc/min.
Delivery Pressure	60psig	40-125psig*
Inlet Connection	NA	1/4" NPT (female)
Outlet	1/8" compression	1/8" compression

Power Requirements	120VAC/amp
Dimensions	16.5 x 10.5 x 17" (h x w x d) 42 x 27 x 43cm
Weight Dry	46lbs / 21kg

*Zero air inlet requires minimum of 40psig compressed air pressure.

Description	qty.	cat. #
FID-1000 Gas Station	ea.	20177

International Power Cord Sets

Location	qty.	cat.# suffix
United Kingdom (230VAC, 50/50Hz)	ea.	-550
Europe (230VAC, 50/60Hz)	ea.	-551
IEC Connector Only (230VAC, 50/60Hz)	ea.	-552
Japan (200VAC, 50/60Hz)	ea.	-556
Japan, for Zero Air (100VAC, 50/60Hz)	ea.	-553
Japan, for Hydrogen (100VAC, 50/60Hz)	ea.	-554
Japan, for Nitrogen (100VAC, 50/60Hz)	ea.	-555

For hydrogen, nitrogen, or zero air generators, and gas purifiers, refer to our general catalog.

Vespel® Ferrules

- 100% high-temperature polyimide.
- Stable to 350°C.
- Durable, tend to permanently seal to the column.

Fitting Size	Ferrule ID	qty.	cat.#	price
1/16"	0.3mm	10-pk.	22213	
1/16"	0.4mm	10-pk.	22214	
1/16"	0.5mm	10-pk.	22215	
1/16"	0.8mm	10-pk.	22216	
1/16"	1.0mm	10-pk.	22217	
1/16"	1.2mm	10-pk.	22218	
1/16"	1/16"	10-pk.	22210	
1/8"	1/8"	10-pk.	22211	
1/4"	1/4"	10-pk.	22212	
1/4"	1/8"	10-pk.	22219	

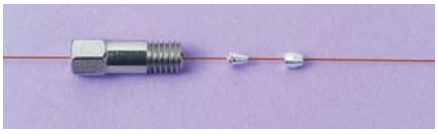
Alumaseal™ Ferrules*

- Aluminum construction, will not crack or fragment.
- Eliminate out-gassing, make leak-tight seals, for less detector noise.
- No retightening after temperature cycles—excellent for GC/MS.
- Unique two-piece design permanently locks on fused silica tubing without causing breakage.
- Will not stick in fittings, like Vespel® or graphite.
- Use with any 1/16" compression-type fitting.

Ferrule ID	Fits Column ID	qty.	cat.#
0.4mm	0.25mm	10-pk.	21472
0.5mm	0.32mm	10-pk.	21473
0.8mm	0.53mm	10-pk.	21474

*Patent pending.

Eliminate Sealing Compromises!



Replacement Lamps for HPLC Detectors

by Rebecca Wittrig, Ph.D., HPLC Product Marketing Manager

- Meet or exceed original manufacturer's performance.
- Simplify paperwork—order parts when you order columns and consumables.

In addition to the lamps listed here, we have an extensive range of HPLC replacement parts and accessories. For a full listing, visit our website, or request publication 59012. If you don't see what you need, please call us—we are constantly adding new parts to meet your needs.

Description	Model #	Similar to OEM part #	qty.	cat.#
For Agilent HPLC Systems				
Detector Lamp, 1090 DA, 1050 VW/DA/MWD	1090, 1050	79883-60002	ea.	25260
Lamp, DAD G1315A, G1365A	1100	2140-0590	ea.	25261
Lamp, VWD G1314A	1100	G1314-60100	ea.	25262
8453 Deuterium Lamp	—	2140-0605	ea.	25263
G1321 Fluorescence Detector Flash Lamp	—	2140-0600	ea.	25264
For Beckman HPLC Systems				
Deuterium Lamp	DU60, 62, 64, 65	596791	ea.	25454
For Hitachi HPLC Systems				
Deuterium Lamp, Prealigned	L4000, L4200, L4250, L7400	885-3570	ea.	25465
For PerkinElmer Instruments				
Deuterium Lamp	PE Lambda--2, 5, 7, 8, 10, 11, 12, 14, 15, 16, 17, 18, 19, 20, 40, 800, 900	B0160917	ea.	25436
Deuterium Lamp	PE 200/785A	N2920149	ea.	25431
For Shimadzu HPLC Systems				
Deuterium Lamp	SPD-6A	062-65056-02	ea.	25283
Deuterium Lamp	SPD-10A, 10AV	228-34016-02	ea.	25284
For Waters™ Detectors				
Xenon Lamp (w/o holder or mirror)	470	—	ea.	25404
Xenon Lamp	474	—	ea.	25405
Deuterium Lamp (UV/Vis)	480, 481	99499	ea.	25403
Deuterium Lamp (UV/Vis)	484	80357	ea.	25406
Deuterium Lamp (UV/Vis)	486	80678	ea.	25407
Deuterium Lamp	996, 2996	WAT052586	ea.	25408
Deuterium Lamp	2487	WAS081142	ea.	25409
Deuterium Lamp, long life (2000 hours)	486	—	ea.	25410

ISO 9001 REGISTERED

Lit. Cat. # 59051

Free Literature!

Genuine Restek Replacement Parts for HPLC Systems

(lit. cat.# 59012*)

Over time, detector lamps, check valves, pump piston seals, and other components wear out or become contaminated. Working with defective parts means poor chromatography and, possibly, shortened column lifetimes. This 4-page publication lists a wide selection of Restek parts for Agilent, Beckman, Hitachi, PerkinElmer, Shimadzu, and Waters instruments, to keep these systems running smoothly and chromatography sharp. Genuine Restek Replacement Parts equal or exceed the performance of original equipment components.

Coming soon… HPLC Columns and Accessories

(lit. cat.# 59241B*)

New, expanded 2004-2005 edition features columns and bulk materials, instrument parts, innovative tools and accessories, and many example chromatograms.

*When requesting literature from outside the US, add "-INT" to the lit. cat.#.

RESTEK

THE RESTEK ADVANTAGE

Turning Visions into Reality

2004 vol. 4

Fast, Sensitive GC Analyses of Semivolatile Organics in Water

new!

Using an 0.18mm ID Rtx®-XLB Column

by Christopher English, Environmental Innovations Chemist

- Extremely low bleed—ideal for GC/MS applications.
- Excellent resolution, high sensitivity for semivolatile compounds in water.
- Stable to 340°C.

A single analysis of semivolatile organic compounds in water, performed according to US EPA Method 8270D or other GC/MS methods, can involve 100 or more analytes having widely diverse chemical properties and reactivity. This complexity puts stringent demands on the column used to perform the analysis. Some analytes elute at high temperatures, for example, so column bleed must be low at high temperature. The column also must exhibit excellent efficiency, to resolve closely eluting compounds with similar mass spectra, and overcome challenges to high sensitivity and low detection limits, for reliable quantification of all target compounds.

In *Advantage* 2004v2 (literature #59037) we showed how a 20m, 0.18mm ID, 0.18µm df Rtx®-5Sil MS column (cat.# 42702) offers excellent selectivity, improves detection limits, and increases productivity in an analysis of a complex mixture of EPA Method 8270 semivolatile compounds. Here, we show equally notable results from our ultra-low-bleed Rtx®-XLB column, in equivalent dimensions, under equivalent conditions.

A 20m, 0.18mm ID, 0.18µm df Rtx®-XLB column (cat.# 42802) is an excellent choice for analyzing semivolatile compounds. The Rtx®-XLB stationary phase is specifically designed for the demanding GC/MS analysis of semivolatiles, and these columns exhibit extremely low bleed. Figure 1 (page 3) is a chromatogram for nearly 90 analytes and surrogates, at 2.5ng each on-column, showing excellent resolution and negligible baseline rise at 330°C. The short length and small internal diameter of these columns ensure faster runtimes, increasing productivity: the last compound elutes in less than 18 minutes. The thin phase film allows satisfactory resolution of structural isomers benzo(b)fluoranthene and benzo(k)fluoranthene in this very short analysis time. Peak shape and response are excellent for active compounds such as pyridine (peak 1), 2,4-dinitrophenol (peak 54), and pentachlorophenol (peak 66); even at this low concentration, all compounds can be quantified with high accuracy.

The temperature program, as well as the physical dimensions of the column, contributes to better resolution of closely eluting peaks and shortens the analysis time. The column accommodates the 330°C final temperature very well, because our QA bleed specification for Rtx®-XLB columns, including the new 0.18mm ID, 0.18µm df column, is less than 6pA at 340°C.

Optimization of injection conditions also is an important consideration in this analysis. To reduce solvent effects with pyridine and N-nitrosodimethylamine we chose to use a splitless injection liner. *(cont. on page 3)*

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

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
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	

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

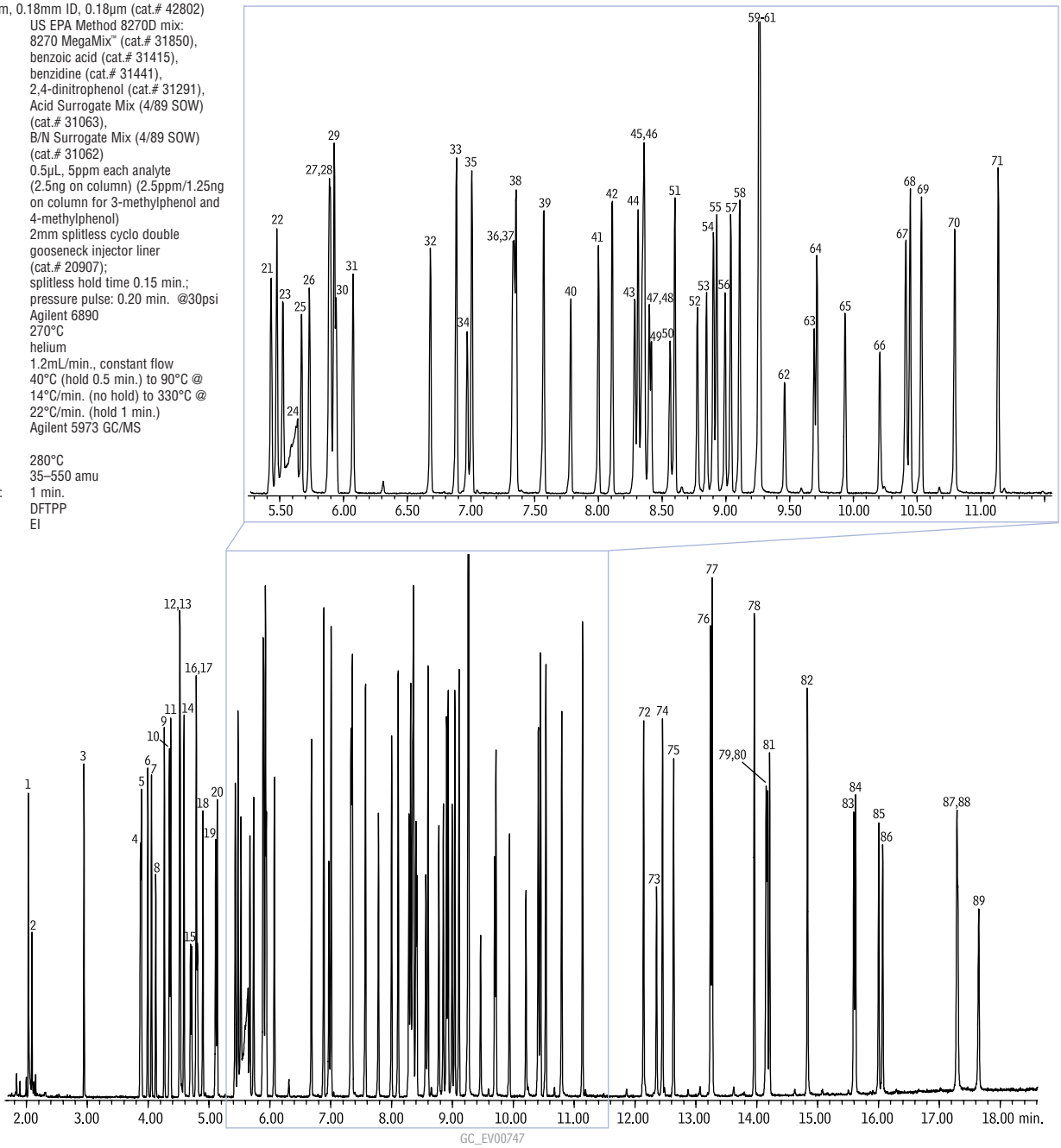
Chromatography on page 3 →

in this issue

Figure 1

70 semivolatile organics, plus surrogates, separated in less than 18 minutes on a 0.18mm ID Rtx®-XLB column.

Rtx®-XLB, 20m, 0.18mm ID, 0.18µm (cat.# 42802)	
Sample:	US EPA Method 8270D mix: 8270 MegaMix™ (cat.# 31850), benzoic acid (cat.# 31415), benzidine (cat.# 31441), 2,4-dinitrophenol (cat.# 31291), Acid Surrogate Mix (4/89 SOW) (cat.# 31063), B/N Surrogate Mix (4/89 SOW) (cat.# 31062)
Inj.:	0.5µL, 5ppm each analyte (2.5ng on column) (2.5ppm/1.25ng on column for 3-methylphenol and 4-methylphenol) 2mm splitless cyclo double gooseneck injector liner (cat.# 20907); splitless hold time 0.15 min.; pressure pulse: 0.20 min. @30psi
GC:	Agilent 6890
Inj. temp.:	270°C
Carrier gas:	helium
Flow rate:	1.2mL/min., constant flow
Oven temp.:	40°C (hold 0.5 min.) to 90°C @ 14°C/min. (no hold) to 330°C @ 22°C/min. (hold 1 min.)
Det.:	Agilent 5973 GC/MS
Transfer line temp.:	280°C
Scan range:	35–550 amu
Solvent delay:	1 min.
Tune:	DFTPP
Ionization:	EI



1. pyridine
2. N-nitrosodimethylamine
3. 2-fluorophenol
4. phenol-d6
5. phenol
6. aniline
7. 2-chlorophenol
8. bis(2-chloroethyl)ether
9. 1,3-dichlorobenzene
10. 1,4-dichlorobenzene-d4
11. 1,4-dichlorobenzene
12. 1,2-dichlorobenzene
13. benzyl alcohol
14. 2-methylphenol
15. bis(2-chloroisopropyl)ether
16. hexachloroethane
17a. 4-methylphenol
17b. 3-methylphenol
18. N-nitroso-di-*n*-propylamine
19. nitrobenzene-d5
20. nitrobenzene
21. isophorone
22. 2,4-dimethylphenol
23. 2-nitrophenol
24. benzoic acid
25. bis(2-chloroethoxy)methane
26. 2,4-dichlorophenol
27. 1,2,4-trichlorobenzene
28. naphthalene-d8
29. naphthalene
30. hexachlorobutadiene
31. 4-chloroanaline
32. 4-chloro-3-methylphenol
33. 2-methylnaphthalene
34. hexachlorocyclopentadiene
35. 1-methylnaphthalene
36. 2,4,6-trichlorophenol
37. 2,4,5-trichlorophenol
38. 2-fluorobiphenyl
39. 2-chloronaphthalene
40. 2-nitroaniline
41. dimethylphthalate
42. acenaphthylene
43. 2,6-dinitrotoluene
44. acenaphthene-d10
45. 1,4-dinitrobenzene
46. acenaphthene
47. 1,3-dinitrobenzene
48. 3-nitroaniline
49. 1,2-dinitrobenzene
50. 4-nitrophenol
51. dibenzofuran
52. 2,3,4,6-tetrachlorophenol
53. 2,3,5,6-tetrachlorophenol
54. 2,4-dinitrophenol
55. diethyl phthalate
56. 2,4-dinitrotoluene
57. 4-chlorophenyl phenyl ether
58. fluorene
59. diphenylamine
60. 4-nitroaniline
61. azobenzene
62. 2,4,6-tribromophenol
63. 4,6-dinitro-2-methylphenol
64. 4-bromophenyl phenyl ether
65. hexachlorobenzene
66. pentachlorophenol
67. phenanthrene-d10
68. phenanthrene
69. anthracene
70. carbazole
71. di-*n*-butylphthalate
72. fluoranthene
73. benzidine
74. pyrene
75. *p*-terphenyl-d14
76. butyl benzyl phthalate
77. bis(2-ethylhexyl)adipate
78. bis(2-ethylhexyl)phthalate
79. benzo(a)anthracene
80. chrysene-d12
81. chrysene
82. di-*n*-octyl phthalate
83. benzo(b)fluoranthene
84. benzo(k)fluoranthene
85. benzo(a)pyrene
86. perylene-d12
87. indeno(1,2,3-cd)pyrene
88. dibenzo(a,h)anthracene
89. benzo(ghi)perylene

A cyclo double gooseneck splitless liner allowed the samples to be completely volatilized in the injection port prior to transfer into the column, and achieved more reproducible results than standard straight splitless liners. A liner with an internal diameter of 2mm worked best with 0.5µL injections. We found that changing the splitless hold time by several seconds could reduce sensitivity by 50%. A pulsed splitless analysis using a pressure pulse 5psi higher than the column backpressure dramatically improved sample transfer onto the column. We extended the pulse 3 seconds (0.05 min.) past the splitless hold time (0.15 min.) to allow excess solvent to be swept away quickly.

We adjusted GC conditions to resolve analytes that coelute and share ions. Phenol and aniline, for example (peaks 5 and 6), were resolved by using an initial ramp rate of 14°C/ min. The key to resolving benzo(b)fluoranthene from benzo(k)fluoranthene (peaks 83 and 84) is to ensure that these analytes elute during the temperature ramp part of the program. If they elute during the final hold time they tend to exhibit band broadening, which affects resolution.

Six reference mixes, including 8270 MegaMix™ calibration mix, were combined to prepare the sample for the analysis in Figure 1. We have carefully determined the components of the MegaMix™ calibration mix for maximum stability. We use highly purified methylene chloride as the solvent, to avoid possible reactions between analytes and trace impurities in the solvent. Because 3-methylphenol and 4-methylphenol coelute, we include each in the MegaMix™ mix at half the concentration of the other components, to enable the user to calibrate at lower levels to quantify these compounds at the required limits. N-nitrosodiphenylamine, a target compound in Method 8270D, readily oxidizes to diphenylamine and nitric oxide, a highly reactive gas that can participate in many chemical reactions or act as a catalyst for other oxidation and reduction reactions in the mix. Consequently, we include diphenylamine, rather than N-nitrosodiphenylamine, in the 8270 MegaMix™ mix, to prevent degradation of other components within the mix. Another target compound, diphenylhydrazine, also oxidizes easily, forming azobenzene, so we include azobenzene, not diphenylhydrazine, in the 8270 MegaMix™ mix to assure stability. The stability of an unopened ampul of 8270 MegaMix™ mix, 18 months, is determined by real-time analysis. In addition to the best choice for analytical column, and stable calibration mixtures, we also have available internal standards, surrogate standards, and other reference mixes recommended for analyses of semivolatiles.

If you are analyzing for semivolatile compounds by a GC/MS method, we recommend you evaluate a 0.18mm ID Rtx®-XLB column, and Restek reference mixes, for highest productivity and most reliable data.

8270 MegaMix™ (76 components)

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

Each	5-pk.	10-pk.
1,000µg/mL each (except where noted) in methylene chloride, 1mL/ampul*		
31850	31850-510	—
	w/data pack	
31850-500	31850-520	31950

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

2,4-Dinitrophenol

Each	5-pk.	10-pk.
31291	31291-510	—
	w/data pack	
31291-500	31291-520	31391

Acid Surrogate Mix (4/89 SOW)

2-fluorophenol
phenol-d6
2,4,6-tribromophenol

Each	5-pk.	10-pk.
2,000µg/mL each in methanol, 1mL/ampul		
31025	31025-510	—
	w/data pack	
31025-500	31025-520	31125
10,000µg/mL each in methanol, 1mL/ampul		
31063	31063-510	—
	w/data pack	
31063-500	31063-520	31163
10,000µg/mL each in methanol, 5mL/ampul		
31087	31087-510	—
	w/data pack	
31087-500	31087-520	31187

B/N Surrogate Mix (4/89 SOW)

2-fluorobiphenyl
nitrobenzene-d5
p-terphenyl-d14

Each	5-pk.	10-pk.
1,000µg/mL each in methylene chloride, 1mL/ampul		
31024	31024-510	—
	w/data pack	
31024-500	31024-520	31124
5,000µg/mL each in methylene chloride, 1mL/ampul**		
31062	31062-510	—
	w/data pack	
31062-500	31062-520	31162
5,000µg/mL each in methylene chloride, 5mL/ampul**		
31086	31086-510	—
	w/data pack	
31086-500	31086-520	31186

**Requires warming and sonication before use.

Benzoic Acid

Each	5-pk.	10-pk.
31415	31415-510	—
	w/data pack	
31415-500	31415-520	31515

Benzidine

Each	5-pk.	10-pk.
31441	31441-510	—
	w/data pack	
31441-500	31441-520	31541

Inlet Liners

For Agilent GCs

Cyclo Double Gooseneck
(2.0mm ID, 6.5mm OD, 78.5mm length)
20907 (ea.)
20908 (5-pk.)

For PerkinElmer GCs

Splitless
(2.0mm ID, 5.0mm OD, 100mm length)
20730 (ea.)
20731 (5-pk.)
20732 (25-pk.)

For Thermo Finnigan 5000-6000 GCs

Splitless
(2.0mm ID, 5.4mm OD, 79.5mm length)
20811 (ea.)
20812 (5-pk.)
20813 (25-pk.)

For Varian 1075/1077GCs

Splitless
(2.0mm ID, 6.3mm OD, 74mm length)
20721 (ea.)
20722 (5-pk.)
20723 (25-pk.)

Siltek™ and Silcosteel®-CR Treated Fittings and Tubing for Demanding Applications

by Gary Barone, Restek Performance Coatings Division

- Siltek™ treatment for exceptional inertness.
- Silcosteel®-CR treatment for protection from acids or seawater.
- Treated surfaces will not chip, flake, or delaminate.
- Custom treatment available.

Siltek™ and Silcosteel®-CR Treated Swagelok® Fittings

Swagelok® fittings are world-renowned for meeting demanding standards. Now, a wide selection of Swagelok® products, available from stock with Restek's unparalleled surface treatments, set the highest standards for inertness and corrosion resistance.

Restek *Performance* Coatings

Siltek™ treated fittings ensure ultimate inertness, and are the ideal choice for systems used to collect, store, and transfer active compounds.* The most reactive sample components can be retained in a Siltek™ treated system: even at parts-per-billion levels, sulfur-containing compounds, chlorinated pesticides, or other very active compounds exhibit virtually no adsorption. And, unlike coatings, the protective layer produced by Siltek™ or other Restek treatments is integral with the surface - it will not chip, flake, or delaminate, not even in the most stressful applications.

Silcosteel®-CR treatment is highly effective protection for stainless steel exposed to hydrochloric acid, nitric acid, or sulfuric acid, or to marine environments. In independent tests, Silcosteel®-CR treatment upgraded the corrosion resistance of 300-grade stainless steel by an order of magnitude (Table 1) and totally protected samples against crevice corrosion (Figure 1).

If you need to construct a tubing system for a demanding application, you will not find more suitable fittings than the Siltek™ and Silcosteel®-CR treated Swagelok® fittings listed on page 5. If you already have the components of your system, or need unusual parts, Siltek™, Silcosteel®-CR, or other Restek surface treatments can be applied to these parts on request. For information, contact our Technical Service chemists or your Restek representative.

Table 1 Silcosteel®-CR treated stainless steel coupons show little weight loss after exposure to 6% w/w ferric chloride solution.

Sample	Weight Loss (g/m²)
Silcosteel®-CR	19
Silcosteel®-CR	25
Silcosteel®-CR	25
Bare Steel	231
Bare Steel	20
Bare Steel	228

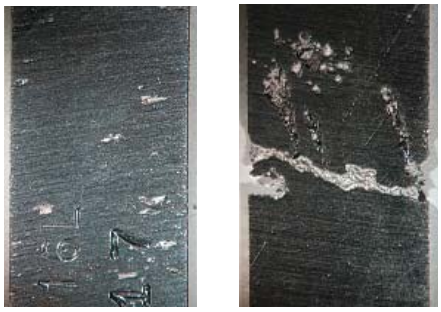
Siltek™ and Silcosteel®-CR Treated Electropolished Stainless Steel Tubing

Restek also sets the highest standard of inertness for transfer tubing for analytical and process applications. The near-mirror finish inside our electropolished tubing (surface roughness of only 5-7 micro-inches), in combination with our unequalled surface treatments, ensures superior inertness or greatly enhanced corrosion resistance. We can provide continuous coils of treated 1/8" tubing up to 100 feet/30.5m long, or coils of 1/4" tubing up to 300 feet/91.4m long; these lengths of treated electropolished tubing are not available anywhere else.

Extremely inert, Siltek™ treated tubing is ideal in systems used for transferring active sulfur-containing compounds, for testing automotive exhaust or sampling stack gas, for process monitoring, or in any other application in which a representative sample of chemically active compounds must be transferred without loss.

In systems used to transfer hydrochloric, nitric, or sulfuric acid, or seawater, Silcosteel®-CR treated electropolished stainless steel tubing will last longer and require less maintenance than untreated tubing. Table 1 and Figure 1 show Silcosteel®-CR treated stainless steel is very well protected from both pitting and crevice corrosion, compared to untreated stainless steel.

Figure 1 Silcosteel®-CR treated 316L stainless steel coupons show no crevice corrosion and only slight pitting corrosion; untreated coupons exhibit severe crevice corrosion.



Silcosteel®-CR treated — untreated

Total Protection

For maximum inertness, we recommend a sample transfer system constructed from Restek treated electropolished stainless steel tubing and Restek treated Swagelok® fittings. To find out how these components can improve the reliability of your data, and/or minimize costly, time-consuming maintenance, contact our Technical Service Group (ext. 4), or your Restek representative, and speak with our surface treatment experts.

*Siltek™ treatment is the multiple-purpose equivalent to Sulfinert® treatment, the surface we apply specifically to systems used to collect, store, and transfer active sulfur-containing compounds.

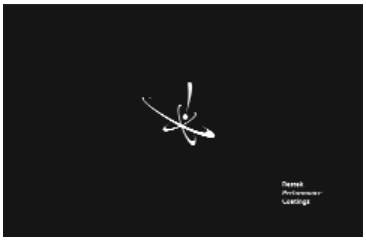
Restek Surface Technology Earns R&D 100 Award

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.

New Publication Features Restek Surface Technology

Learn more about our precisely applied, highly durable surface treatments: request our new 38-page brochure today (lit. cat.# 59493), or review it on our **new** Performance Coatings Division website:

www.restekcoatings.com



Fittings from Swagelok®

- Siltek™ treatment ensures ultimate inertness.
- Silcosteel®-CR treatment enhances corrosion resistance tenfold, or more.
- Custom treatment available for other Swagelok® fittings or other system parts.

Fitting Type	Size	Similar to Swagelok® #	Siltek™ qty.	Siltek™ cat.#	Silcosteel®-CR qty.	Silcosteel®-CR cat.#
Union	1/16"	SS-100-6	ea.	22540	ea.	22575
	1/8"	SS-200-6	ea.	22541	ea.	22576
	1/4"	SS-400-6	ea.	22542	ea.	22577
Tee	1/16"	SS-100-3	ea.	22543	ea.	22578
	1/8"	SS-200-3	ea.	22544	ea.	22579
	1/4"	SS-400-3	ea.	22545	ea.	22580
Reducing Union	1/8" to 1/16"	SS-200-6-1	ea.	22546	ea.	22581
	1/4" to 1/16"	SS-400-6-1	ea.	22547	ea.	22582
	1/4" to 1/8"	SS-400-6-2	ea.	22548	ea.	22583
Elbow	1/8"	SS-200-9	ea.	22549	ea.	22584
	1/4"	SS-400-9	ea.	22550	ea.	22585
Port Connector	1/8"	SS-201-PC	ea.	22557	ea.	22592
	1/4"	SS-401-PC	ea.	22558	ea.	22593
	1/8" tube to 1/4"	SS-401-PC-2	ea.	22559	ea.	22594
Male Connector	1/8" to 1/8" NPT	SS-200-1-2	ea.	22561	ea.	22595
	1/4" to 1/4" NPT	SS-400-1-4	ea.	22562	ea.	22596
	1/16" to 1/8" NPT	SS-100-1-2	ea.	22563	ea.	22610
	1/8" to 1/4" NPT	SS-200-1-4	ea.	22564	ea.	22611
	1/4" to 1/8" NPT	SS-400-1-2	ea.	22565	ea.	22612
Female Connector	1/8" to 1/8" NPT	SS-200-7-2	ea.	22566	ea.	22613
	1/4" to 1/4" NPT	SS-400-7-4	ea.	22567	ea.	22614
	1/4" to 1/8" NPT	SS-400-7-2	ea.	22568	ea.	22615
	1/8" to 1/4" NPT	SS-200-7-4	ea.	22569	ea.	22616
Female Connector	1/8"	SS-200-61	ea.	22570	ea.	22617
	1/4"	SS-400-61	ea.	22571	ea.	22618

Internal surface smoothness in stainless steel tubing: a smoother surface is less adsorptive.
Top: electropolished finish, surface roughness average number: 5-10.
Bottom: conventional finish, surface roughness average number: approx. 23-27.

Silcosteel®-CR-Treated Electropolished Tubing

ID	OD	cat.#	5-24 ft.	25-99 ft.	100-299 ft.	>300 ft.
0.085"	1/8"	22536				
0.180"	1/4"	22537				

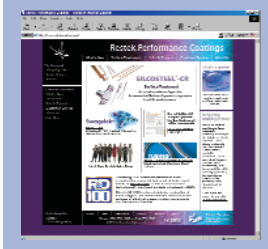
Siltek™-Treated Electropolished Tubing

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

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.

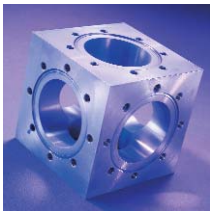
Performance Coatings Division Website Now Up and Running!

www.restekcoatings.com



- Descriptions and performance information about our innovative surface treatments.
- Frequently-asked questions.
- Bibliography of technical articles discussing surface passivation.
- Restek literature to download or request by mail.
- Stock treated tubing, fittings, and other items.
- Electronic custom request form.

We welcome your comments and suggestions! Discover our capabilities here, then give us your toughest surface activity problems, and let us do what "cannot be done" for you.



did you know?

Restek surface treatments are not only used in 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.

Above-Ambient GSC Analysis of Permanent Gases and Light Hydrocarbons

Using ShinCarbon ST Micropacked Columns

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

- Rapid separations, including CO/CO_2, without cryogenics.
- Minimal baseline rise with GC/GSC detectors.
- Fast stabilization - ready to use in 30 minutes.

ShinCarbon ST, a high surface area carbon molecular sieve (~1500 m^2/g), is the GSC medium of choice for gases and highly volatile compounds. A 2-meter, 1mm ID ShinCarbon ST micropacked column separates the permanent gases in about 10 minutes, without cryogenic cooling (Figure 1), and separates permanent gas / light hydrocarbon mixtures quickly and effectively (Figure 1). Additional applications for ShinCarbon ST include natural gas (Figure 2), sulfur dioxide, and Freon® fluorocarbons (Figure 3).*

ShinCarbon ST is highly stable. A 330ºC upper temperature limit ensures minimal bleed and baseline rise during temperature programming and makes the material compatible with most detection systems used for gas analysis, including TCDs and HIDs. ShinCarbon ST columns are conditioned in an oxygen/moisture free environment to minimize stabilization time (less than 30 minutes) when installing a new column.

ShinCarbon ST columns eliminate the temperature and bleed problems that can complicate analyses of permanent gases, light hydrocarbons, and other highly volatile compounds, and provide excellent resolution and analysis times for these applications. Micropacked columns and 2-meter, 2mm ID packed columns are available from stock; custom columns are available on request.

Figure 1

Rapidly separate permanent gases or permanent gas / light hydrocarbon mixtures, without cryogenic cooling, on a ShinCarbon ST micropacked column.

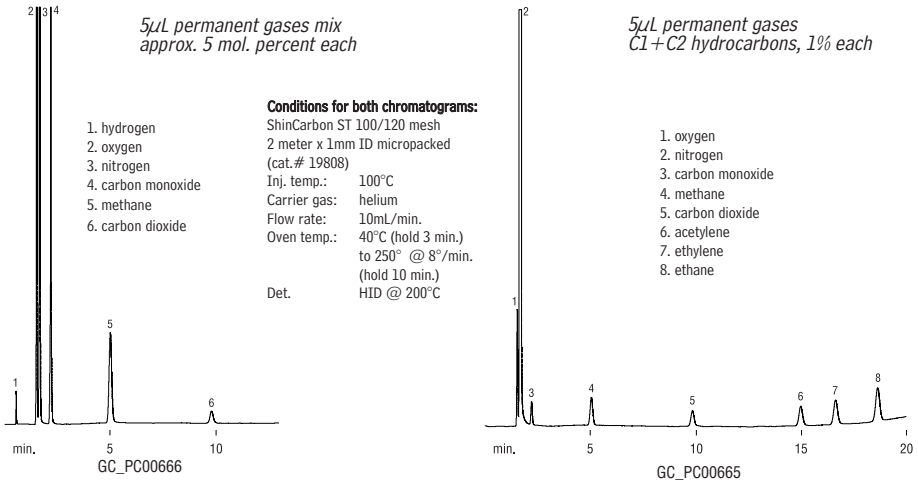


Figure 2

Monitor components of natural gas on a Shincarbon ST micropacked column.

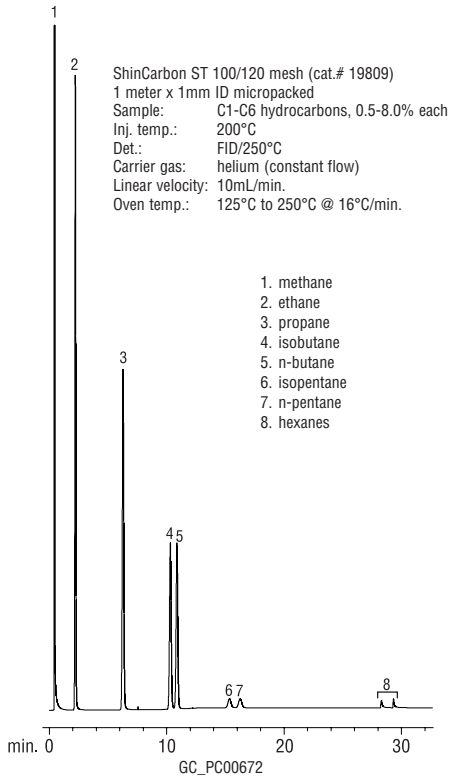
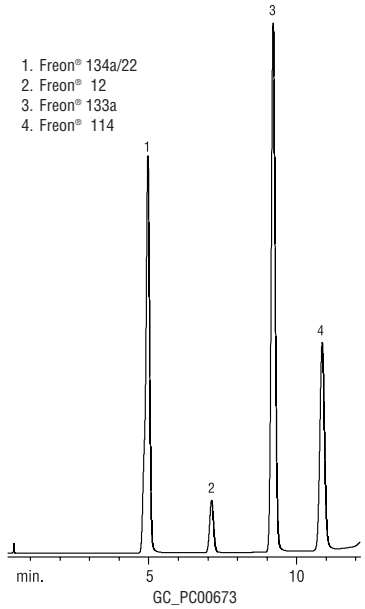


Figure 3

Fluorocarbons separated in 11 minutes on a ShinCarbon ST column.



ShinCarbon ST 100/120 mesh (cat.# 19809)
1 meter x 1mm ID micropacked
Sample: 5µL, ~1-3% each
Inj. temp.: 200°C
Det.: FID/250°C
Carrier gas: helium
Linear velocity: 10mL/min.
Oven temp.: 125°C to 320°C @ 16°C/min.

ShinCarbon ST 100/120 Micropacked Columns (Silcosteel®-treated stainless steel)

OD	ID	1-Meter	2-Meter
1/16"	1.0mm	19809	19808
0.95mm	0.75mm	19810	—

ShinCarbon ST 80/100 Packed Columns (Silcosteel®-treated stainless steel)

OD	ID	2-Meter**
1/8"	2.0mm	80486-

Installation Kits for Micropacked Columns

	for 0.75mm ID col.	for 1mm ID col.
For valve applications	21062	21065
For split applications	21063	—
For all Agilent GCs	21064	—
For direct injections	—	21066

Refer to our catalog or website for
Scott gas standards for permanent gases and light hydrocarbons

*For a chromatogram of a sulfur dioxide analysis, visit our website or request publication 59519A.
** Refer to our catalog or website for available configurations.

Redesigned Rt-QPLOT™ GSC Columns

For Improved Inertness, Reproducibility, and Column Lifetime

by Barry Burger, Petroleum Chemist

- Sharp peaks and excellent resolution for alcohols.
- Effective particle bonding eliminates the need for particle traps.
- Stable to 310ºC, for effective reconditioning.

The most common mode of gas chromatography, gas-liquid chromatography (GLC), has limitations in analyses of gases and highly volatile analytes. Subambient temperatures often are required in these applications, and cryogenic cooling systems are costly and inconvenient. In gas-solid chromatography (GSC) these small molecules are absorbed into the pore structure of the packing material, which provides strong retention and unique selectivity. By this approach, difficult-to-separate gaseous or highly volatile analytes can be separated at above ambient temperatures.

Just as capillary columns offer advantages over packed GLC columns, porous layer open tubular columns—PLOT columns—offer significant advantages over packed GSC columns. PLOT columns provide faster and more sensitive analyses. Their open design gives PLOT columns greater permeability, and their narrow diameter ensures sharper peaks. The open construction affords a smaller pressure drop per unit length, so longer columns can be used. This means much higher column efficiency and, therefore, superior resolution.

Rt-QPLOT™, Rt-SPLOT™, and Rt-UPLOT™ columns are porous polymer based PLOT columns that incorporate polar functional groups in a styrene/divinylbenzene matrix. The least polar of these, the nonpolar Rt-QPLOT™ columns, are made with divinylbenzene. Rt-QPLOT™ columns are well suited to a wide variety of ambient-temperature analyses, including hydrocarbon mixtures and solvents. An Rt-QPLOT™ column is an excellent choice for analyzing alcohols (Figure 1) or polar solvents (Figure 2). Additional separations on Rt-QPLOT™ columns are in the GC Applications section of our catalog (Permanent Gases, Hydrocarbon Gases, and Solvents sub-sections), and on our website.

Why Use RESTEK PLOT Columns?

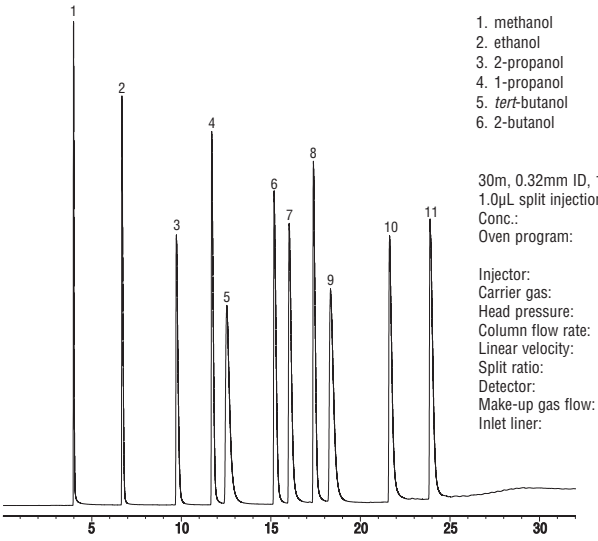
1. Uniform, highly consistent porous materials, for the most efficient and consistent analyses.
2. Advanced manufacturing technology and demanding quality assurance ensure highly reproducible quality.
3. Choice of alumina, three porous polymers, and molecular sieve—you can perfectly match a column to your application.
4. Highly efficient particle bonding - no loose particles to enter your system, no need for particle traps.

An Rt-QPLOT™ column requires no particle trap and exhibits minimum bleed (<20pA at 310°C). Unlike alumina or molecular sieve PLOT columns, porous polymer PLOT columns are unaffected by moisture, and thus are particularly useful for applications in which water is likely to be part of the sample.

We think you will find the performance, column lifetime, and reproducibility of our PLOT columns superior to any other PLOT columns available.

Figure 1

Sharp peaks and excellent resolution of alcohols on an Rt-QPLOT™ column.



1. methanol
2. ethanol
3. 2-propanol
4. 1-propanol
5. *tert*-butanol
6. 2-butanol
7. isobutyl alcohol
8. 1-butanol
9. 2-methyl-2-butanol
10. 3-methyl-1-butanol
11. 4-methyl-2-pentanol

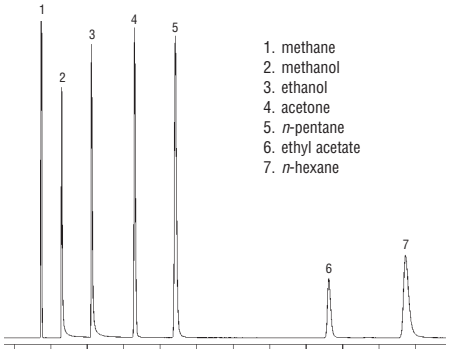
30m, 0.32mm ID, 10µm Rt-QPLOT™ (cat.# 19718)
1.0µL split injection of alcohol mixture

Conc.:	1% of each compound in water
Oven program:	100°C to 240°C @ 5°C/min. (hold 10 min.)
Injector:	split @ 250°C
Carrier gas:	helium (constant pressure mode)
Head pressure:	18.0psi
Column flow rate:	1.1cc/min. @ 100°C
Linear velocity:	31cm/sec. @ 100°C
Split ratio:	70:1
Detector:	FID @ 270°C
Make-up gas flow:	45cc/min.
Inlet liner:	4mm single gooseneck (cat.# 20798)

GC_CH00495

Figure 2

Fast, baseline resolution of polar and nonpolar solvents on an Rt-QPLOT™ column.



1. methane
2. methanol
3. ethanol
4. acetone
5. *n*-pentane
6. ethyl acetate
7. *n*-hexane

GC_PC00181

30m, 0.32mm ID Rt-QPLOT™ PLOT (cat.# 19718)
20µL split injection. 50ppm (w/v) each in helium with a Cyclosplitter® liner (cat.# 20755)

Oven temp.:	150°C
Inj./det. temp.:	200°C
Carrier gas:	hydrogen
Det.:	FID
Split ratio:	20/1

Rt-QPLOT™ PLOT Columns (fused silica)

(divinylbenzene)

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.32mm	10	to 310°C	19717	19718
0.53mm	20	to 310°C	19715	19716

For performance and ordering information for Rt-SPLOT™ and Rt-UPLOT™ columns, refer to our catalog or website.

New Analytical Reference Materials in 2004

by Katia May, Ph.D., Senior R&D Chemist

- Environmental mixes: volatiles - semivolatiles - pesticides/herbicides - petroleum products.
- Miscellaneous mixes: deactivation reagent - drinking water odor standard.
- Custom mixes and packaging always available.

Restek's chemists worked hard during 2004 to introduce many new reference mixes. For your convenience, we've listed them all here, because they are not included in our 2004 general catalog. They also are available on our website. Look for many more new Restek mixes in 2005.

Volatile Organics

Drinking Water VOA MegaMix™, 524.2 Rev. 4.1 (73 components)

acrylonitrile
allyl chloride
benzene
bromobenzene
bromochloromethane
bromodichloromethane
bromoform
n-butylbenzene
sec-butylbenzene
tert-butylbenzene
carbon disulfide
carbon tetrachloride
chloroacetonitrile
chlorobenzene
1-chlorobutane
chlorodibromomethane (dibromochloromethane)
chloroform
2-chlorotoluene
4-chlorotoluene
1,2-dibromo-3-chloropropane (DBCP)
1,2-dibromoethane (ethylene dibromide)
dibromomethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
trans-1,4-dichloro-2-butene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
cis-1,2-dichloroethene
trans-1,2-dichloroethene
1,2-dichloropropane
1,3-dichloropropane
2,2-dichloropropane
1,1-dichloropropene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
diethyl ether (ethyl ether)
ethylbenzene
ethyl methacrylate
hexachlorobutadiene
hexachloroethane
iodomethane (methyl iodide)
isopropylbenzene (cumene)
4-isopropyltoluene (p-cymene)
methacrylonitrile
methyl acrylate
methylene chloride (dichloromethane)
methyl methacrylate
methyl tert-butyl ether (MTBE)
naphthalene
nitrobenzene
2-nitropropane
pentachloroethane
propionitrile (ethylcyanide)
n-propylbenzene
styrene
1,1,1,2-tetrachloroethane
1,1,2,2-tetrachloroethane
tetrachloroethene
tetrahydrofuran
1,2,3-trichlorobenzene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethene
1,2,3-trichloropropane
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
toluene
m-xylene
o-xylene
p-xylene

Each	5-pk.	10-pk.
2,000µg/mL each in P&T methanol, 1mL/ampul		
30601	30601-510	—
	w/data pack	
30601-500	30601-520	30701

Ketones Mix, 524.2 Rev. 4.1

acetone
2-butanone (MEK)
1,1-dichloro-2-propanone
2-hexanone
4-methyl-2-pentanone (MIBK)

Each	5-pk.	10-pk.
5,000µg/mL each in 90% P&T methanol:10% water, 1mL/ampul		
30602	30602-510	—
	w/data pack	
30602-500	30602-520	30702

524 Internal Standard/Surrogate Mix

4-bromofluorobenzene
1,2-dichlorobenzene-d4
fluorobenzene

Each	5-pk.	10-pk.
2,000µg/mL each in P&T methanol, 1mL/ampul		
30201	30201-510	—
	w/data pack	
30201-500	30201-520	30301

524.2 Surrogate Standard

1-bromo-4-fluorobenzene
1,2-dichlorobenzene-d4

Each	5-pk.	10-pk.
2,000µg/mL in P&T methanol, 1mL/ampul		
30607	30607-510	—
	w/data pack	
30607-500	30607-520	30707

Volatiles MegaMix™ with Gases

(60 components)

benzene
bromobenzene
bromochloromethane
bromodichloromethane
bromoform
bromomethane (methyl bromide)
n-butylbenzene
sec-butylbenzene
tert-butylbenzene
carbon tetrachloride
chlorobenzene
chloroethane (ethyl chloride)
chloroform
chloromethane (methyl chloride)
2-chlorotoluene
4-chlorotoluene
dibromochloromethane
1,2-dibromo-3-chloropropane
1,2-dibromoethane (EDB)
dibromomethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
dichlorodifluoromethane (CFC-12)
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethylene
cis-1,2-dichloroethylene
trans-1,2-dichloroethylene
1,2-dichloropropane
1,3-dichloropropane
2,2-dichloropropane
1,1-dichloropropene
trans-1,3-dichloropropene
cis-1,3-dichloropropylene
ethylbenzene
hexachloro-1,3-butadiene (hexachlorobutadiene)
isopropylbenzene (cumene)
4-isopropyltoluene (p-cymene)
methylene chloride (dichloromethane)
naphthalene
n-propylbenzene
styrene
toluene
1,1,1,2-tetrachloroethane
1,1,2,2-tetrachloroethane
tetrachloroethylene
1,2,4-trichlorobenzene
1,2,3-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethylene
trichlorofluoromethane (CFC-11)
1,2,3-trichloropropane
1,3,5-trimethylbenzene
1,2,4-trimethylbenzene
vinyl chloride
m-xylene
o-xylene
p-xylene

Each	5-pk.	10-pk.
200µg/mL each in P&T methanol, 1mL/ampul		
30603	30603-510	—
	w/data pack	
30603-500	30603-520	30703

Formaldehyde-DNPH Mix

Each	5-pk.
500µg/mL in acetonitrile, 1mL/ampul	
31837	31837-510

free literature

Optimizing the Analysis of Volatile Organic Compounds

72-page guide to analyzing volatile compounds in environmental samples, with many example chromatograms. An excellent resource for the new or experienced analyst. Request lit. cat.# 59887A from your Restek representative.

Semivolatile Organics

8270 MegaMix™ (76 components)

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

Each	5-pk.	10-pk.
1,000µg/mL each (except where noted) in methylene chloride, 1mL/ampul		
31850	31850-510	—
	w/data pack	
31850-500	31850-520	31950

*3-methylphenol and 4-methylphenol concentration is 500µg/mL.
[1]1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).
[2]N-nitrosodiphenylamine (8270-listed analyte) decomposes to diphenylamine (mix component).

8270 Matrix Spike Mix (76 components)

same list as 8270 MegaMix™ above

Each	5-pk.	10-pk.
200µg/mL each in methylene chloride, 5mL/ampul**		
31851	31851-510	—
	w/data pack	
31851-500	31851-520	31951

**3-methylphenol and 4-methylphenol concentration is 100µg/mL.

1,4-Dioxane

Each	5-pk.	10-pk.
2,000µg/mL in methylene chloride, 1mL/ampul		
31853	31853-510	—
	w/data pack	
31853-500	31853-520	31953

8270 Benzidines Mix

benzidine
3,3'-dimethylbenzidine
3,3'-dichlorobenzidine

Each	5-pk.	10-pk.
2,000µg/mL in methylene chloride, 1mL/ampul		
31852	31852-510	—
	w/data pack	
31852-500	31852-520	31952

PAH Mixes

EPA Method 8310 Quality Control Check*

(18 components)

acenaphthene	100µg/mL	dibenzo(a,h)anthracene	10
acenaphthylene	100	fluoranthene	10
anthracene	100	fluorene	100
benzo(a)anthracene	10	indeno(1,2,3-cd)pyrene	10
benzo(a)pyrene	10	1-methylnaphthalene	100
benzo(b)fluoranthene	10	2-methylnaphthalene	100
benzo(ghi)perylene	10	naphthalene	100
benzo(k)fluoranthene	5	phenanthrene	100
chrysene	10	pyrene	10

Each	5-pk.	10-pk.
In acetonitrile, 1mL/ampul		
31843	31843-510	—
	w/data pack	
31843-500	31843-520	31943

Sonicate before using.

EPA Method 8310 PAH Mixture*

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

Each	5-pk.	10-pk.
500µg/mL each in acetonitrile, 1mL/ampul		
31841	31841-510	—
	w/data pack	
31841-500	31841-520	31941

Sonicate before using.

EPA Method 8310 Surrogate Standard

decafluorobiphenyl

Each	5-pk.	10-pk.
1,000µg/mL in acetonitrile, 1mL/ampul		
31842	31842-510	—
	w/data pack	
31842-500	31842-520	31942

Phthalate and Adipate Mixes

506 Laboratory Performance Check Mix

benzyl butyl phthalate	250µg/mL	di-n-octyl phthalate	650
bis(2-ethylhexyl)adipate	1200	diethylphthalate	100
bis(2-ethylhexyl)phthalate	250	dimethylphthalate	100
di-n-butylphthalate	100		

Each	5-pk.	10-pk.
In P&T methanol, 1mL/ampul		
31844	31844-510	—
	w/data pack	
31844-500	31844-520	31944

506 Calibration Mix

benzyl butyl phthalate	di-n-octyl phthalate
bis(2-ethylhexyl)adipate	diethylphthalate
bis(2-ethylhexyl)phthalate	dimethylphthalate
di-n-butylphthalate	

Each	5-pk.	10-pk.
1,000µg/mL in isooctane, 1mL/ampul		
31845	31845-510	—
	w/data pack	
31845-500	31845-520	31945

Why choose Restek reference mixes?

- Carefully formulated for dependability and long shelf life.
- Convenient, economical one-stop shopping: columns, supplies, reference mixes.
- Plus 1™ service, always.

Pesticide and Herbicide Mixes

Phenylurea Pesticide Mixture

diflubenzuron	propanil
diuron	siduron
fluometuron	tebuthiuron
linuron	thidiazuron

Each	5-pk.	10-pk.
200µg/mL each in acetonitrile:acetone, 1mL/ampul		
32434	32434-510	—
	w/data pack	
32434-500	32434-520	32534

Phenylurea Surrogate Mixture

carbazole	monuron

Each	5-pk.	10-pk.
500µg/mL each in methanol:acetonitrile, 1mL/ampul		
32433	32433-510	—
	w/data pack	
32433-500	32433-520	32533

531.2 Carbamate Pesticide Calibration Mixture

aldicarb	methiocarb
aldicarb sulfone	methomyl
aldicarb sulfoxide	1-naphthol
carbaryl (sevin)	oxamyl
carbofuran	propoxur (baygon)
3-hydroxycarbofuran	

Each	5-pk.	10-pk.
100µg/mL in acetonitrile, 1mL/ampul		
32435	32435-510	—
	w/data pack	
32435-500	32435-520	32535

Paraquat & Diquat Calibration Mix

diquat dibromide	paraquat dichloride

Each
1,000µg/mL each in water, 1mL/ampul
32437
w/data pack
32437-500

Ultra Quat Reagent Solution

Each	10-pk.
In water, 20mL/ampul	
32441	32541

Petroleum Reference Standards

MA VPH Standard with Surrogate (Revised)

benzene	n-nonane (C9)
n-butylcyclohexane	n-pentane (C5)
n-decane (C10)	toluene
2,5-dibromotoluene	1,2,4-trimethylbenzene
ethylbenzene	2,2,4-trimethylpentane (isooctane)
2-methylpentane	m-xylene
methyl tert-butyl ether (MTBE)	o-xylene
naphthalene	p-xylene

Each	5-pk.	10-pk.
10,000µg/mL in P&T methanol, 1mL/ampul		
30604	30604-510	—
	w/data pack	
30604-500	30604-520	30704

MA VPH Matrix Spike Mix with Surrogate (Revised)

benzene	n-nonane (C9)
n-butylcyclohexane	n-pentane (C5)
n-decane (C10)	toluene
2,5-dibromotoluene	1,2,4-trimethylbenzene
ethylbenzene	2,2,4-trimethylpentane (isooctane)
2-methylpentane	m-xylene
methyl tert-butyl ether (MTBE)	o-xylene
naphthalene	p-xylene

Each	5-pk.	10-pk.
50µg/mL in P&T methanol, 1mL/ampul		
30605	30605-510	—
	w/data pack	
30605-500	30605-520	30705

Hydraulic Oil Standard

Each	5-pk.	10-pk.
50,000µg/mL in methylene chloride, 1mL/ampul		
31839	31839-510	—
	w/data pack	
31839-500	31839-520	31939

Creosote Oil Standard

Each	5-pk.	10-pk.
50,000µg/mL in methylene chloride, 1mL/ampul		
31838	31838-510	—
	w/data pack	
31838-500	31838-520	31938

Other Mixes

Dimethyldichlorosilane (DMDCS)

Each	5-pk.
Neat, 20mL/ampul	
31840	31840-510

Drinking Water Odor Standard

(+/-)-geosmin 2-methylisoborneol

Each	5-pk.
100µg/mL in P&T methanol, 1mL/ampul	
30608	30608-510

Pinnacle™ DB Base-Deactivated HPLC Columns

Effective Replacements for Hypersil® BDS Columns

by Rebecca Wittrig, Ph.D., HPLC Product Marketing Manager,
and Vernon Bartlett, HPLC Innovations Team Manager

- Sharp, symmetric peaks for basic analytes.
- Excellent reproducibility, column-to-column and lot-to-lot.
- Chromatography equivalent to Hypersil® BDS material.

With Pinnacle™ DB base-deactivated silica, we have created an exceptional material for analyses of basic compounds, matching or exceeding the chromatographic performance of Hypersil® BDS material. Pinnacle™ DB silica and bonded phase packings are made through efficient processes that ensure reproducible performance and reliable stock levels—we ship 90% of all analytical columns ordered within 24 hours.

In Figure 1, peak symmetry for the basic compound pyridine is noticeably better on the Pinnacle™ DB C18 column than on a typical Type B C18 column, and the analysis time is shorter by more than 30%. You can expect similar results if you make your own comparisons. Figure 2 shows how closely matched the important physical characteristics of particle size and pore size distribution are for Pinnacle™ DB and Hypersil® BDS materials. Physical/chemical characteristics of Pinnacle™ DB packings are listed in Table I.

For certain physical parameters - most notably, metals content - we intentionally deviate from Hypersil® BDS material, as Figure 3 shows. The major difference in sodium content is especially important, as metal ions on the surface of silica particles negatively affect peak symmetry and otherwise interfere with chromatography, particularly for basic analytes. But, there is another important problem with silicas that have a high metal content, especially sodium—they are structurally inferior to silicas containing lesser amounts of metals. To strengthen silica particles containing high concentrations of sodium, some manufacturers use an annealing process to embed the sodium into the framework of the particles. Although annealing imparts strength to the particles, it destroys some of their useable surface area. And, as the particles age or crack, embedded ions are re-exposed. Deactivation for bases is lost, and stability in highly aqueous mobile phases erodes.

Our manufacturing process strips metals and other impurities from the silica surface. By removing metals, rather than covering them, we make a more rugged silica that does not need annealing to impart strength. Peak shapes for bases are more consistent as the column ages, and the potential lifetime for a Pinnacle™ DB column is longer. Also, large pores (140Å) make Pinnacle™ DB material an excellent choice when you want to shorten analysis time.

But - how do separations on Pinnacle™ DB columns and Hypersil® BDS columns compare? The chromatograms in Figures 4 and 5 are analyses of a base/neutral test mix and an acid/base test mix, respectively. Behavior of each of these analytes will vary, based on differences in carbon load, ligand density, degree of base deactivation, endcapping, and exposed metals. Columns that are similar for these characteristics should provide similar chromatography, and Figures 4 and 5 show Pinnacle™ DB C18 columns and Hypersil® BDS columns perform nearly identically.

If you are looking for rugged, high-quality columns for analyzing basic compounds, or as replacements for Hypersil® BDS columns, and you want them supplied quickly and reliably, Pinnacle™ DB columns are the ideal answer. In addition to the product quality you expect, you'll receive Plus 1™ service and prompt, expert technical help when you deal with Restek.

Table I

Physical/chemical characteristics of Pinnacle™ DB packings.

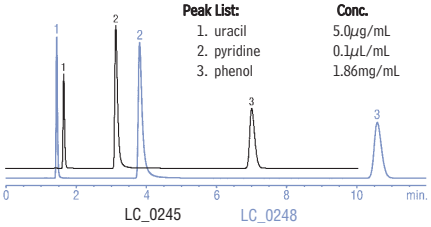
Particle:	5µm, spherical
Pore size:	140Å
Pore volume:	0.65mL/g
Carbon load:	C18—11%
	C8—6%
	Cyano—4%

Figure 1

Superior peak symmetry for pyridine, and a faster analysis, on a Pinnacle™ DB column.

Column:	**Pinnacle™ DB C18**	Conventional C18
Cat. #:	9414565	n/a
Dimensions:	150 x 4.6mm	150 x 4.6mm
Particle size:	5µm	5µm
Pore size:	140Å	100Å

Peak List:	**Conc.**
1. uracil	5.0µg/mL
2. pyridine	0.1µL/mL
3. phenol	1.86mg/mL



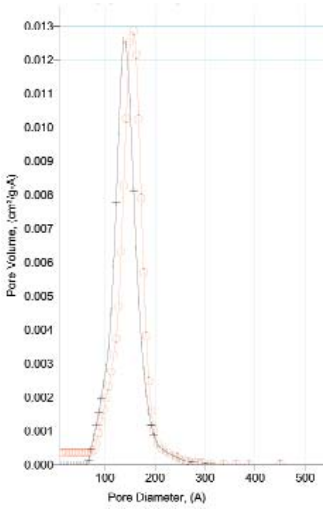
Sample:
Inj.: 5.0µL
Solvent: mobile phase

Conditions:
Mobile phase: 20mM potassium phosphate, pH 7.0: acetonitrile (80:20 v/v)
Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 254nm

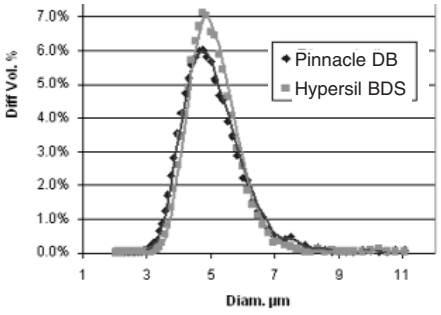
Figure 2

Physical characteristics of Pinnacle™ DB silica and Hypersil® BDS silica are remarkably similar.

Pore Size Distribution



Particle Size Distribution



Particle Size analysis	**Pinnacle DB**	**Hypersil BDS**
Geometric Mean (µm):	4.81±1.20	4.89±1.70
Arithmetic Mean (µm):	4.89±0.94	4.95±0.82
Median (µm):	4.69	4.81
Mode:	4.69	4.79
Max Diff. (%):	6.04	7.11

Figure 3

Low metals content in Pinnacle™ DB silica ensures sharper, more symmetric peaks for basic analytes.

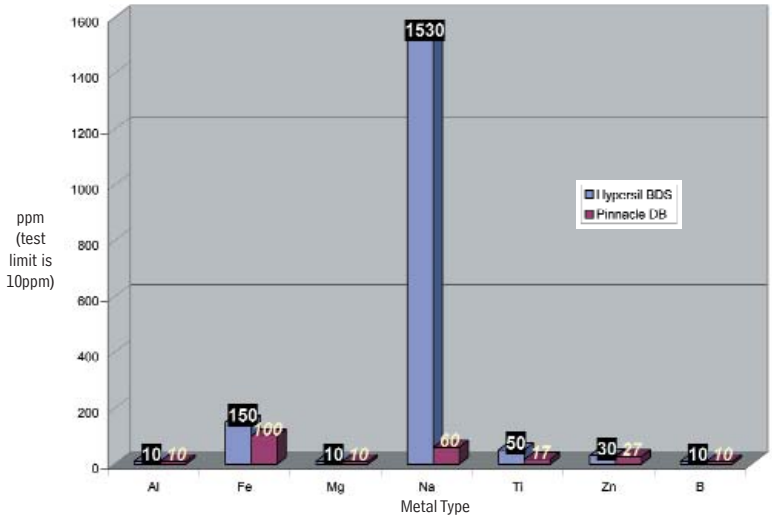
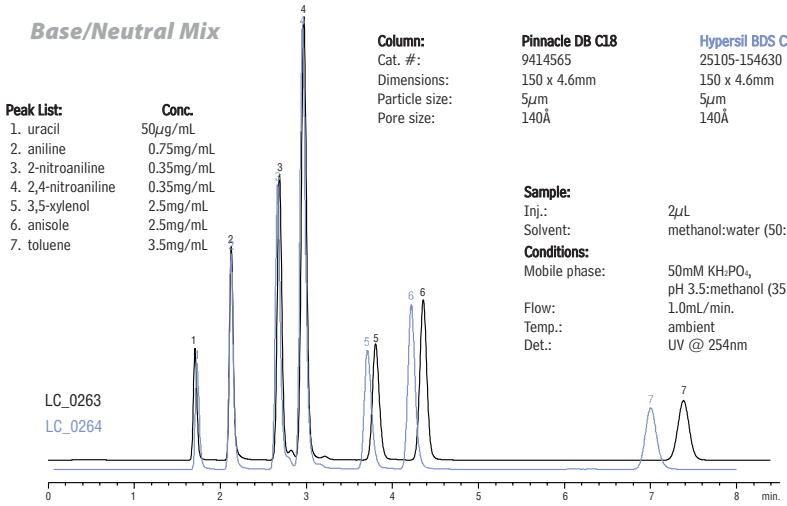


Figure 4

Pinnacle™ DB columns and Hypersil® BDS columns provide nearly identical retention, peak symmetry, and efficiency.



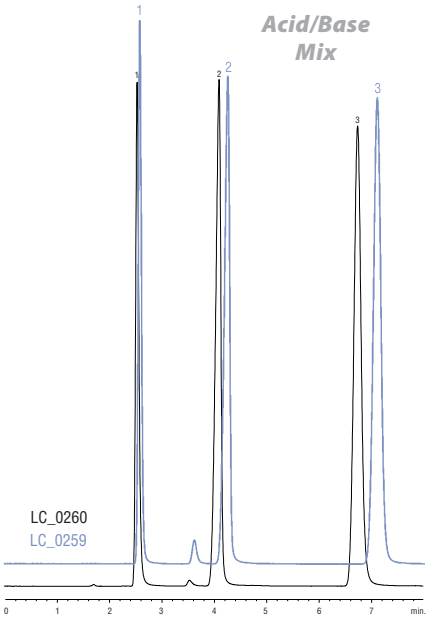
Column:	Pinnacle DB C18	Hypersil BDS C18
Cat. #:	9414565	25105-154630
Dimensions:	150 x 4.6mm	150 x 4.6mm
Particle size:	5µm	5µm
Pore size:	140Å	140Å

Peak List:	Conc.
1. uracil	50µg/mL
2. aniline	0.75mg/mL
3. 2-nitroaniline	0.35mg/mL
4. 2,4-nitroaniline	0.35mg/mL
5. 3,5-xylenol	2.5mg/mL
6. anisole	2.5mg/mL
7. toluene	3.5mg/mL

Sample:	
Inj.:	2µL
Solvent:	methanol:water (50:50 v/v)
Conditions:	
Mobile phase:	50mM KH_2PO_4, pH 3.5:methanol (35:65 v/v)
Flow:	1.0mL/min.
Temp.:	ambient
Det.:	UV @ 254nm

Figure 5

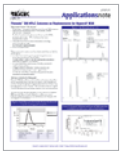
Pinnacle™ DB columns and Hypersil® BDS columns provide nearly identical retention, peak symmetry, and efficiency.



Column:	Pinnacle™ DB C18	Hypersil® BDS C18
Cat #:	9414565	25105-154630
Dimensions:	150 x 4.6mm	150 x 4.6mm
Particle size:	5µm	5µm
Pore size:	140Å	140Å

Peak List:	Conc.
1. caffeine	0.5mg/mL
2. acetylsalicylic acid	3.5mg/mL
3. benzoic acid	2.8mg/mL

Sample:	
Inj.:	2µL
Solvent:	methanol:water (50:50 v/v)
Conditions:	
Mobile phase:	5 mM KH_2PO_4, pH 3.5:methanol (40:60 v/v)
Flow:	1.0mL/min.
Temp.:	ambient
Det.:	UV @ 254nm



for more info

To see additional performance comparisons, request *Pinnacle™ DB HPLC Columns as Replacements for Hypersil® BDS* (lit. cat.# 59742).

Pinnacle™ DB Columns

The Pinnacle™ DB column line includes silica and C18, C8, and cyano bonded phases. Other phases and particle sizes are available on request.

Pinnacle™ DB C18 (USP L1), 5µm

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm	9414531	9414532	9414533	9414535
50mm	9414551	9414552	9414553	9414555
100mm	9414511	9414512	9414513	9414515
150mm	9414561	9414562	9414563	9414565
200mm	9414521	9414522	9414523	9414525
250mm	9414571	9414572	9414573	9414575

Pinnacle™ DB C8 (USP L7), 5µm

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm	9413531	9413532	9413533	9413535
50mm	9413551	9413552	9413553	9413555
100mm	9413511	9413512	9413513	9413515
150mm	9413561	9413562	9413563	9413565
200mm	9413521	9413522	9413523	9413525
250mm	9413571	9413572	9413573	9413575

Pinnacle™ DB Cyano (USP L10), 5µm

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm	9416531	9416532	9416533	9416535
50mm	9416551	9416552	9416553	9416555
100mm	9416511	9416512	9416513	9416515
150mm	9416561	9416562	9416563	9416565
200mm	9416521	9416522	9416523	9416525
250mm	9416571	9416572	9416573	9416575

Pinnacle™ DB Silica (USP L3), 5µm

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm	9410531	9410532	9410533	9410535
50mm	9410551	9410552	9410553	9410555
100mm	9410511	9410512	9410513	9410515
150mm	9410561	9410562	9410563	9410565
200mm	9410521	9410522	9410523	9410525
250mm	9410571	9410572	9410573	9410575

new!

HPLC Autosampler Syringes

Now Available from Restek—Convenient, One-Stop Shopping

by Donna Lidgett, GC Accessories Product Marketing Manager

- Made by leading syringe manufacturers.
- Designed and tested for reliable performance.
- Fast delivery.

These syringes join an extensive selection of GC and HPLC syringes available from Restek. To see other syringes, refer to our catalog, or visit our website.

SGE Syringes for Agilent 1090 & 1100 LC Autosamplers

- Termination: 1/4-32 UNEF thread

	SGE			Restek
Volume	Model	cat.#	qty.	cat.#
25µL	25D-HP1090-GT	003670	ea.	22290
250µL	250D-HP1090-GT	006670	ea.	22291

SGE Syringe for Hitachi LC Autosamplers

- Termination: M10x1 thread

	SGE			Restek
Volume	Model	cat.#	qty.	cat.#
500µL	500C-HITACH1	007660	ea.	22292

SGE Syringes for PerkinElmer LC Autosamplers

- Termination: 1/4-28 UNF thread

	SGE			Restek
Volume	Model	cat.#	qty.	cat.#
50µL	50D-CX-GT	004995	ea.	22295
100µL	100D-CX-GT	005990	ea.	22296
250µL	250D-CX-GT	006995	ea.	22297
500µL	500D-CX-GT	007995	ea.	22298
1mL	1MD-C-GT	008185	ea.	22299

SGE Syringes for Waters™ WISP® LC Autosamplers

- Termination: 1/4-28 UNF thread

	SGE			Restek
Volume	Model	cat.#	qty.	cat.#
25µL	25D-WISP	003990	ea.	22293
250µL	250D-WISP	006690	ea.	22294

Hamilton Syringes for Waters™ WISP® LC Autosamplers

- Teflon® PTFE-tipped plunger

	Hamilton			Restek
Volume	Model	cat.#	qty.	cat.#
25µL	1702	80020	ea.	24528
250µL	1725	80024	ea.	24529

free literature

Genuine Restek Replacement Parts for HPLC Systems

Defective parts can cause poor chromatography and shorten column lifetimes. Use Restek check valves, pump piston seals, detector lamps, and other parts to keep your Agilent, Beckman, Hitachi, PerkinElmer, Shimadzu, or Waters system running smoothly and chromatographically sharp. Genuine Restek Replacement Parts equal or exceed the performance of original equipment components.
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Gas-Tight Syringes for Rheodyne® & Valco® Valves

- Teflon®-tipped plungers.
- Removable needles: point style 3 (90° tip); 2"/51mm.
- Replaceable syringe barrels, plungers, and plunger tips.

Hamilton Syringes

10µL syringe: 26s needle
All others: 22s needle

	Hamilton		Restek
Volume	Model	cat.#	
10µL	1701	21260	
25µL	1702	21261	
50µL	1705	21262	
100µL	1710	21263	
250µL	1725	21264	

SGE Syringes

Needle gauge: 22

	SGE	Restek
Volume	Model	cat.#
10µL	10R-GT-LC	24866
25µL	25R-GT-LC	24867
50µL	50R-GT-LC	24868
100µL	100R-GT-LC	24869
250µL	250R-GT-LC	24870
500µL	500R-GT-LC	24871

For many more HPLC and GC syringes, request our catalog or visit our website.

Super-Clean™ Gas Trapping System for LC/MS

Quick-Change Cartridge System for Removing Hydrocarbon Impurities from Nitrogen

by Donna Lidgett, Purus™ Gas Systems Product Marketing Manager

- "Quick connect" fittings, for easy, leak-tight cartridge changes—no tools required to install the traps.
- Spring-loaded check valves—no loosening/tightening fittings each time you change a cartridge.
- No oxygen introduced during filter change-outs—decreases down time.
- Cartridges pre-purged with inert gas—saves time.
- Durable, patented full glass/metal design—combines safety and impermeability.

High-Purity Outlet Gas

Outlet Gas Purity %	Maximum Pressure	Estimated Filter Lifetime
99.9999%	11 bar/159psi	3 to 6 months

The Super-Clean™ Gas Trapping System is the latest technology in cartridge-style gas filtration for removing hydrocarbons from nitrogen, and is ideal for use in LC/MS systems. The cartridge-based system makes changing filters quick and easy. A two-position baseplate (1/4" fittings), installed in the gas line, allows cartridges to be exchanged without introducing oxygen into the system. Spring-loaded check valves seal when a cartridge is removed and open only when a new cartridge has been locked in place. There is no need for loosening and tightening fittings every time you change cartridges, and your system cannot become contaminated during the changing process.

To meet the high flow needs of the LC/MS system, the incoming gas stream is split equally between the two charcoal-filled cartridges, and the two streams are rejoined after purification but before the gas exits the base plate. This approach allows longer contact between the nitrogen and the adsorbent, ensuring higher gas purity and eliminating a potential source of contaminants to your analyses. The system delivers flows of up to 20L of hydrocarbon-free nitrogen per minute.

A handy date wheel, included with the system, indicates the cartridge installation date and the recommended replacement date.

20L of purified nitrogen per minute!

Super-Clean™ Gas Trapping System for LC/MS

Description	qty.	cat.#
Super-Clean™ Gas-Trapping System (2-position base plate, 2 charcoal filters)	ea.	22062
2-Position Base Plate	ea.	22060
Replacement Charcoal Filters	2-pk.	22061

Replacement O-Rings for Cartridge Baseplates

Pack includes 10 large O-rings and 10 small O-rings.

Description	qty.	cat.#
Replacement O-Rings for Cartridge Baseplates	20-pk.	22023

1/8-Inch to 1/4-Inch Tube-End Unions

To adapt the 1/8" baseplate fittings to 1/4", use these 1/8" to 1/4" tube-end unions.

Description	qty.	cat.#
1/8" to 1/4" Tube-End Unions	5-pk.	21833

For descriptions of Super-Clean™ systems for GC, request our catalog or visit our website.

free literature

HPLC Columns and Accessories

General-purpose and unique special-purpose columns, guard columns, column kits, and bulk materials, of course, but also instrument parts, innovative tools, accessories, and many example chromatograms. Our four silica bases have characteristics tailored to meet specific analytical requirements.
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FID Jets for Agilent Equipment

Performance Equivalent to Original Manufacturer's Parts

by Donna Lidgett, GC Accessories Product Marketing Manager

Standard Version:

- Fluted tip to guide the capillary column into the jet.
- Threads specially coated for easy installation and removal.
- Special processing ensures the highest degree of cleanliness.

High Performance Version:

- Similar to the standard version, but Siltek™ treated.
- Extremely inert—use with active compounds.

tech tip

Which FID Jet Should I Use?

There are two FID jet configurations for Agilent GCs. The longer "adaptable" jet fits both 5890 and 6890 GCs, and can be used with capillary or packed columns. The shorter "dedicated" jet is for the FID in the 6890 GC that is designed only for use with capillary columns.

Capillary Adaptable FID Replacement Jet for Agilent 5890/6890/6850 GCs (0.011-inch ID tip)

Description	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard	19244-80560	ea.	20670	3-pk.	20671
High-Performance Siltek™	19244-80560	ea.	20672	3-pk.	20673

Capillary Dedicated FID Replacement Jet for Agilent 6890/6850 GCs

Description	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard	G1531-80560	ea.	21621	3-pk.	21682
High-Performance Siltek™	G1531-80560	ea.	21620	3-pk.	21683

Packed Column Replacement Jets for Agilent 5890/6890/6850 GCs

0.018-Inch ID	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard	18710-20119	ea.	21694	3-pk.	21695
High-Performance Siltek™	18710-20119	ea.	21696	3-pk.	21697
0.030-Inch ID	**Similar to Agilent part #**	**qty.**	**cat.#**	**qty.**	**cat.#**
Standard	18789-80070	ea.	21688	3-pk.	21689
High-Performance Siltek™	18789-80070	ea.	21686	3-pk.	21687

FID Maintenance Kits for Agilent GCs

- Include the most common consumable supplies.
- All parts meet or exceed perfomance by instrument manufacturer's parts.
- Parts list makes reordering easy.

FID kits include: 1/4-inch, 0.4, 0.5, and 0.8mm ID graphite ferrules, FID/NPD capillary adaptor, capillary nuts, jet reamers/ferrule removers, 1/4-inch nut, scoring wafer, capillary column caps, ignitor for either Agilent 5890 or 6890/6850 GCs, FID flow measuring adaptor, 1/4- x 5/16-inch wrench, installation gauge, wire cleaning brush, high-performance Siltek™-treated FID jet for either Agilent 5890 or 6890/6850 GCs, 1/4-inch nut driver for jet removal.

Description	qty.	cat.#
FID Maintenance Kit for Agilent 5890 GCs	kit	21070
FID Maintenance Kit for Agilent 6890/6850 GCs	kit	21071

1/4-Inch Nut Driver

- Ideal for changing Agilent 5890/6890/6850 FID jets.
- Securely grips jet for easy removal or installation.

Description	qty.	cat.#
1/4-Inch Nut Driver	ea.	21076

FID Gauge Pack

Pressure regulators and gauges for air & hydrogen. The 1/8-inch bulkhead allows easy hookup to instrument. Rated for inlet pressures to 250psi, outlet pressures of 0 to 60psi.

Description	qty.	cat.#
FID Gauge Pack	ea.	20129

Cool Tools!

Restek Innovations Save You Time and Money

Inlet Liner Removal Tool

- Easily remove liner from injector—no more burned fingers.
- Made from high-temperature silicone.
- Won't chip or crack the liner.

No more burned fingers!

Gently push the liner removal tool onto the liner in the injection port, with a slight circular motion.

Slowly pull the liner out of the injection port.

Place a new liner in the injection port, avoiding hot metal surfaces.

Description	qty.	cat.#
Inlet Liner Removal Tool	3-pk.	20181

Scoring Wafer with Handle

- Unique, ergonomic handle is made of soft, comfortable rubber.
- Ceramic wafer cuts both fused silica and metal tubing cleanly.

Make clean, square cuts!

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

The tubing should fall off or break with a slight tap of the wafer.

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

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

Septum Puller

- Many uses—keep several on hand.
- Use hooked end for removing septa and O-rings; pointed end works well for removing stuck ferrules or fragments.

Easily remove septa and ferrules!

Remove septum without damaging an expensive weldment.

Dislodge a stuck ferrule quickly and easily—without scoring the fitting.

Description	qty.	cat.#
Septum Puller	ea.	20117

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- Helpful technical information.

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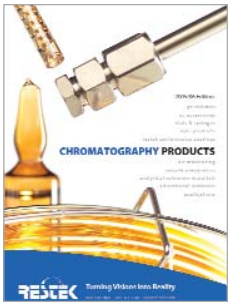
New in the 2005 Restek Catalog, with more coming……

GC Columns

Rtx®-440 - Low-bleed, high-resolution, intermediate-polarity column for many applications.
Rtx®-5Sil MS, 0.18mm ID - Monitor nanogram quantities of 78 semivolatile pollutants in 15 minutes.
Rtx®-Dioxin2 - Improved separation of dioxin or furan congeners, compared to diphenyl or high-cyano columns.



HPLC Columns

Allure™ Aqueous C18 - Excellent retention and selectivity for polar analytes in aqueous mobile phases.
Ultra Quat - Monitor paraquat and diquat without ion pairing reagents.

GC Accessories

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

LC Autosampler Syringes
Instrument parts - for Agilent, Beckman, Hitachi, PerkinElmer, Shimadzu, Thermo Separation Products, and Waters equipment.

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Siltek™, Sulfinert®, and Silcosteel®-CR treated Swagelok® fittings - Inert fittings for demanding applications.
Siltek™ and Silcosteel®-CR treated electropolished stainless steel tubing - For the most inert sample pathways available.

Air Monitoring

Improved SilcoCan™ and TO-Can™ air sampling canisters - Superior deactivation; superior protection for the valve.
Canister Heating Jacket - Uniformly heats entire canister, and valve, for faster more efficient cleaning.

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Calibration mixes, check mixes, surrogates, and internal standards for environmental analyses - volatile organics…semivolatile organics…pesticides & herbicides…UST monitoring
Dimethyldichlorosilane (DMDCS) deactivation reagent
Drinking water odor compounds

Look for your copy in January, or request one from your Restek representative!

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Please direct your comments on this publication to Carrie Sprout at carrie.sprout@restekcorp.com or call 814-353-1300, ext. 2151.

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

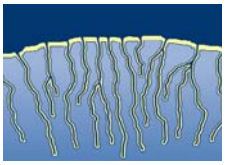
- 67% of available surface area can interact with proteins, peptides, other large molecules.
- Larger surface area than other commercially available 300Å materials.
- Manufactured by Restek, quality controlled by Restek.

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 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, 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.

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



in this issue

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

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

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 porositometry, BET measurements[1], and BJH calculation[2] 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 represented 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.

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.)

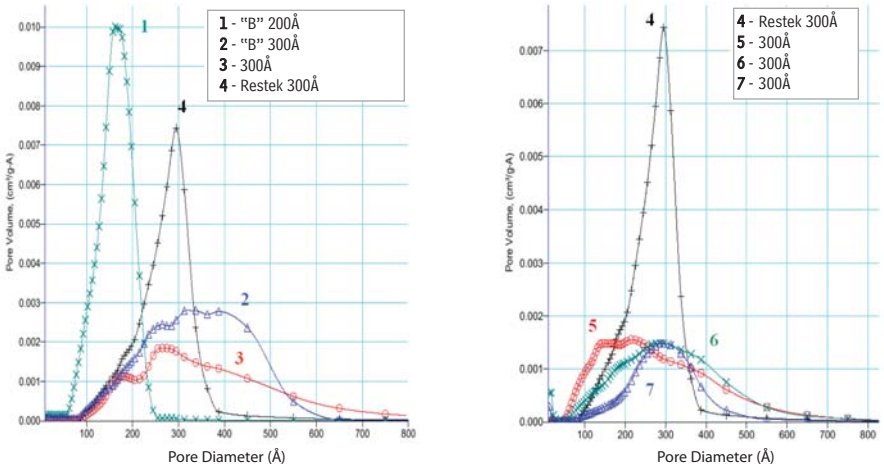
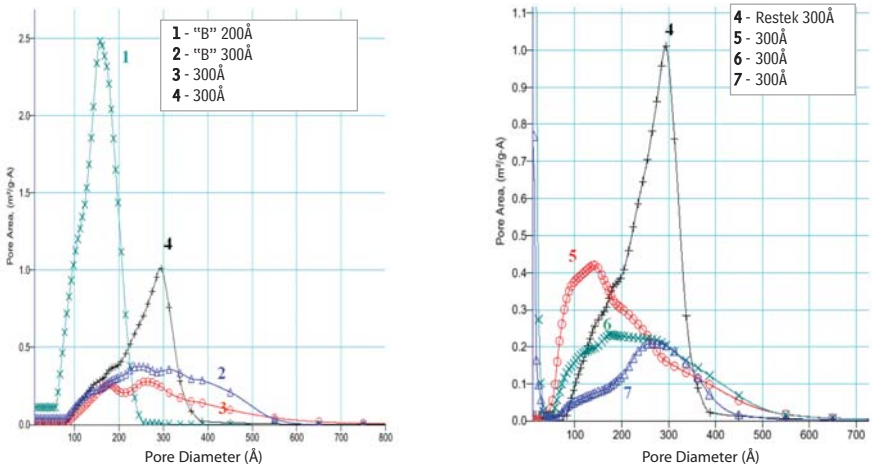


Figure 3 Pore area vs pore diameter for commercial wide pore silicas (BJH desorption). Viva™ silica shows a highly desirable distribution. (Change in scale for plots at right.)



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.

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.

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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).

ordering note

For guard columns for Viva™ wide pore columns, refer to our current catalog, or visit our website.

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

Example:
100mm x 4.6mm ID Viva™ Wide Pore C18 column with Trident™ Integral Inlet Fitting: 9514515-700

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.
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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.

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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

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.

new!

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

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

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 Dimandja[1], 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.

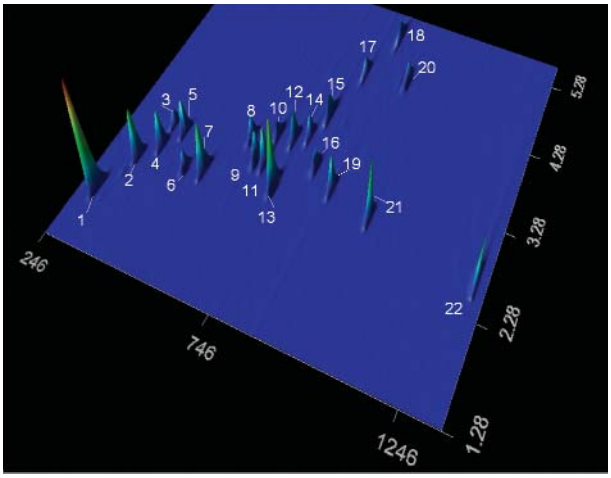
In determining which column pair to use for a GCxGC application, it is important to choose stationary phases that differ in selectivity. For this application, we choose an Rtx®-5 column for a volatility-based separation, in series with an Rtx®-200 column which is selective for halogenated compounds. 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.

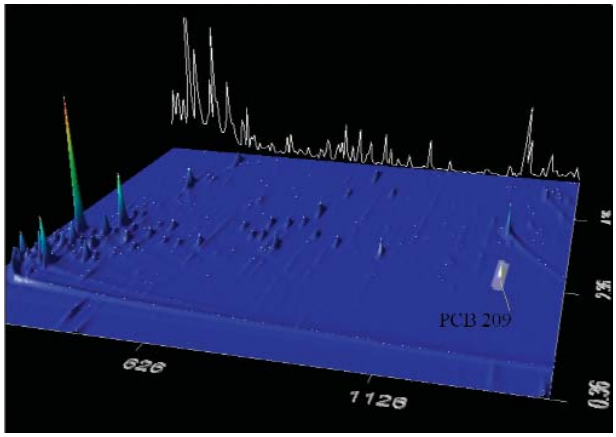
Figure 1 GCxGC analysis of organochlorine pesticides combines primary column and confirmation column results.



Columns: Rtx®-5 9m, 0.18mm ID, 0.20µm (10m column, cat.# 40201, with 1m removed)
Rtx®-200 1m, 0.18mm ID, 0.20µm (1m of 10m column, cat.# 45001)
Inj.: 1µL, split, 250°C, split ratio 50:1
Oven: Primary: 50°C (0.2 min.), 30°C/min. to 140° (no hold), 5°C/min. to 250°C (no hold)
Secondary: 50°C offset from primary oven
Instrument: LECO GCxGC/ECD
Modulator: Temperature offset: 30°C
Modulation time: 6 sec
Det.: ECD, 325°C, 150mL/min. nitrogen makeup gas, 50Hz

Peak identifications listed in Table I and Table II.

Figure 2 Organochlorine pesticides separated from interferences in tomato extract.



Conditions: see Figure 1

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-*m*-xylene	294	1.9
2. α-BHC	342	2.44
3. ß-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	Sample Amount Quantified	Spike Sample Amount Quantified	Spike Amount Theoretical	Recovery (%)
1. tetrachloro-*m*-xylene	0	14.4	16	90
2. α-BHC	0	7.8	8	98
3. β-BHC	0	7.1	8	89
4. γ-BHC	0	6.8	8	85
5. δ-BHC	0	6.5	8	81
6. heptachlor	0	9.3	8	116
7. aldrin	0	7.2	8	90
8. heptachlor epoxide	0	9.3	8	116
9. γ-chlordane	0	6.9	8	86
10. endosulfan I	9.5	18.7	8	115
11. α-chlordane	0	7.5	8	94
12. dieldrin	0	17.4	16	109
13. 4,4'-DDE	0	16.8	16	105
14. endrin	0	14.2	16	89
15. endosulfan II	15.4	27.9	16	78
16. 4,4'-DDD	0	13.9	16	87
17. endrin aldehyde	0	12.8	16	80
18. endosulfan sulfate	13.1	28.2	16	94
19. 4,4'-DDT	0	15.7	16	98
20. endrin ketone	0	13	16	81
21. methoxychlor	0	76.8	80	96
22. decachlorobiphenyl	0	16.2	16	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
a-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
- GC/MS
- Environmental GC
- HPLC Method Development (2 days)
- GC Hands-On Maintenance and Troubleshooting
- Foods, Flavors, and Fragrances

How can I quickly choose the correct column for my application?

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

How does mobile phase composition influence retention in HPLC analysis?

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

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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.
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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 sulfide, 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.

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.

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

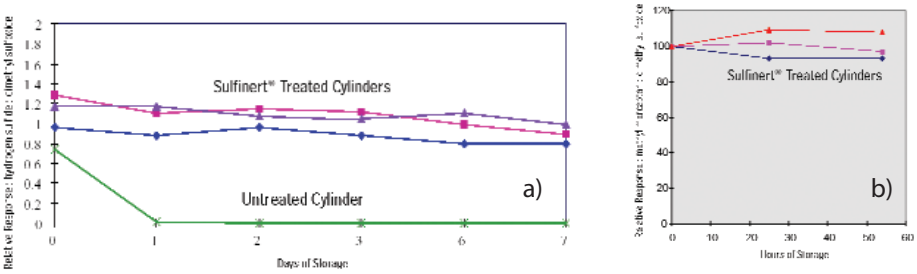


Figure 2 Sulfinert® treated electropolished seamless stainless steel tubing (red) does not adsorb methyl mercaptan (500ppbv). Blue-untreated electropolished tubing; violet-raw tubing.

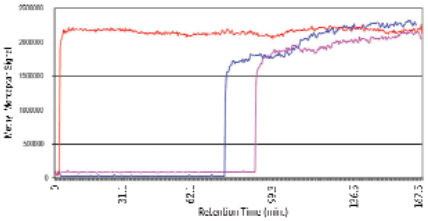
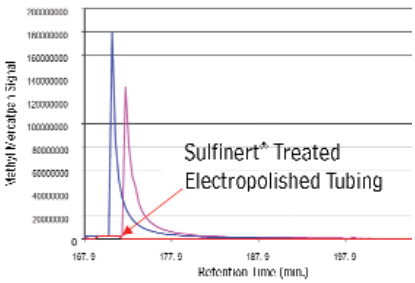


Figure 3 Sulfur memory is prolonged in raw commercial grade stainless steel tubing (violet). Red-Sulfinert® treated electropolished tubing; blue-untreated electropolished tubing. (500ppbv methyl mercaptan in helium)



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 loose $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				

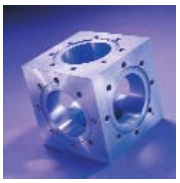
1/8" OD: 5 ft. to 100 ft. in one continuous coil; 1/4" OD: 5 ft. to 300 ft. in one continuous coil. Longer lengths will be more than one coil.
Note: (required length in meters) x (3.2808) = length in feet.
** 0.035" wall thickness.

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



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.

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).

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, 0.18μm ID, 0.18μm 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 minutes, 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, theTOFMS system offers sensitivity sufficient to allow calibration beyond the 20 to 160ng/μL "normal" calibration range, to a range of 0.2 to 160ng/μL, 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

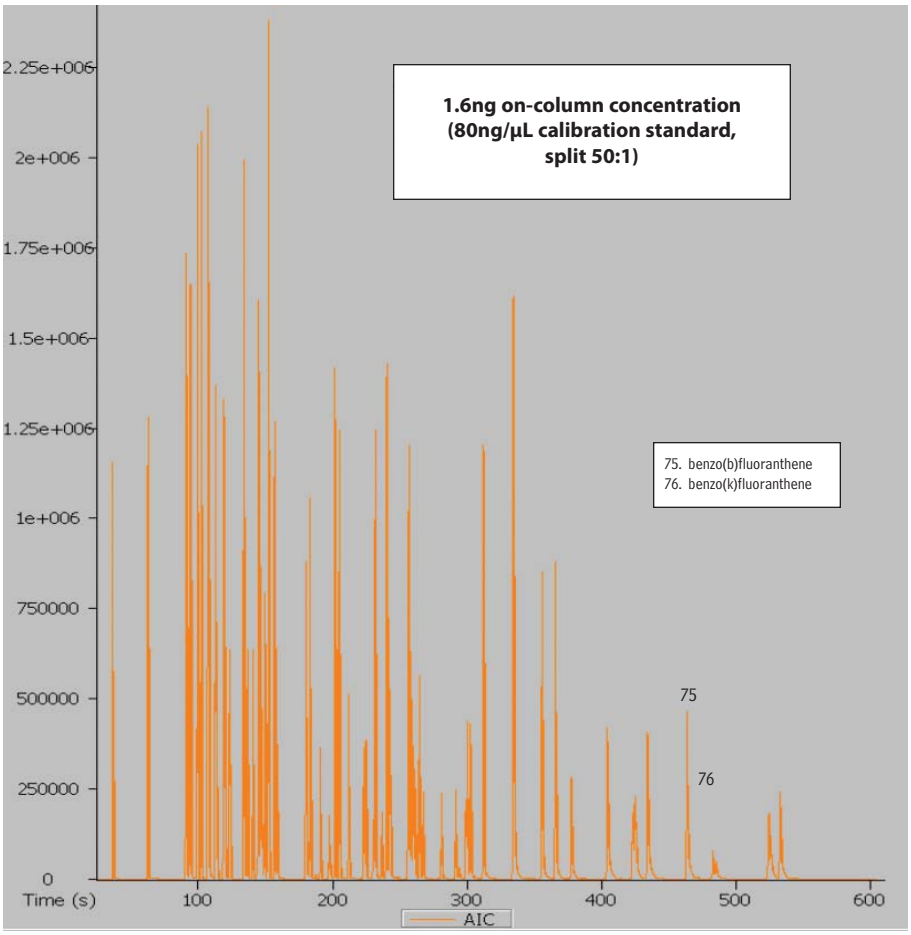
1. Improved Sensitivity and Analysis Time for Semivolatile Organic Compounds, Using GC-TOFMS: Can this Analysis Really be Performed in Less Than 10 Minutes? Frank L. Dorman, Jack W. Cochran (LECO Corporation), Gary B. Stidsen, Chris M. English, Michael S. Wittrig
PittCon 2005, Monday, Feb. 28. Oral Session 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-trrichlorophenol	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®-5SilMS, 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.:	1uL, split, 4mm Siltek® treated inlet liner with Siltek® treated wool, 250ºC, split ratio 50:1, 25 sec. solvent delay
Carrier gas:	helium
Flow rate:	2mL/min., constant flow
Oven:	40ºC (0.1 min.) to 340ºC @ 30ºC/min. (no hold)
Mass spectrometer:	LECO Pegasus 3 ToF-MS
Source temp.:	250ºC
Electron ionization:	70 eV
Stored mass range:	35 - 500 u
Acquisition rate:	20 spectra/sec.
Total run time:	10 min.

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

[1]1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).
[2]N-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

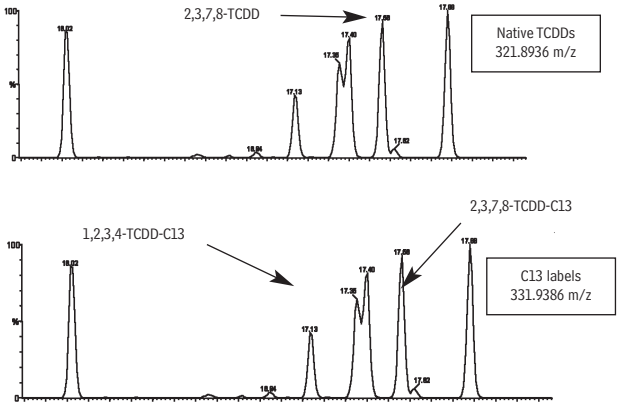
by Gary Stidsen, GC Columns Marketing Manager

- Resolves all 2,3,7,8-substituted dioxinsfrom 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 320ºC.

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.

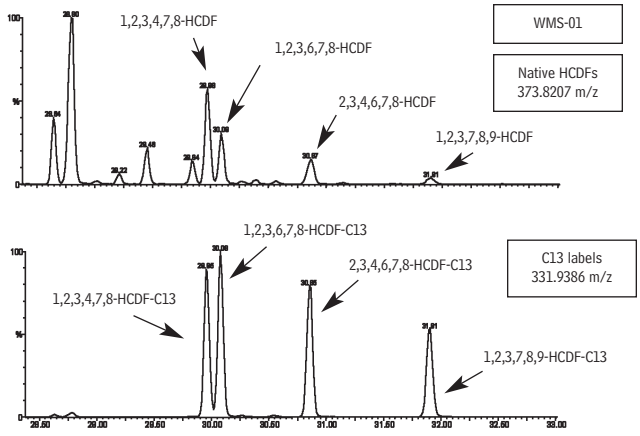


Column and Conditions for Figures 1 and 2:
Column: 60m, 0.25mm ID, 0.25µm Rtx®-Dioxin2 (cat.# 10758)

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

restek innovation!

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.

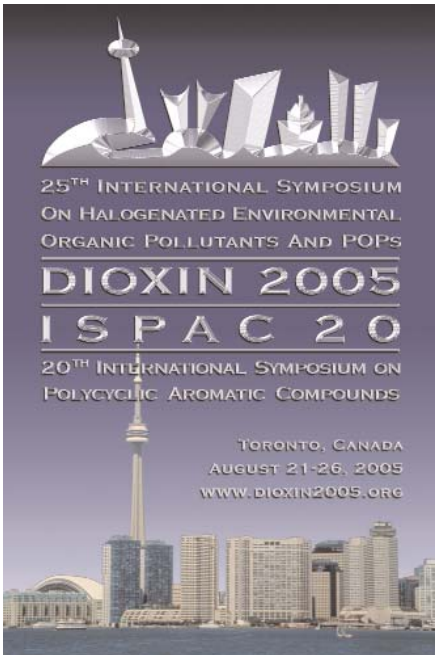
Coelutions of toxic and non-toxic congeners also can make quantification of the hexachlorodibenzofurans difficult, but an Rtx®-Dioxin2 column resolves furan congeners as effectively as dioxins. Figure 2, a chromatogram for the HCDF congener group in reference material WMS-01, shows the congeners are 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

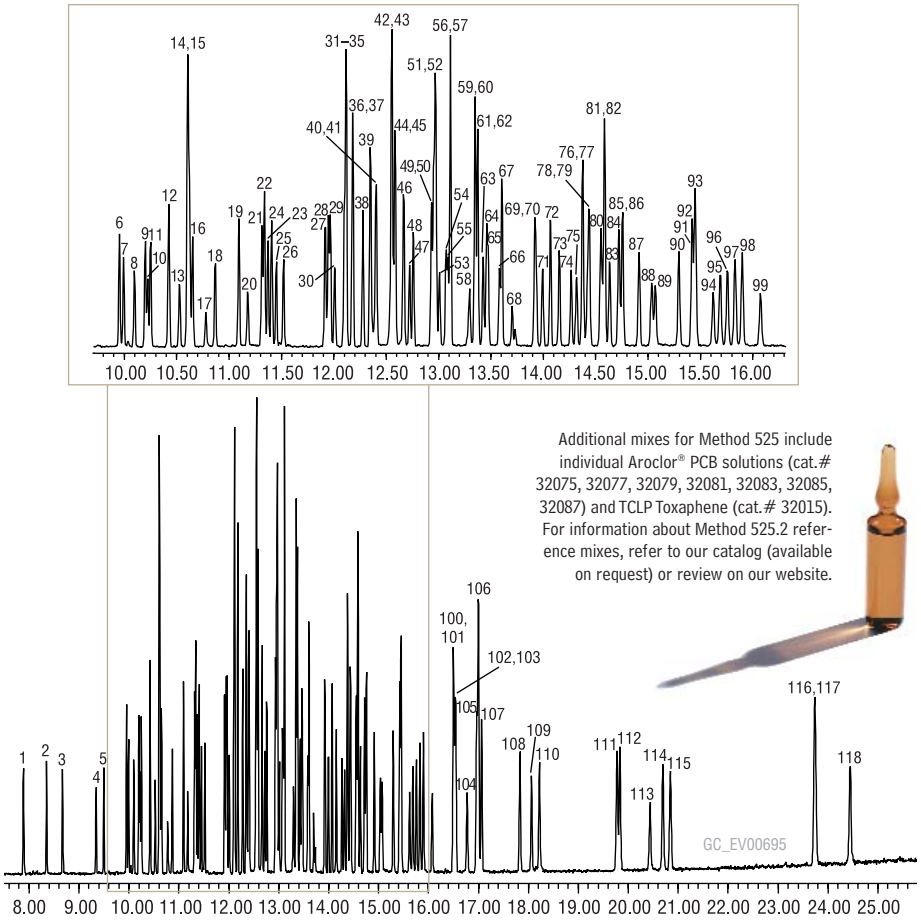
For Maximum Performance from High-Sensitivity GC/MS Systems

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 360ºC; 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 360ºC. 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.



Additional mixes for Method 525 include individual Aroclor® PCB solutions (cat.# 32075, 32077, 32079, 32081, 32083, 32085, 32087) and TCLP Toxaphene (cat.# 32015). For information about Method 525.2 reference mixes, refer to our catalog (available on request) or review on our website.

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 340ºC), 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-trichlorobipenyl (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. tricyclazole
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

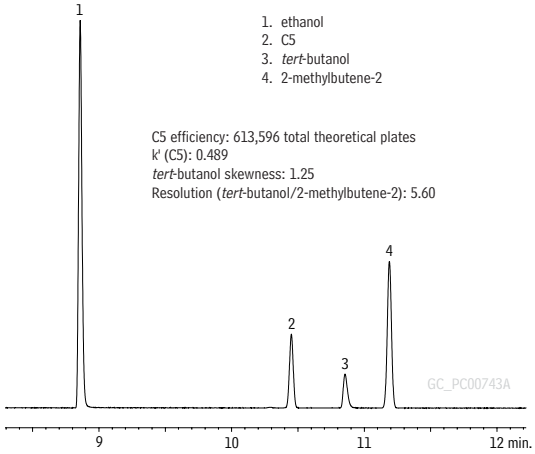
by Gary Stidsen, GC Columns Marketing Manager, and Barry Burger, Petroleum Applications Chemist

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

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



1. ethanol
2. C5
3. *tert*-butanol
4. 2-methylbutene-2

C5 efficiency: 613,596 total theoretical plates
k' (C5): 0.489
tert-butanol skewness: 1.25
Resolution (*tert*-butanol/2-methylbutene-2): 5.60

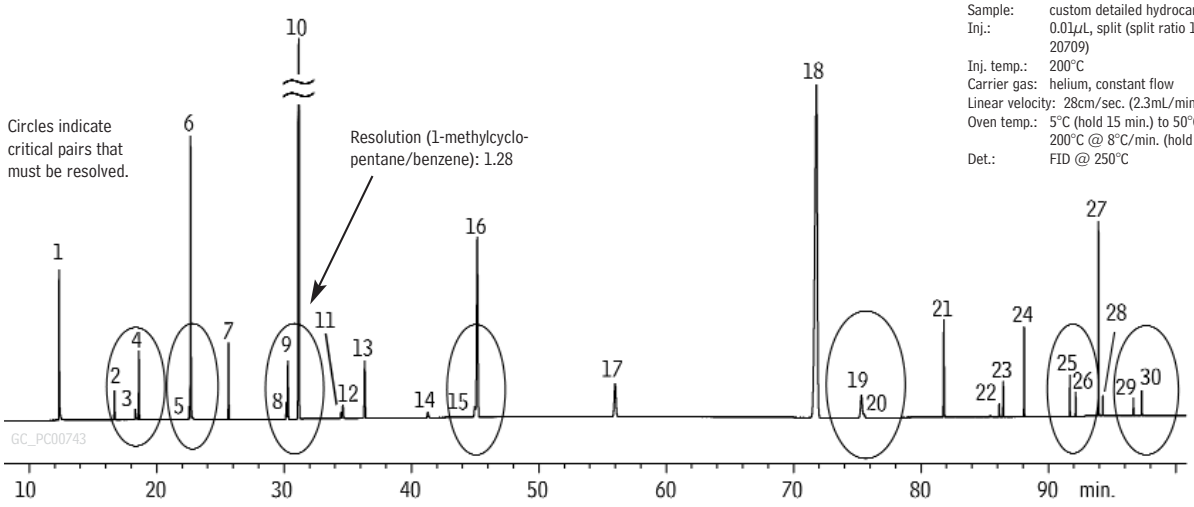
Rtx®-1PONA, 100m, 0.25mm ID, 0.5µm (cat.# 10195) w/ Rtx®-5 tuning column, 2.62m, 0.25mm ID, 1.0µm, connected via Press-Tight® connector (cat.# 20446)

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.

Figure 2 Critical pairs of gasoline components resolved per ASTM specifications, 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.:	5°C (hold 15 min.) to 50°C @ 5°C/min. (hold 50 min.) to 200°C @ 8°C/min. (hold 10 min.)
Det.:	FID @ 250°C

1. ethanol
2. C5
3. tert-butanol
4. 2-methylbutene-2
5. 2,3-dimethylbutane
6. methyl tert-butyl ether (MTBE)
7. C6
8. 1-methylcyclopentene
9. benzene
10. cyclohexane
11. 3-ethylpentane
12. 1-tert-2-dimethylcyclopentane
13. C7
14. 2,2,3-trimethylpentane
15. 2,3,3-trimethylpentane
16. toluene
17. C8
18. ethylbenzene
19. p-xylene
20. 2,3-dimethylheptane
21. C9
22. 5-methylnonane
23. 1,2-methylethylbenzene
24. C10
25. C11
26. 1,2,3,5-tetramethylbenzene
27. naphthalene
28. C12
29. 1-methylnaphthalene
30. C13

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.

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

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

Exclusive Polymer with Unique Selectivity

by Gary Stidsen, GC Columns Marketing Manager

new!
unique polymer
technology

- 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 340ºC.

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 discovered 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 I 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
	193	7	22.23	0.6	not be terribly biased by 193.
	200	8	22.30	1.1	
	191	7	22.37	1.1	

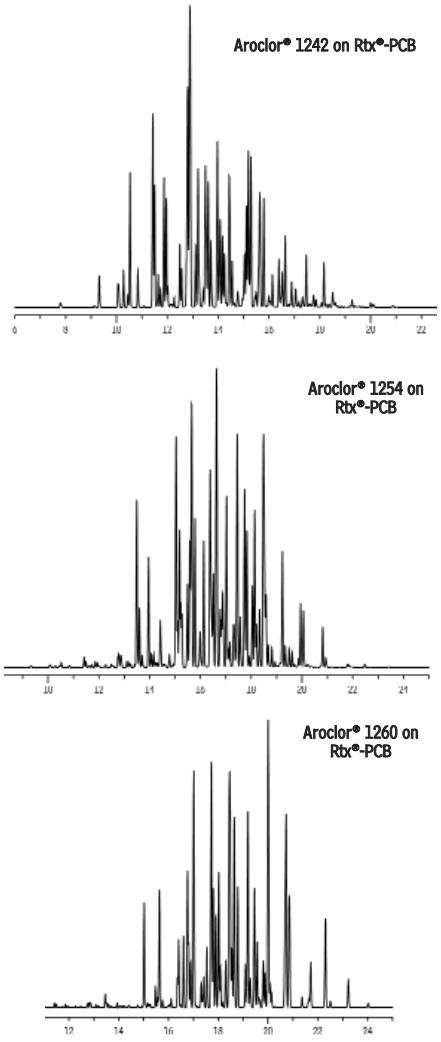
Mix of Aroclor® 1242-1254-1262 used for resolution check.
Relaxed resolution criteria based on visual inspection of closely eluting congeners.

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	

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 320ºC), 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.:	250ºC
Carrier gas:	hydrogen, constant pressure
Linear velocity:	71cm/sec. @ 110ºC
Oven temp.:	100ºC (hold 1.0 min.) to 300ºC @ 10ºC/min. (hold 4 min.)
Det.:	ECD @ 310ºC

Fast GC/MS Analysis of Semivolatile Organic Compounds

Using a 0.18mm ID / 0.36μm 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.

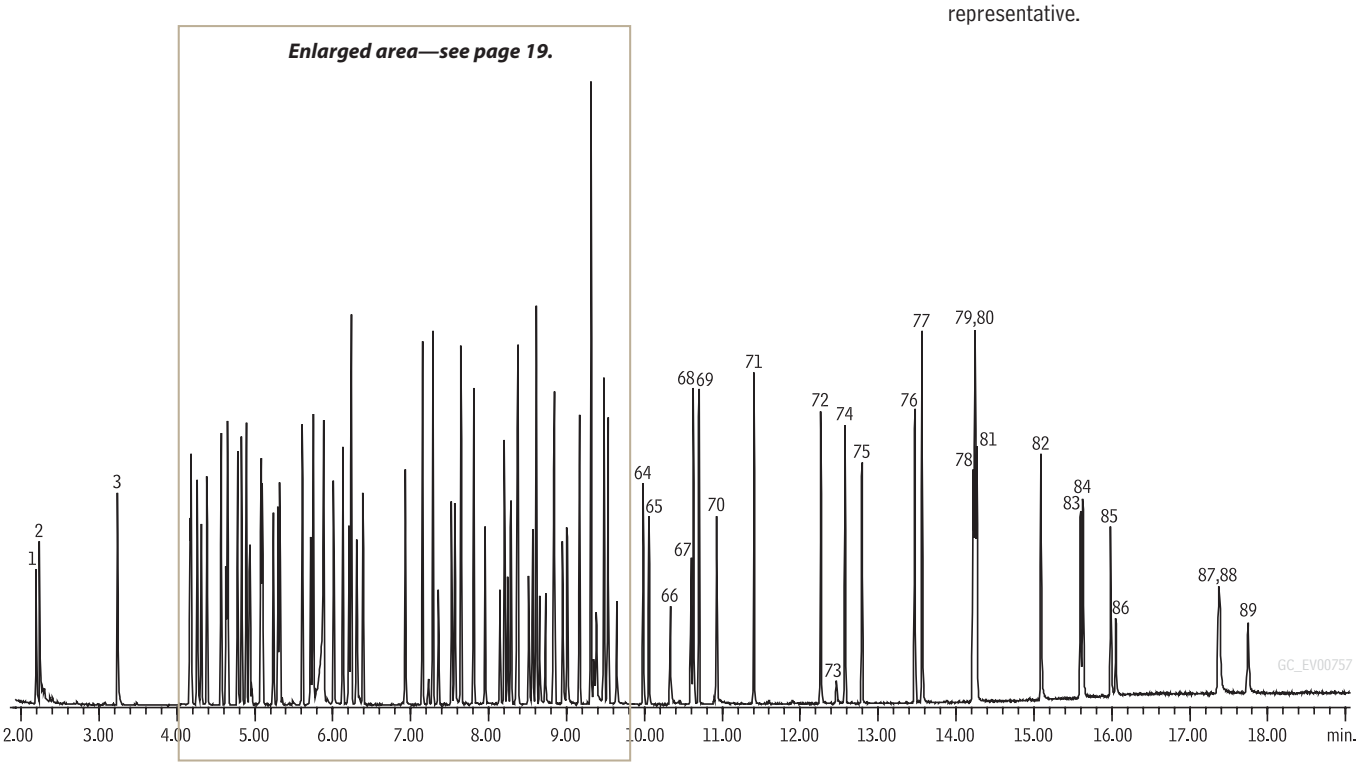
The new generation of high sensitivity mass spectrometers enables analysts to reduce the amount of sample injected onto the column. In analyses of semivolatiles, this has lead to using split injections and columns having smaller internal diameter, resulting in shorter analysis times. Split injection 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, 0.36μm 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, 0.36μm 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, 0.36µm film Rtx®-5Sil MS column.



1. N-nitrosodimethylamine
2. pyridine
3. 2-fluorophenol

peaks 4-63: see page 19

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

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

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 340ºC—excellent for trace analysis by GC/MS.

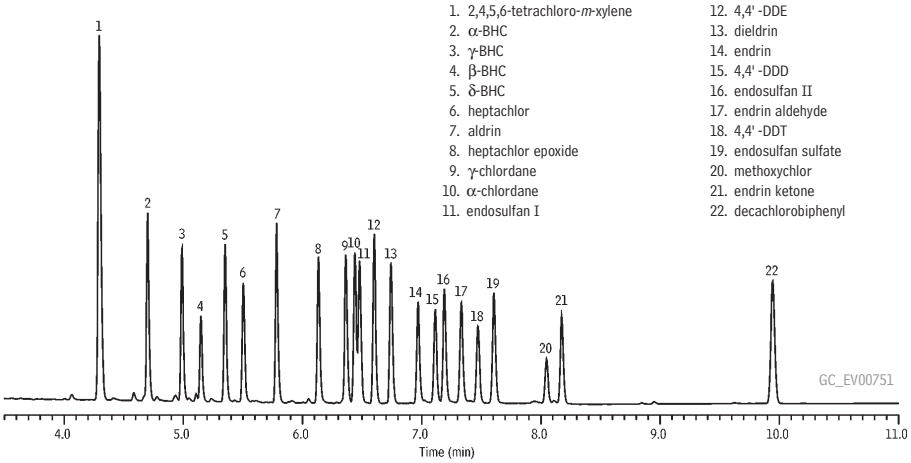
new!

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 minutes (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.

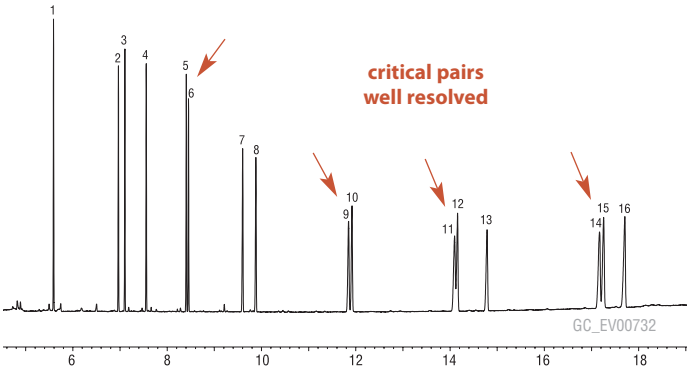
Our chemists are continuing their applications research with this new phase. Please call our Technical Service team to obtain the latest information about Rtx®-440 columns.

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



Column:	Rtx®-440 30m, 0.32mm ID, 0.50μm (cat.# 12939)
Sample:	50ng/mL Organochlorine Pesticides Mix AB standard (cat#32291) & 50ng/mL Pesticide Surrogate Mix (cat#32000) in hexane
Inj.:	1.0μL 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.



1. naphthalene
2. acenaphthylene
3. acenaphthene
4. fluorene
5. phenanthrene
6. anthracene
7. fluoranthene
8. pyrene
9. benzo(a)anthracene
10. chrysene
11. benzo(b)fluoranthene
12. benzo(k)fluoranthene
13. benzo(a)pyrene
14. indeno(1,2,3-cd)pyrene
15. dibenzo(a,h)anthracene
16. benzo(ghi)perylene

Column:	Rtx®-440 30m, 0.25mm ID, 0.25μm (cat.# 12923)
Sample:	610 PAH Mix (cat.# 31011) diluted to 20ppm each compound in methylene chloride
Inj.:	1.0μL 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 (μm)	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
α-BHC
β-BHC
δ-BHC
γ-BHC (lindane)
α-chlordane
γ-chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
dieldrin
endosulfan I
endosulfan II
endosulfan sulfate
endrin
endrin aldehyde
endrin ketone
heptachlor
heptachlor epoxide (B)
methoxychlor

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

Pesticide Surrogate Mix

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

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

SV Calibration Mix #5 / 610 PAH Mix

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

2,000μg/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 if chloral hydrate is not being analyzed. Qualitative confirmation of the target compounds is required by GC/MS analysis or by GC on two dissimilar columns. The listed primary analytical column is a bonded methyl polysiloxane stationary phase Restek column, Rtx®-1 (30m, 0.25mm ID, 1.0μm film, cat.# 10153); the listed confirmation column is a bonded 6% 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 should be stored away from heat, because heating chloral hydrate with alkali produces chloroform. Note that chloral hydrate is a hypnotic depressant included in Schedule IV of the Controlled Substance Act. We have the required license and exception approval to offer chloral hydrate as a reference material.

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.

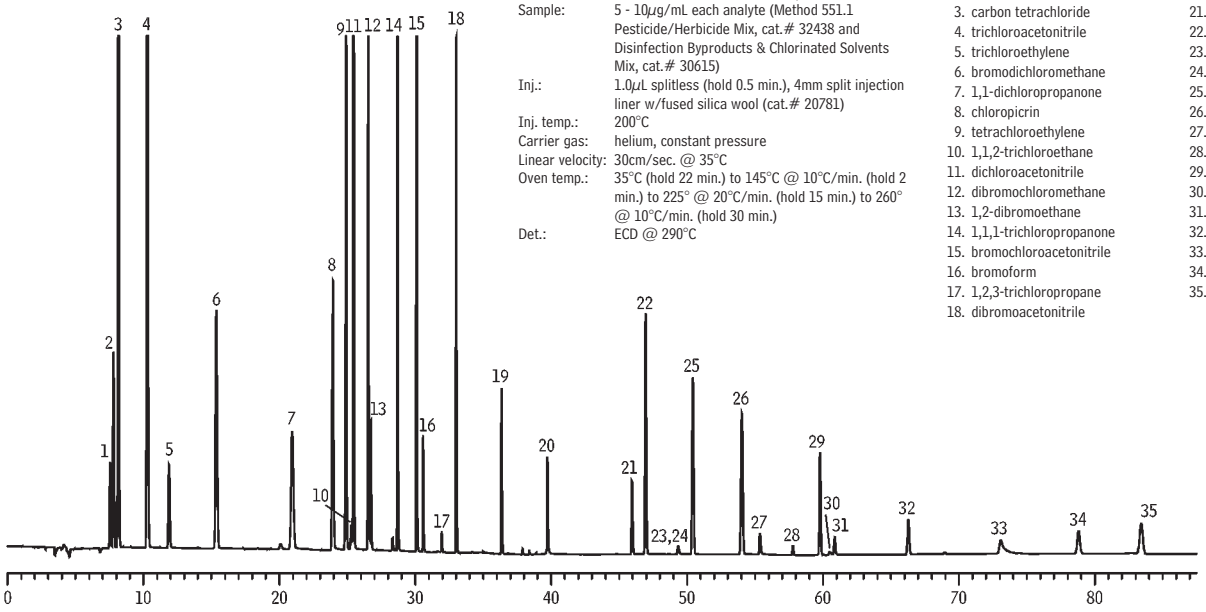
We offer an internal standard, bromofluorobenzene, and a surrogate standard, decafluorobiphenyl, in acetone, per method recommendations. The method recommends the use of a laboratory performance check (LPC) solution in MTBE, the extraction solvent. The check solution is a mix of method analytes used 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.

References

1. *US Environmental Protection Agency National Interim Primary Drinking Water Regulations: Control of Trihalomethanes in Drinking Water, Final Rule* Fed. Reg. 44 (231): 68624 (1979).
2. Yue Feng Xie, *Disinfection By-Product Analysis in Drinking Water* American Laboratory, Nov. 2000, p. 50.

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



Method 551.1 Pesticide/Herbicide Mix

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

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

Disinfection by-Product and Chlorinated Solvents Mix

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

2000µg/mL each in acetone, 1mL/ampul
cat. # 30616 (ea.)

Method 551.1 MTBE Lab Performance Check Mix

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

In methyl *tert*-butyl ether, 1mL/ampul
cat. # 32440 (ea.)

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)

(Crossbond® 100% dimethyl polysiloxane)
Temp. limits: -60 to 320/340°C

ID	df (µm)	length	cat. #
0.25mm	1.00	30-Meter	10153

Rtx®-1301 Column (fused silica)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)
Temp. limits: -20 to 260°C

ID	df (µm)	length	cat. #
0.25mm	1.00	30-Meter	16053

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 meeting 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).

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

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.

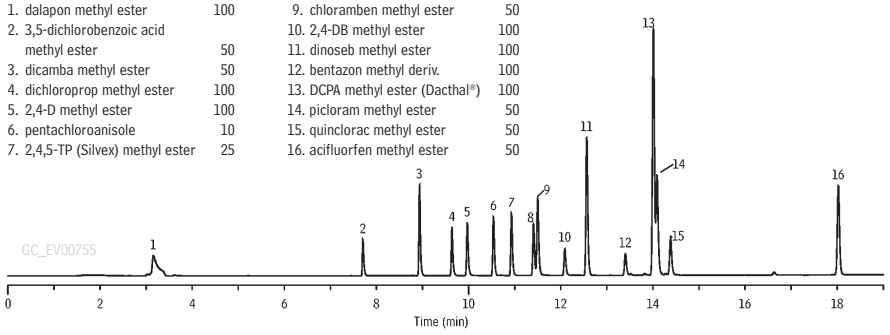
When monitoring these methylated esters by GC/ECD two columns are needed, to provide identification and confirmation. Further, it is important to select stationary phases that have low bleed and high thermal stability, because the columns should be heated between analyses to drive off any retained materials. The primary column chosen for this analysis is a 30m, 0.32mm ID, 0.25µm 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 340ºC. Figure 2 shows an analysis of methylated chlorophenoxyacid herbicides on a 30m, 0.32mm ID, 0.25µm Rtx®-440 column. Resolution is good, and the column exhibits very low bleed at 340ºC. 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.

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.

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

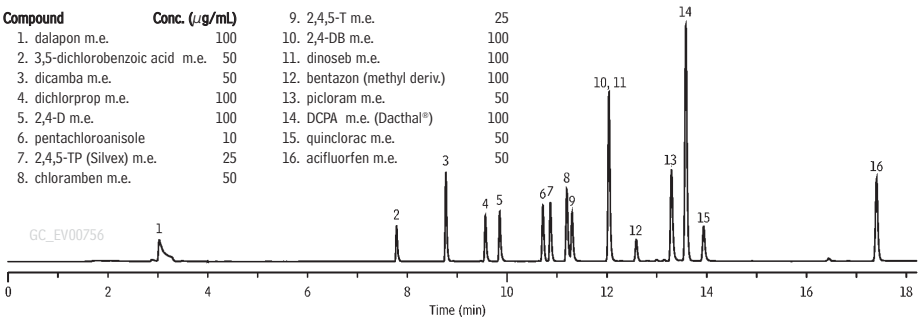
Compound	Conc. (µg/mL)
1. dalapon methyl ester	100
2. 3,5-dichlorobenzoic acid methyl ester	50
3. dicamba methyl ester	50
4. dichloroprop methyl ester	100
5. 2,4-D methyl ester	100
6. pentachloroanisole	10
7. 2,4,5-TP (Silvex) methyl ester	25
8. 2,4,5-T methyl ester	25
9. chloramben methyl ester	50
10. 2,4-DB methyl ester	100
11. dinoseb methyl ester	100
12. bentazon methyl deriv.	100
13. DCPA methyl ester (Dacthal®)	100
14. picloram methyl ester	50
15. quinclorac methyl ester	50
16. acifluorfen methyl ester	50



Column:	Rtx-CLPesticides2® 30m, 0.32mm ID, 0.25µm (cat.# 11324)
Sample:	10-100µg/mL each methyl ester in MTBE (cat.# 32444)
Inj.:	1.0µL splitless (hold 0.45 min.), 4mm Siltek® double gooseneck splitless liner (cat.# 20784)
Inj. temp.:	225°C
Carrier gas:	helium, constant pressure
Inlet pressure:	10.5psi set @ 70°C
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

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.

Compound	Conc. (µg/mL)
1. dalapon m.e.	100
2. 3,5-dichlorobenzoic acid m.e.	50
3. dicamba m.e.	50
4. dichlorprop m.e.	100
5. 2,4-D m.e.	100
6. pentachloroanisole	10
7. 2,4,5-TP (Silvex) m.e.	25
8. chloramben m.e.	50
9. 2,4,5-T m.e.	25
10. 2,4-DB m.e.	100
11. dinoseb m.e.	100
12. bentazon (methyl deriv.)	100
13. picloram m.e.	50
14. DCPA m.e. (Dacthal®)	100
15. quinclorac m.e.	50
16. acifluorfen m.e.	50



Column:	Rtx®-440 30m, 0.32mm ID, 0.25µm (cat.# 12924)
Sample:	10-100µg/mL each methyl ester in MTBE (cat.# 32444)
Inj.:	1.0µL splitless (hold 0.45 min.), 4mm Siltek® double gooseneck splitless liner (cat.# 20784)
Inj. temp.:	225°C
Carrier gas:	helium, constant pressure
Inlet pressure:	10.5psi set @ 70°C
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

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
DCPA 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 ether	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
DCPA 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-dibromooctafluorobiphenyl

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

515.4 Surrogate Mix

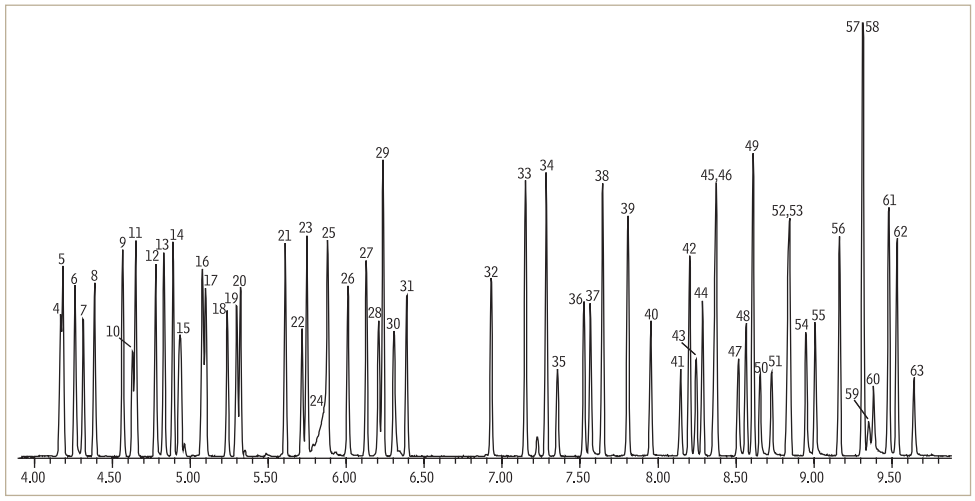
2,4-dichlorophenylacetic acid

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
5. phenol
6. aniline
7. bis(2-chloroethyl)ether
8. 2-chlorophenol
9. 1,3-dichlorobenzene
10. 1,4-dichlorobenzene-d4
11. 1,4-dichlorobenzene
12. benzyl alcohol
13. 1,2-dichlorobenzene
14. 2-methylphenol
15. bis(2-chloroisopropyl)ether
16a. 4-methylphenol
16b. 3-methylphenol
17. N-nitroso-di-*n*-propylamine
18. hexachloroethane
19. nitrobenzene-d5
20. nitrobenzene
21. isophorone
22. 2-nitrophenol
23. 2,4-dimethylphenol
24. benzoic acid
25. bis(2-chloroethoxy)methane
26. 2,4-dichlorophenol
27. 1,2,4-trichlorobenzene
28. naphthalene-d8
29. naphthalene
30. 4-chloroaniline
31. hexachlorobutadiene
32. 4-chloro-3-methylphenol
33. 2-methylnaphthalene
34. 1-methylnaphthalene
35. hexachlorocyclopentadiene
36. 2,4,6-trichlorophenol
37. 2,4,5-trichlorophenol
38. 2-fluorobiphenyl
39. 2-chloronaphthalene
40. 2-nitroaniline
41. 1,4-dinitrobenzene
42. dimethylphthalate
43. 1,3-dinitrobenzene
44. 2,6-dinitrotoluene
45. 1,2-dinitrobenzene
46. acenaphthylene
47. 3-nitroaniline
48. acenaphthene-d10
49. acenaphthene
50. 2,4-dinitrophenol
51. 4-nitrophenol
52. 2,4-dinitrotoluene
53. dibenzofuran
54. 2,3,4,6-tetrachlorophenol
55. 2,3,5,6-tetrachlorophenol
56. diethyl phthalate
57. fluorene
58. 4-chlorophenyl phenyl ether
59. 4-nitroaniline
60. 4,6-dinitro-2-methylphenol
61. diphenylamine
62. azobenzene
63. 2,4,6-tribromophenol

Searching for a chromatogram?
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Instrument Innovations!

Simplify Life in Your Laboratory

by Donna Lidgett, GC Accessories Product Marketing Manager

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new!

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

Liner for Varian 1177 GCs

new!

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

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.
Drilled Uniliner® (hole on top)	4.0 ID 6.3 OD x 78.5	21054	21055
Siltek® Drilled Uniliner® (hole on top)	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5
Drilled Uniliner® (hole on bottom)	4.0 ID 6.3 OD x 78.5	20756*	20771
Double Gooseneck Drilled Uniliner® (hole on top)	4.0 ID 6.3 OD x 78.5	20508	20509
Double Gooseneck Drilled Uniliner® (hole on bottom)	4.0 ID 6.3 OD x 78.5	20954**	20989
Siltek® 1mm Drilled Uniliner® (hole on top)	1.0 ID 6.3 OD x 78.5	21390-214.1	21391-214.5

DI Liners for Shimadzu 17A & 2010 GCs (For 0.32/0.53mm ID Columns)	ID/OD & Length (mm)	ea.	cat.# 5-pk.
Open-top Drilled Uniliner® (hole on top)	3.5 ID 5.0 OD x 95	21285	21286
Open-top Drilled Uniliner® (hole on bottom)	3.5 ID 5.0 OD x 95	21287	21288
Gooseneck Drilled Uniliner® (hole on top)	3.5 ID 5.0 OD x 95	21289	21290
Gooseneck Drilled Uniliner® (hole on bottom)	3.5 ID 5.0 OD x 95	21291	21292

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.
Auto SYS Drilled Uniliner® (hole on top)	4.0 ID 6.2 OD x 92.1	20819	20822
Auto SYS Drilled Uniliner® (hole on bottom)	4.0 ID 6.2 OD x 92.1	21293	21294
Auto SYS Gooseneck Drilled Uniliner® (hole on top)	4.0 ID 5.0 OD x 92.1	21295	21296
Auto SYS Gooseneck Drilled Uniliner® (hole on bottom)	4.0 ID 6.2 OD x 92.1	21297	21298

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

Direct Injection Liners for Thermo Finnigan 8000 & TRACE™ Series GCs (0.32 & 0.53mm ID columns)	ID/OD & Length (mm)	ea.	cat.# 5-pk.
Drilled Uniliner® (hole on top)	5.0 ID 8.0 OD x 105	22411	22412
Drilled Uniliner® (hole on bottom)	5.0 ID 8.0 OD x 105	22413	22414

*Similar to Agilent part # G1544-80730.
**Similar to Agilent part # G1544-80700.

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 new!

- 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 new!

- Max. temp.: 450°C.

Description	qty.	cat.#
Graphite O-Rings for Shimadzu Split Liners	5-pk.	20243
Graphite O-Rings for Shimadzu Splitless Liners	5-pk.	20244

Viton® O-Rings for Shimadzu 17A and 2010 GCs new!

- Max. temp.: 250°C.

Description	qty.	cat.#
Viton® O-Rings for Shimadzu 17A and 2010 GCs	10-pk.	21477

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 1/16" 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 1/16" compression style nuts			
Injector Adaptor for PerkinElmer Auto SYS	—	ea.	22319
Siltek®-Treated Injector Adaptor for PerkinElmer Auto SYS XL	—	ea.	22321

free literature

Genuine Restek Replacement Parts for Agilent GCs will be available soon. This 60-page catalog details innovative, high-performance supplies for your Agilent GC—from injector to detector. (lit. cat.# 59627E).

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

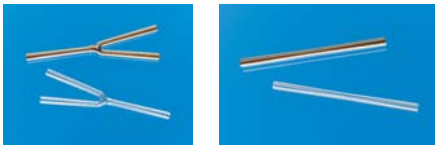
Graphite Ferrules for SeCure™ "Y" Connectors

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

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.

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

Universal Press-Tight® Connectors

Description	5-pk.	25-pk.	100-pk.
Universal Press-Tight® Connectors	20400	20401	20402
Siltek®-treated Universal Press-Tight® Connectors	20480	20449	20481

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

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.

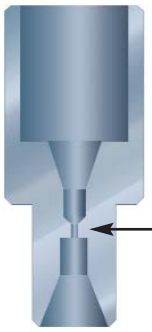


Figure 1 A laser machined orifice, coupled with the 100µm ID transfer line, minimizes the amount of oxygen entering the MS, which eliminates the need for purge gases.

Laser drilled to 100µm ID

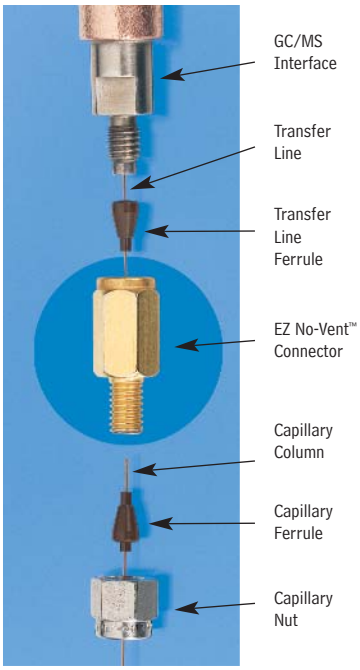
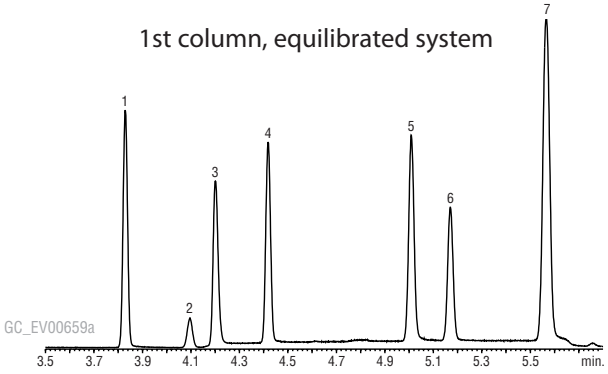
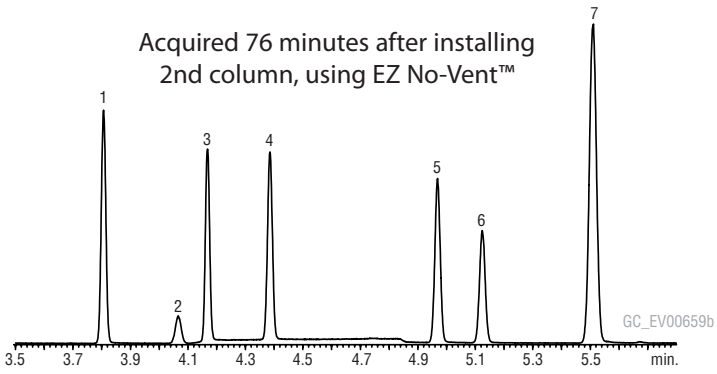


Figure 2 Sharp, symmetric peaks for gases show the EZ No-Vent™ connector does not add dead volume and allows rapid column changes.



502.2 Calibration Mix#1 (gases) cat# 30042

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



Column:	Rtx®-624 60m, 0.25mm ID, 1.4µm (cat# 10969)	Oven temp.:	60°C
Inj.:	purge & trap	Det:	Agilent 5973 GC/MS
GC	Agilent 6890	Transfer line temp.:	280°C
Inj. temp.:	300°C	Scan range:	35-550 amu
Carrier gas:	helium, constant flow	Tune	BFB
Flow rate:	1.0mL/min.	Ionization:	EI

Description	qty.	cat.#
EZ No-Vent™ Connector Kit for Agilent 5971/5972 and 5973 GC/MS	kit	21323
EZ No-Vent™ Connector Kit for Varian Saturn 2000 Series MSs **new!**	kit	22423
Replacement ferrules for connecting capillary column to EZ No-Vent™:		
0.4mm ID	2-pk.	21015
0.5mm ID	2-pk.	21016
Replacement ferrules for connecting transfer line to EZ No-Vent™: 0.4mm ID	2-pk.	21043
Replacement 100µm deactivated transfer line	3 ft.	21018
Replacement EZ No-Vent™ Column Nut	5-pk.	21900
Replacement EZ No-Vent™ Plug	2-pk.	21915
Open-End Wrenches (1/4" x 5/16")	2-pk.	20110

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 transfer line (3 ft.), column plug, column nut.

did you know?

Restek offers many innovative tools and supplies for your MS. Refer to our catalog or website.

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®-5Sil 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

LC Autosampler Syringes
Instrument parts - for Agilent, Beckman, Hitachi, PerkinElmer, Shimadzu, Thermo Separation Products, and Waters equipment.
Mobile Phase Pre-heater - Accurately and reproducibly warms mobile phase, for more consistent analyses.

Restek Performance Coatings

Siltek®, Sulfinert®, and Silcosteel®-CR treated Swagelok® fittings - Inert fittings for demanding applications.
Siltek® and Silcosteel®-CR treated electropolished stainless steel tubing - For the most inert sample pathways available.

Air Monitoring

Improved SilcoCan™ and TO-Can™ air sampling canisters - Superior deactivation; superior protection for the valve.
Canister Heating Jacket - Uniformly heats entire canister, and valve, for faster more efficient cleaning.

Analytical Reference Materials

Calibration mixes, check mixes, surrogates, and internal standards for environmental analyses - volatile organics…
semivolatile organics…pesticides & herbicides…UST monitoring...Canadian environmental methods
Dimethyldichlorosilane (DMDCS) deactivation reagent
Drinking water odor compounds

If you haven't received your copy, request one from your Restek representative.
Or, visit our website: **www.restek.com** or our new performance coatings website:
www.restekcoatings.com

Please direct your comments on this publication to Carrie Sprout at carrie.sprout@restekcorp.com or call 814-353-1300, ext. 2151.

RESTEK 20 YEARS

Lit. Cat.# 59077-INT

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ISO 9001:2000 cert.# FM80397

the RESTEK Advantage
Innovators of High Resolution Chromatography Products

Stx®-1HT Columns

new

Low Bleed for High-Temperature Analysis

by Neil Mosesman, GC Columns Product Marketing Manager,
& Dinesh Patwardhan, Ph.D., Senior R&D Chemist

- ✔ Thermally stable to 380°C
- ✔ True dimethyl polysiloxane polarity
- ✔ Low bleed (<10pa @ 380°C)
- ✔ Unique Siltek™-deactivated high-temperature tubing

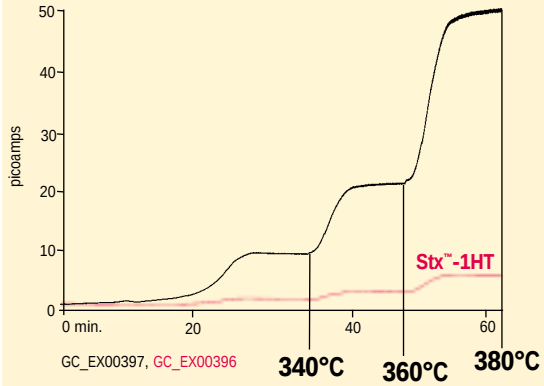
Analyzing high molecular weight compounds by gas chromatography (GC) has been difficult because of the limited thermal stability of the stationary phases and the fused silica tubing. At temperatures above 340°C most stationary phases exhibit high bleed that can interfere with peak identification and affect quantitaion. In addition, the polyimide coating on standard fused silica tubing can become brittle at temperatures above 360°C, causing spontaneous breakage when the column is exposed to elevated temperatures too long.

Extensive research by Restek R&D scientists has led to a significant innovation in high-temperature analysis. They combined the unique properties of Siltek™ deactivation, high-temperature fused silica tubing, and a specially synthesized dimethyl polysiloxane phase to create a non-polar capillary column with thermal stability up to 380°C. The result is the new Stx®-1HT column, which shows significant low bleed, even at temperatures above 340°C (Figure 1).

Total petroleum hydrocarbon (TPH) analysis is a common test performed in environmental laboratories. In addition to gasoline range and diesel range organics (GRO and DRO, respectively), samples often contain high molecular weight hydrocarbons from motor oil or hydraulic fluid. To elute these high molecular weight hydrocarbons from the column requires high oven temperatures. The Stx®-1HT column has the necessary thermal stability and low bleed to perform the analysis of hydrocarbons up to C58 in approximately 50 minutes (Figure 2).

To ensure continued low-bleed performance at high temperatures, it is critical to prevent oxygen from entering the column. Therefore, the system should be leak-checked before heating the column. Even a small leak will cause air to enter and bleed to increase. Also, we recommend using graphite fer-

Figure 1
The Stx™-1HT column exhibits extremely low bleed at high temperatures.



30m, 0.32mm ID, 0.10µm Stx™-1HT (cat.# 11709) & 30m, 0.32mm ID, 0.10µm Rtx®-1 (cat.# 10109); **GC:** Agilent 6890; **Oven program:** 100°C to 340°C at 10°C/min. (hold 10 min.) to 360°C @ 10°C/min. (hold 10 min.) to 380°C @ 10°C/min. (hold 10 min.); **Detector:** FID @ 380°C; **Injector:** split at 250°C; **Septum purge:** 4.0cc/min.; **Carrier gas:** helium; **Split vent flow rate:** 25cc/min., 20:1 split; **Head pressure:** 8.0psi; **Linear velocity:** 26cm/sec.; **Make-up gas flow:** 45cc/min., nitrogen

in this issue

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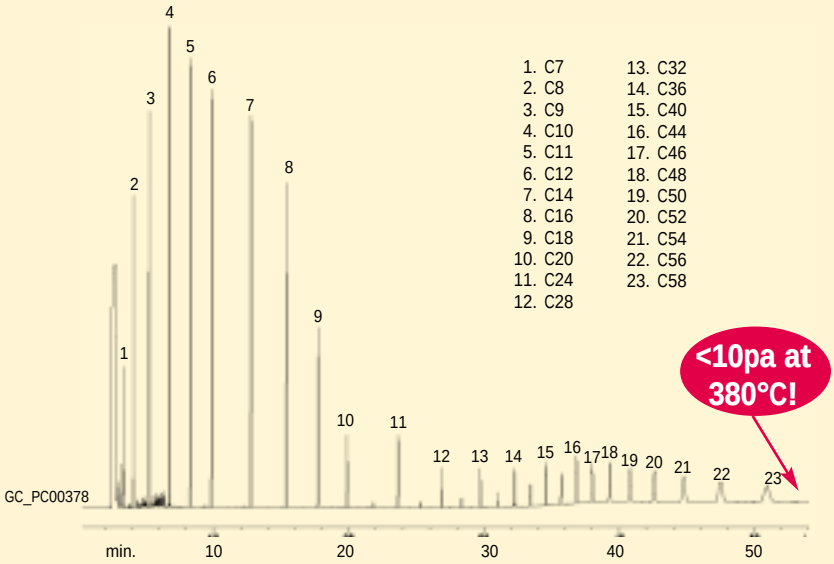
Spring'01 INTERNATIONAL

Figure 2

The Stx™-1HT column is an excellent choice for high-temperature, low-bleed hydrocarbon analysis up to C58.



30m, 0.32mm ID, 0.10µm Stx™-1HT (cat.# 11709); 0.20µL injection of D2887 calibration mix (cat.# 31222), plus C46 to C58 hydrocarbons; **Solvent:** carbon disulfide; **GC:** Agilent 6890; **Oven program:** 30°C to 380°C at 10°C/min. (hold 15 min.); **Detector:** FID @ 380°C; **Injector:** cool on-column/track oven temperature; **Carrier gas:** helium; **Flow rate:** 1.3cc/min. @30°C; **Linear velocity:** 22cm/sec. @ 30°C, constant pressure with EPC; **Make-up gas flow:** 45cc/min.; **Head pressure:** 6.0psi

✔ Stx®-1HT Columns (100% dimethylpolysiloxane) Stable to 380°C

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.10	-60 to 380°C	11705	11708
0.32mm	0.10	-60 to 380°C	11706	11709

✔ Siltek™ Guard Colums

ID	Nominal OD	5-Meter	10-Meter
0.25mm	0.37 ±0.04mm	10026	10036
0.32mm	0.45 ±0.04mm	10027	10037

✔ Siltek™ Press-Tight® Connectors

Type	5-pk.	25-pk.	100-pk.
straight	20480	20449	20481
angled	20482	20483	20484

✔ Siltek™ Inlet Liners Add the appropriate suffix to your Restek liner catalog number.

For a complete inlet liner offering, refer to the Restek Annual Chromatography Products Guide *(lit. cat.# 59960).*

qty.	Siltek™		Siltek™ with Siltek™ wool		Siltek™ with CarboFrit™	
each	-214.1	addl. cost	-213.1	addl. cost	-216.1	addl. cost
5-pack	-214.5	addl. cost	-213.5	addl. cost	-216.5	addl. cost
25-pack	-214.25	addl. cost	-213.25	addl. cost	-216.25	addl. cost

Stx®-1HT Columns cont.

rules, high-temperature septa, and checking connectors for leaks after every thermal cycle.

Stringent quality assurance specifications ensure consistent column-to-column reproducibility for Stx®-1HT columns. Each column is tested for inertness, efficiency, and bleed level at its maximum temperature (380°C). In fact, Stx®-1HT columns are guaranteed to exhibit less than 10pA of bleed at 380°C in a leak-free system.

The Stx®-1HT column is a major advancement for high-temperature analysis. The combination of Siltek™ deactivation, high-temperature fused silica tubing, and a specially synthesized stationary phase has created a column that exhibits low bleed at temperatures as high as 380°C.

for more info

request the "Stx®-1HT Benefits Brochure" (lit. cat.# #59283).

✔ D2887 SimDist Calibration Mix

Compound	Conc. (% w/w)	Compound	Conc. (% w/w)
n-hexane (C6)	6	*n*-octadecane (C18)	5
n-heptane (C7)	6	*n*-eicosane (C20)	2
n-octane (C8)	8	*n*-tetracosane (C24)	2
n-nonane (C9)	8	*n*-octacosane (C28)	1
n-decane (C10)	12	*n*-dotriacontane (C32)	1
n-undecane (C11)	12	*n*-hexatriacontane (C36)	1
n-dodecane (C12)	12	*n*-tetracontane (C40)	1
n-tetradecane (C14)	12	*n*-tetratetracontane (C44)	1
n-hexadecane (C16)	10		

Packaged 1mL per ampul

Ea.	10-pk.w/data pack
31222	31322

✔ Thermolite® Septa

Diameter	25-pk.	50-pk.	100-pk.
5mm	20351	20352	20353
9.5mm	20359	20360	20361
10mm	20378	20379	20380
11mm	20363	20364	20365
11.5mm	22385	22386	22387
17mm	20384	20385	20386
Plug	20372	20373	20374

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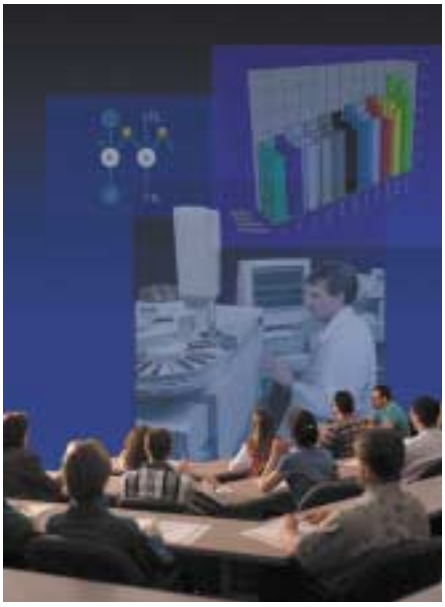

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State	City	Type	Hotel	Cat. #	Date
AZ	Phoenix	GC	Hilton Phoenix Airport (480-894-1600)	65220	Oct. 8
AZ	Tucson	GC	Embassy Suites, Tucson International Airport (520-573-0700)	65221	Oct. 9
CA	Orange County	GC	Wyndham Garden Hotel, Orange County Arprt. (714-751-5100)	65229	Oct. 16
CA	San Diego	GC	The Westin, Horton Plaza (619-239-2200)	65228	Oct. 15
CA	Walnut Creek	GC	Embassy Suites (925-934-2500)	65230	Oct. 18
CO	Denver	GC	Doubletree Hotel Denver Southeast (in Aurora) (303-337-2800)	65218	Oct. 4
CT	Groton	GC	Groton Inn & Suites (860-445-9784)	65199	Sep. 21
DE	Wilmington	GC	Wyndham Grand Hotel (302-655-0400)	65211	Sep. 28
FL	Miami	GC	Marriott Miami Airport (305-649-5000)	65226	Oct. 11
FL	Tallahasse	GC	Radisson Hotel (850-224-6000)	65224	Oct. 8
FL	Tampa	GC	Embassy Suites Hotel (813-875-1555)	65225	Oct. 9
GA	Atlanta	GC	Sheraton Gateway Hotel, Atlanta Airport (770-997-1100)	65191	Sep. 14
GA	Savannah	GC	Hilton Savannah DeSoto (912-232-9000)	65227	Oct. 12
HI	Honolulu	GC	Ala Moana Hotel (808-955-4811)	65187	Nov. 9
IL	Schaumburg	GC	Holiday Inn O'Hare Intl. (in Rosemont) (847-671-6350)	65206	Sep. 27
IN	Indianapolis	GC	Adam's Mark Hotel (317-248-2481)	65202	Sep. 20
LA	Baton Rouge	GC	Baton Rouge Marriott (225-924-5000)	65212	Oct. 1
MA	Waltham	GC	Four Points Hotel by Sheraton (781-890-0100)	65198	Sep. 20
MI	Ann Arbor	GC	Best Western Executive Plaza (734-665-4444)	65207	Sep. 28
MN	Minneapolis	GC	Minneapolis Airport Marriott (in Bloomington) (952-854-7441)	65204	Sep. 24
MO	Kansas City	GC	Park Place Hotel (816-483-9900)	65200	Sep. 17
MO	St. Louis	GC	Sheraton West Port Hotel Plaza (314-878-1500)	65201	Sep. 18
NC	Durham	GC	Durham Marriott at the Civic Center (919-683-6664)	65189	Sep. 11
NJ	Edison	GC	Clarion Hotel & Towers (732-287-3500)	65208	Sep. 24
NJ	Princeton	GC	Holiday Inn Princeton (609-452-2400)	65209	Sep. 25
NM	Albuquerque	GC	Courtyard by Marriott Journal Center (505-823-1919)	65219	Oct. 1
NV	Las Vegas	GC	Riviera Hotel & Casino (702-734-5110)	65216	Oct. 5
NY	Albany	GC	Holiday Inn Turf (518-458-7250)	65197	Sep. 18
NY	Rochester	GC	Four Points Hotel by Sheraton Rochester (716-546-6400)	65196	Sep. 17
OH	Cincinnati	GC	Crowne Plaza Hotel (513-381-4000)	65203	Sep. 21
OH	Cleveland	GC	Hilton Cleveland South (216-447-1300)	65194	Sep. 13
OH	Columbus	GC	Adams Mark Hotel (614-228-5050)	65193	Sep. 11
OK	Tulsa	GC	Sheraton Tulsa Hotel (918-627-5000)	65223	Oct. 12
PA	King of Prussia	GC	Hilton Valley Forge (610-337-1200)	65210	Sep. 27
PA	Pittsburgh	GC	Holiday Inn Monroeville (412-372-1022)	65195	Sep. 14
TN	Knoxville	GC	Hilton Knoxville Airport (865-970-4300)	65190	Sep. 13
TX	Austin	GC	Doubletree Hotel Austin (512-454-3737)	65222	Oct. 11
TX	Dallas	GC	Harvey Hotel, DFW Airport (in Irving) (972-929-4500)	65214	Oct. 4
TX	Houston	GC	Sheraton (281-442-5100)	65213	Oct. 2
TX	San Antonio	GC	Doubletree Hotel (210-366-2424)	65215	Oct. 5
UT	Salt Lake City	GC	Wyndham Hotel, Salt Lake City (801-531-7500)	65217	Oct. 2
VA	Richmond	GC	Wyndham Garden Hotel (804-226-4300)	65188	Sep. 10
WA	Seattle	GC	Doubletree Airport (206-246-8600)	65231	Oct. 19
WI	Madison	GC	Madison Concourse Hotel & Governor's Club (608-257-6000)	65205	Sep. 25
WV	Charleston	GC	Ramada Plaza Hotel (304-744-4641)	65192	Sep. 10
CA	Concord	HPLC	Hilton Concord (925-827-2000)	65611	Oct. 2
CA	San Diego	HPLC	The Westin, Horton Plaza (619-239-2200)	65608	Sep. 26
CA	Simi Valley	HPLC	Grand Vista Hotel (805-583-2000)	65609	Sep. 27
CA	Sunnyvale	HPLC	Sheraton Sunnyvale Hotel (408-745-6000)	65610	Oct. 1
IL	Schaumburg	HPLC	Holiday Inn O'Hare Intl. (in Rosemont) (847-671-6350)	65607	Sep. 21
IN	Indianapolis	HPLC	Adam's Mark Hotel (317-248-2481)	65606	Sep. 19
NJ	Princeton	HPLC	Holiday Inn Princeton (609-452-2400)	65601	Sep. 11
NJ	Rahway	HPLC	Crowne Plaza Hotel (in Clark) (732-574-0100)	65602	Sep. 12
NY	Rochester	HPLC	Four Points Hotel by Sheraton Rochester (716-546-6400)	65603	Sep. 14
OH	Cincinnati	HPLC	Crowne Plaza Hotel (513-381-4000)	65605	Sep. 18
OH	Columbus	HPLC	Ramada Plaza Hotel & Conference Cntr. (614-846-0300)	65604	Sep. 17
PA	King of Prussia	HPLC	Hilton Valley Forge (610-337-1200)	65600	Sep. 10
CO	Denver	ENV	Doubletree Hotel Denver Southeast (in Aurora) (303-337-2800)	65417	Nov. 5
IL	Schaumburg	ENV	Embassy Suites (847-397-1313)	65415	Nov. 1
IN	Indianapolis	ENV	Adam's Mark Hotel (317-248-2481)	65414	Oct. 30
LA	Baton Rouge	ENV	Baton Rouge Marriott (225-924-5000)	65420	Nov. 9
MA	Waltham	ENV	Four Points Hotel by Sheraton (781-890-0100)	65409	Oct. 22
MN	Minneapolis	ENV	Minneapolis Airport Marriott (in Bloomington) (952-854-7441)	65416	Nov. 2
NJ	Edison	ENV	Clarion Hotel & Towers (732-287-3500)	65411	Oct. 25
NY	Albany	ENV	Holiday Inn Turf (518-458-7250)	65410	Oct. 23
OH	Columbus	ENV	Ramada Plaza Hotel & Conference Cntr. (614-846-0300)	65413	Oct. 29
PA	King of Prussia	ENV	Hilton Valley Forge (610-337-1200)	65412	Oct. 26
TX	Austin	ENV	Doubletree Hotel Austin (512-454-3737)	65418	Nov. 6
TX	Houston	ENV	Holiday Inn Intercontinental Airport (281-449-2311)	65419	Nov. 8

Separating *m*- and *p*-Xylene Isomers

Using an Rtx®-200 GC Column

by Christopher English, Environmental Innovations Chemist

- ✔ Unique separation of VOCs satisfy state requirements
- ✔ Low bleed at high operating temperatures (stable to 360°C)

Xylenes are aromatic hydrocarbons that naturally occur in petroleum and coal tar; they also can be commercially derived from these substrates. The US Environmental Protection Agency (EPA) does not require separation of the three xylene isomers(*m*-, *p*-, *o*-xylene), but rather requests their calculation as totals or sums.[1] Some states, such as New York, have action limits based on *m*- and *p*-xylene separately. However, the isomers of *m*- and *p*-xylene are difficult to resolve using gas chromatography (GC) and most capillary columns.

The common way to perform a GC separation of *m*- and *p*-xylene is by using a polyethylene glycol (PEG) stationary phase, such as the Restek Stabilwax® column. Chromatographically, baseline separation is possible with this column; however, the highly polar phase does not separate many of the other volatile organic compounds (VOCs).

The more ideal column choice for this separation is the Rtx®-200 column. The Crossbond® trifluoropropylmethyl polysiloxane (TFP) stationary phase features exceptionally low bleed at operating temperatures up to 360°C.

The Rtx®-200 column provides unique separation of VOCs listed in US EPA Methods 524 and 8260 (Figure 1), and is the best column to separate xylene isomers for specific state requirements (Figure 2). The only limitation of this column is the resolution of the gases.

Trifluoropropyl stationary phases, like that of the Rtx®-200 column, have a unique selectivity because of the electrophilic nature of the fluorine-containing polymer. This creates interactions with electron-rich molecules like ketones and halogenated compounds. The unique selectivity results in different elution orders and resolves compounds that phenyl, cyano, and methyl phases cannot. The Rtx®-200 column can be used to confirm tentatively identified compounds and resolve multiple coelutions, making it ideal for requirements such as those dictated by New York State.

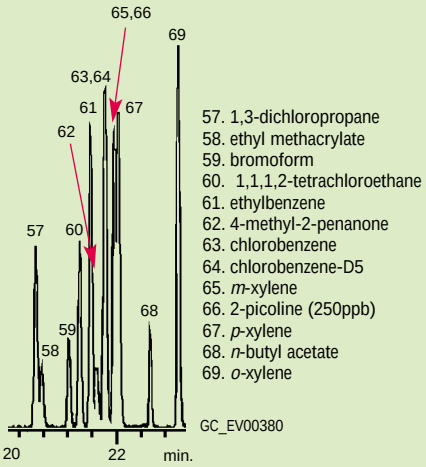
1. Toxicologicological Profile for Total Xylenes. Prepared by Clements Associates, Inc., under Contract No. 205-88-0608. Prepared for Agency of Toxic Substances and Disease Registry, US Public Health Services, Atlanta, GA. December 1990. Reference not available from Restek.

for more info

Request Applications Note #59190.

Figure 1

The Rtx®-200 column provides unique separation of the VOCs listed in US EPA Method 8260.

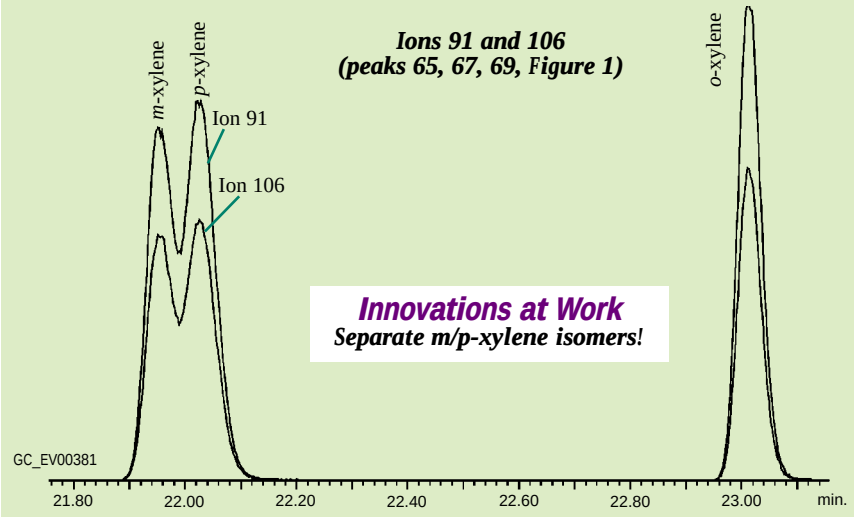


57. 1,3-dichloropropane
58. ethyl methacrylate
59. bromoform
60. 1,1,1,2-tetrachloroethane
61. ethylbenzene
62. 4-methyl-2-penanone
63. chlorobenzene
64. chlorobenzene-D5
65. *m*-xylene
66. 2-picoline (250ppb)
67. *p*-xylene
68. *n*-butyl acetate
69. *o*-xylene

Conditions for Figures 1 & 2
60m, 0.25 mm ID, 1.0µm Rtx®-200 (cat.# 15056) 10ppb each component in 5mL of RO water; **Concentrator:** Tekmar LSC-3100 Purge and Trap; **Trap:** Vocarb® 3000 (type K); **Purge:** 11 min. @ 40mL/min. @ ambient temperature; **Dry purge:** 1 min. @ 40mL/min.; **Desorb preheat:** 245°C; **Desorb:** 250°C for 2 min., flow 10mL/min.; **Bake:** 260°C for 8 min.; **Interface:** transfer line 0.53mm ID Silcosteel® MXT® tubing; **Oven program:** 40°C (hold 10 min.) to 100°C @ 6°C/min. (hold 1 min.) to 210°C @ 30°C/min. (hold 7 min.); **Carrier gas:** helium @ ~1.3mL/min. constant flow (adjust dichlorodifluoromethane to a retention time of 4.1 min. @ 40°C); **Detector:** Agilent 5973 MS, scan range 35 to 300 AMU

Figure 2

An extracted ion chromatogram shows the Rtx®-200 column separates *m/p*-xylene isomers.



✔ **Rtx®-200 Columns** (Crossbond® trifluoropropylmethyl polysiloxane) Stable to 360°C

For the complete Rtx®-200 column offering, refer to Restek's Annual Chromatography Products Guide *(lit. cat.# 59960).*

Rtx®-200 (Fused Silica Tubing)

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter	105-Meter
0.25mm	1.00	-20 to 290/310°C	15050	15053	15056	15059

MXT®-200 (MXT® Tubing)

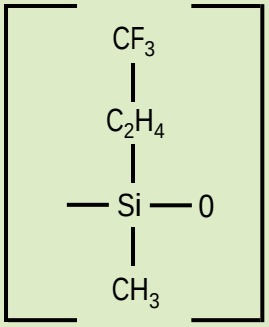
ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	1.00	-20 to 310°C	75050	75053

An extensive selection of **analytical reference materials** for US EPA Method 8260B is available from Restek. Please see page 16 of this newsletter or visit our web site at **www.restekcorp.com** for ordering information.

Restek TFP Phase

Low Bleed with a High Degree of Inertness and Efficiency

by Frank Dorman, Ph.D., Environmental Innovations Chemist



TFP phase structure of the Rtx®-200 column.

Several factors are important when designing a trifluoropropyl polysiloxane (TFP) stationary phase (i.e., Restek's Rtx®-200 columns): bleed level, inertness, and efficiency. In order to obtain a low bleed level and high thermal stability, it is necessary to apply a suitable deactivation to the fused silica tubing before coating it with the stationary phase. The deactivation modifies the fused silica surface so that it is more compatible with the stationary phase, otherwise the stationary phase will not coat evenly and will not bond, or link to, the tubing wall. Other manufacturers' "200-type phases" have this problem and, as a result, suffer from low thermal stability. However, Restek designed a deactivation layer that is matched to the TFP phase, so you get low bleed levels and thermal stability to 360°C.

Inertness also can be a function of the deactivation chemistry. Restek tests all Rtx®-200 columns using compounds that are highly susceptible to on-column breakdown and would indicate a problem with column inertness. Testing with a quality assurance mixture including reactive phenolics, anilines, and alcohols ensures that each Rtx®-200 column has the inertness to function at high performance levels.

Finally, efficiency is a measure of the interaction between the compounds in the sample and the stationary phase. If there is a high resistance to mass transport in the stationary phase, then the compounds elute as broad peaks in the chromatogram and resolution is negatively effected. The Rtx®-200 columns maintain a high efficiency, resulting in peak widths that are similar to those obtained with the Rtx®-1 column—one of the most efficient phases available.

While other manufacturers struggle with "200-type" phases that have poor efficiency, inertness, and thermal stability, Restek customers can expect to achieve the unique selectivity of TFP phases without compromising these other important chromatographic conditions.

New Reference Texts

For GC, GC/MS, HPLC, & More!

by Jack Crissman, Training & Education Manager

Analytical Gas Chromatography, 2nd Ed.
W. Jennings, E. Mittlefehldt and P. Stremple, Academic Press, 1997, 389pp.
cat.# 21362, (ea.)

Basic Gas Chromatography
H. M. McNair and J. M. Miller, John Wiley, 1997, 200pp.
cat.# 21366, (ea.)

Capillary Gas Adsorption Chromatography
V. G. Berezkin and J. de Zeeuw, Wiley-VCH, 1998, 320pp.
cat.# 21097, (ea.)

Chiral Chromatography
T. E. Beesley and R. P. W. Scott, John Wiley, 1999, 506pp.
cat.# 21094, (ea.)

Environmental Sampling and Analysis Lab Manual
M. Csuros, CRC Press LLC, 1997, 373pp. (softcover)
cat.# 21375, (ea.)

GC/MS in Clinical Chemistry
P. Gerhards, U. Bons, J. Sawazki, J. Szigan and A. Wertmann, Wiley-VCH, 1999, 241pp.
cat.# 21096, (ea.)

Handbook of Chemistry and Physics, 81st Ed.
D. R. Lide, CRC Press LLC, 2000, 2480pp.
cat.# 21376, (ea.)

Handbook of Environmental Analysis
P. Patnaik, CRC Press LLC, 1997, 584pp.
cat.# 21381, (ea.)

HPLC Columns. Theory, Technology, and Practice
U. D. Neue, John Wiley, 1997, 393pp.
cat.# 21368, (ea.)

HPLC in Enzymatic Analysis, 2nd Ed.
E. F. Rossomando, John Wiley, 1998, 451pp.
cat.# 21364, (ea.)

Liquid Chromatography, Essential Data
D. Patel, John Wiley, 1997, 146pp. (softcover)
cat.# 21372, (ea.)

The Merck Index, 12th Ed.
S. Budvari, Merck, 1996, 2240pp.
cat.# 21383, (ea.)

Modern Chromatographic Analysis of Vitamins, 3rd Ed.
A. P. De Leenheer, W. E. Lambert and J. F. Van Bocxlaer, Marcel Dekker, 2000, 616pp.
cat.# 21092, (ea.)

Molecular Basis of Chromatographic Separation
E. Forgacs and T. Cserhati, CRC Press LLC, 1997, 243pp.
cat.# 21378, (ea.)

Practical High-Performance Liquid Chromatography, 3rd Ed.
V. R. Meyer, John Wiley, 1999, 338pp. (softcover)
cat.# 21095, (ea.)

Practical HPLC Method Development, 2nd Ed.
L. R. Snyder, J. J. Kirkland and J. L. Glajch, John Wiley, 1997, 765pp.
cat.# 21363, (ea.)

Practical Introduction to GC-MS Analysis with Quadrupoles
M. Oehme, Wiley-VCH, 1999, 195pp. (softcover)
cat.# 21098, (ea.)

Practical Problem Solving in HPLC
S. Kromidas, Wiley-VCH, 2000, 178pp. (softcover)
cat.# 21099, (ea.)

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Fast Analysis of Dioxin & Related Compounds

Using an Rtx®-5MS Column

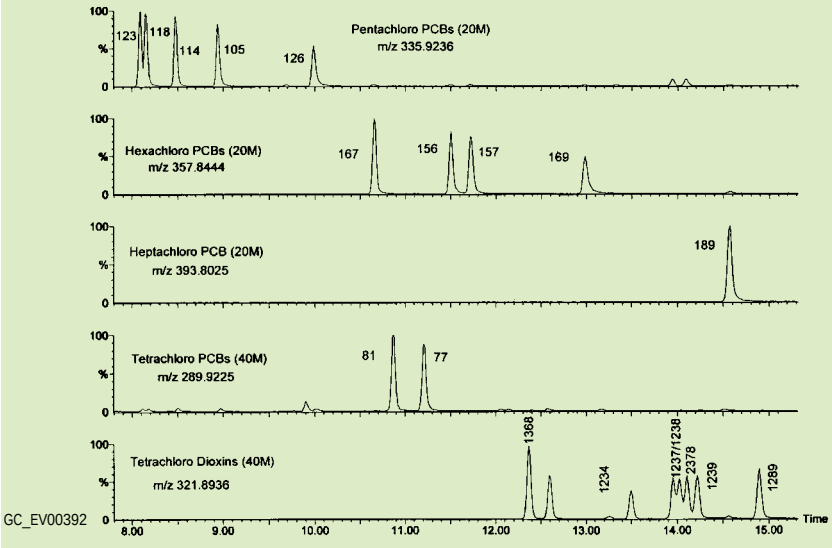
by Karen MacPherson, Eric Reiner, Ph.D.,* & Frank Dorman, Ph.D.

Historically, chlorinated dioxins and furans have been analyzed by gas chromatography (GC) separately from polychlorinated biphenyls (PCBs). In 1998, the World Health Organization (WHO) reported toxic equivalent factors (TEFs) for the 12 dioxin-like PCB congeners.[1] This enabled concentrations of PCBs to be expressed in terms of 2,3,7,8-TCDD, the most toxic form of dioxin. Using similar methods to analyze dioxins and PCBs allows detection limits up to three orders of magnitude lower than that of conventional of PCB congener methods. The toxicity of a single sample now can be reported in toxic equivalents of 2,3,7,8,-TCDD (i.e., toxic equivalent quantities [TEQ]) by summing the toxic equivalents of each of the 17 toxic dioxin congeners and 12 dioxin-like PCB congeners.

Extracts were prepared according to Canada's Ministry of the Environment (MOE) Method 3418, which is similar to the combination of US Environmental Protection Agency (EPA) Methods 1613 and 1668. The extracts are further cleaned using activated carbon.[2] This allows for the collection of two sample extract fractions: one containing the dioxins, furans, and coplanar PCBs; and the other containing the remaining PCBs, chlorinated and brominated diphenyl ethers, and other non-planar organic compounds. The chlorinated diphenyl ethers interfere with the furans and, therefore, they need to be analyzed separately. Normally, dioxins and furans, and PCBs (congeners) are analyzed separately on a 60m analytical column using GC/high resolution mass spectrometry (GC/HRMS) with analysis times of 50 to 90 minutes each.

Figure 1

The Rtx®-5MS column and a combined system can separate dioxin-like PCBs and dioxins/furans.



Inj. temp.: 280°C; **Carrier gas:** helium; **Det.:** Agilent 6890 GC coupled to a Micromass Ultima HRMS @10,000RP.

20m, 0.1mm ID, 0.1µm Rtx®-5MS (custom cat.# 58136)
Column head pressure: 100psi; **Oven program:** 100°C (hold 1 min.) to 200°C @ 100°C/min, to 235°C @ 13°C/min., to 300°C @ 27°C/min. (hold 4 min.); **Inj. volume:** 0.2mL

40m, 0.18mm ID, 0.18µm Rtx®-5MS (custom cat.# 550590)
Column head pressure: 61psi; **Oven program:** 100°C (hold 0.62 min.) to 200°C @ 64.5°C/min, to 235°C @ 4.8°C/min. (hold 6.2 min.), to 300°C @ 9.7°C/min.(hold 5.6 min.); **Inj. volume** 1.0mL

✔ **Rtx®-5MS Columns** (Crossbond® 5% diphenyl - 95% dimethyl polysiloxane) Stable to 360°C

For the complete Rtx®-5MS column offering, refer to Restek's Annual Chromatography Products Guide *(lit. cat.# 59960).*

Length	ID	df (µm)	temp. limits	cat.#
20-Meter	0.10mm	0.10	-60 to 330/350°C	58136
40-Meter	0.18mm	0.18	-60 to 330/350°C	550590

*Karen MacPherson and Dr. Eric Reiner, Ontario Ministry of the Environment.

**For more information on the system set-up, request Applications Note #59343.

Because an MS is used for detection, many analysts want a column with the lowest bleed possible. Some laboratories may use silarylene columns (e.g., Rtx®-5Sil MS or DB-5MS® columns) due to their low bleed feature. However, these columns yield a coelution between 2,3,7,8-TCDD and 1,2,3,9-TCDD; and their elution orders and retention times will differ from the phase for which the window performance mixtures were designed. The Rtx®-5MS (5% diphenyl/95% dimethyl polysiloxane) column is better suited to meet the performance standards for this analysis. It separates all of the important compounds, and each one is individually tested to provide low bleed levels for MS detection.

Chromatographic resolution and analysis time also are dependent on column dimensions (i.e., length, ID, phase thickness). Experimentally, we have found 175,000 plates are required to obtain separation of 2,3,7,8-TCDD from its nearest neighbors (1,2,3,7- and 1,2,3,8-TCDD—the unresolved pair eluting before; and 1,2,3,9-TCDD—the compound eluting after).[3] A 40m Rtx®-5MS column meets this criterion, and can complete the analysis in approximately half as much time as a 60m column. A 20m column is capable of meeting these requirements in about one-quarter the time of a 60m column; however, there is little tubing length available for trimming to maintain column performance. Therefore, we suggest using a 40m column.

To minimize the number of ions that must be monitored simultaneously, elute the bulk of PCB compounds prior to eluting dioxin and furan compounds. Accomplish this by injecting the non-coplanar PCB fraction into a 20m Rtx®-5MS column that is set up parallel (i.e., two separate injectors) to a 40m Rtx®-5MS column, which is used for the separation of the dioxin/furan/coplanar PCB fraction. Both fractions are injected simultaneously. The columns are installed into the MS ion source in parallel.** The resulting analysis time is less than that for a single fraction on a conventional 60m column (Figure 1).

For the analysis of dioxin-like PCBs and dioxins/furans, method consolidation and throughput increase is possible when using a parallel, dual-column system with GC/HRMS. This method allows the combination of several different analytical methods to a single system, and results in a total analysis time of less than 30 minutes for elution of octachlorodibenzodioxin. If your laboratory is involved in the analysis of dioxin and related compounds, and you would like more detailed information on this method, please contact Restek Technical Service at 800-356-1688 or 814-353-1300, ext. 4.

References

1. Berg, M. V., L. Birnbaum, A.T.C. Bosveld, B. Brunstrom, P. Cook, M. Feeley, J.P. Giesy, A. Hanberg, R. Hasegawa, S.W. Kennedy, T. Kubiak, J.C. Larsen, F.X.R. Leeuwen, A.K.D. Liem, C. Nolt, R.E. Peterson, L. Poellinger, S. Safe, D. Schrenk, D. Tillitt, M. Tysklind, M. Younes, F. Waern, and T. Zacharewski, Environmental Health Perspectives, 106 (1998), p. 775.
2. Kolic T.M., K. A. MacPherson, E.J. Reiner, T. Gobran, and A. Hayton, Organohalogen Compounds, 46 (2000), p. 562.
3. Reiner E.J., K.A. MacPherson, R. Brunato, T. Chen, M.A. Bogard, A.R. Boden, and G. Ladwig, Organohalogen Compounds, 45 (2000), p. 17.

TO-Can™ Air Monitoring Canisters

Optimized for EPA Methods T0-14 and TO-15

by David Shelow, Air Monitoring Innovations Chemist

- ✔ SUMMA® can equivalent
- ✔ Excellent recovery—even after 14 days of storage

US Environmental Protection Agency (EPA) Compendium of Air Methods TO-14 and TO-15 regulate the collection, storage, and analysis of volatile organic compounds (VOCs) using treated air sampling canisters. Restek now offers a complete line of TO-Can™ canisters (SUMMA® can equivalent), which are electropolished using a proprietary process and extensively cleaned using an ultrasonic method. This ensures a high-quality, passivated surface to maintain stability of the TO-14/TO-15 compounds during storage. Also, the design of the frame surrounding the electropolished canister eliminates the need for weld marks on the sphere, thereby preventing active sites on the canisters. And the addition of a Parker Hannifin metal-to-metal diaphragm valve further improves the performance of the canister.

To collect VOCs in ambient air, the TO-Can™ canisters should be pre-cleaned and pre-evacuated prior to being sent to the field. Once in the field, the sample is drawn through a sampling train that will regulate the rate and duration of sampling. The TO-Can™ canister is then sent to an analytical laboratory for analysis. In the laboratory, a known amount of sample is drawn from the canister and concentrated onto a concentrating trap. The sample is analyzed according to Method TO-14/TO-15, typically using a 60m, 0.32mm ID, 1.0µm Rtx®-1 capillary column and a GC/MS system.

To show the stability of these canisters, and how well they meet the holding time criteria for Methods TO-14/15, a 62-component TO-15 standard (10ppbv) was injected into a TO-Can™ canister and humidified to 70% relative humidity. The standard was analyzed on day 1, day 7, and day 14. The TO-Can™ canister demonstrated excellent stability for these polar and non-polar compounds. The resulting analysis shows excellent stability after 14 days storage of the compounds (Figure 1).

✔ TO-Can™ Canisters

size (L)	cat.#
1	24150
3	24152
6	24153
15	24154

✔ TO-Can™ Canisters with Vacuum/Pressure Gauge

size (L)	cat.#
1	24155
3	24156
6	24157
15	24158

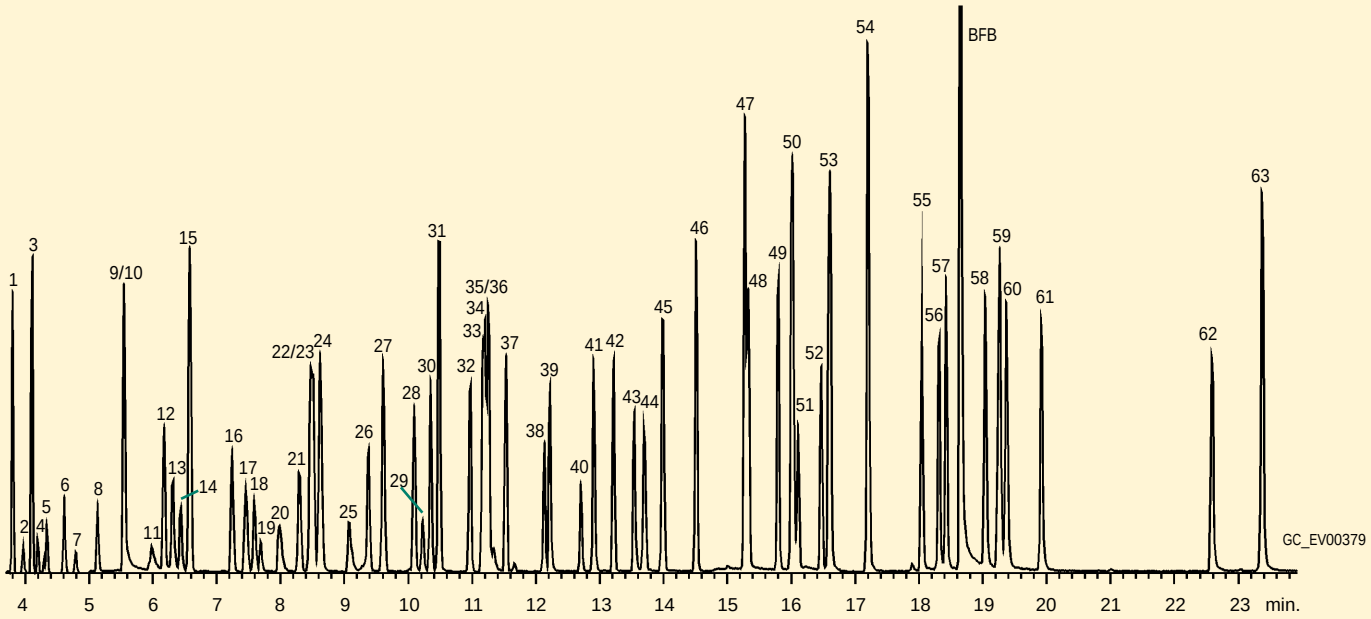
for more info

including complete stability test data, request Applications Note #59189.

Restek also offers SilcoCan™ canisters for the analysis of low-level sulfur compounds. For more information on SilcoCan™ canisters, request Fast Facts #59311.

Figure 1

TO-Can™ canisters ensure excellent recovery for TO-14/TO-15 compounds, even after 14 days of storage.



60m, 0.32mm ID, 1.0µm Rtx-1 (cat.# 10157). 200mL injection of a 10ppbv TO-15 standard (cat.# 34436), made in TO-Can canister and humidified to 70% RH. **Concentrator:** Nutech 3550 Preconcentrator; 200mL of sample concentrated at 160°C, thermally desorbed at 150°C, and cryofocused at 185°C. **Oven temp.:** 30°C (hold 4 min.) to 175°C @ 9°C/min. to 220°C @ 40°C/min. **Carrier gas:** helium @1.2mL/min. **Det.:** Agilent 5971 MS, scan range 35-265amu ***(For peak identifications, request Applications Note #59189.)***

Chromatogram courtesy of Gina Maio at Severn Trent Laboratories, Inc., Burlington, VT.

Column Bleed & System Contamination

Identifying and Reducing Sources of Rising Baselines in GC Analysis

by Neil Mosesman, GC Columns Product Marketing Manager

- ✔ Improve qualitative and quantitative reliability
- ✔ Increase column lifetime

Rising baselines are a common occurrence during temperature-programmed gas chromatography (GC) (Figure 1). The rise in the baseline can be caused by several factors: stationary phase bleed from the analytical column, contamination in the injection or detection system, and/or a change in the flow rate. The magnitude of the baseline rise often depends on the sensitivity of the detection system. With very sensitive detectors, even a small amount of bleed or contamination can cause a significant rise in the baseline. Reducing or eliminating rising baselines can improve qualitative and quantitative reliability of your chromatographic analyses.

Because both the column and the system can contribute to rising baselines, it is important to distinguish between the two sources when troubleshooting. The simplest way to do this is to remove the analytical column from the system, cap off the detector, and determine the background level during a temperature-programmed run. If the baseline is unstable, follow the recommendations in "How can detector effects be reduced?" If the baseline is stable, connect a jumper (i.e., a short length of clean, uncoated fused silica tubing) from the injector to the detector and perform another temperature-programmed run to show the effects of the injector on baseline stability. If the baseline is unstable, follow the recommendations in "How can injector effects be reduced?" If the baseline from the injector and detector is stable, install the analytical column and perform a temperature-programmed run without making an injection to determine if addition of the column increases the background level.

How can injector effects be reduced?

Injector contamination can be a major cause of baseline instability. High molecular weight, nonvolatile residue from the sample can slowly migrate through the analytical column and cause a rise in the baseline during a temperature-programmed run. It often is difficult to determine if the baseline rise is caused by the column or by injector contamination. Removing the column from the GC and running a jumper (see above) will isolate the source of the baseline rise. If the injector is contributing to the bleed level, maintenance should be performed. In particular, replace the septum, liner, and seal. In cases of extreme contamination, rinsing the injection port with solvent may be necessary. After maintenance, confirm the cleanliness of the injection port by performing a blank injection with the jumper installed between the injector and the detector.

How can detector effects be reduced?

Baseline rise from the detector is usually caused by contamination or impure gas. Proper detector maintenance, including periodic cleaning, is critical to minimizing baseline rise. Make-up gas and/or fuel gases also can contribute to bleed. Figure 2 shows an unstable flame ionization detector (FID) baseline caused by trace impurities in the compressed air supply to the flame. Switching to a high-purity air generator that employs traps to remove trace hydrocarbons can greatly improve FID baseline stability (Figure 3). Using high-quality gas purifiers for make-up gas and fuel gases is critical to reducing background levels caused by the detection system.

How can column bleed be reduced?

If the baseline rise caused by the injector and detector has been reduced and the baseline still remains high, then the most likely cause is column bleed. The magnitude of column bleed is related to the final oven temperature. Higher final tempera-

Figure 1

Rising baselines are a common occurrence during temperature-programmed GC.

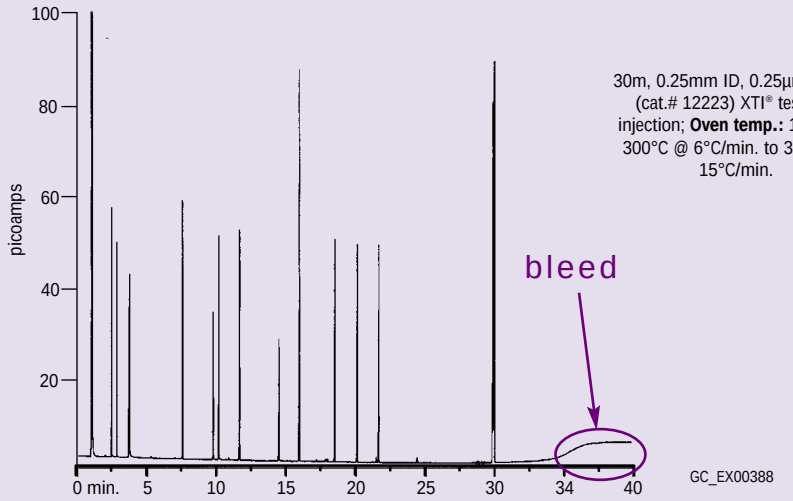


Figure 2

Trace impurities in the carrier gas can cause an unstable FID baseline.

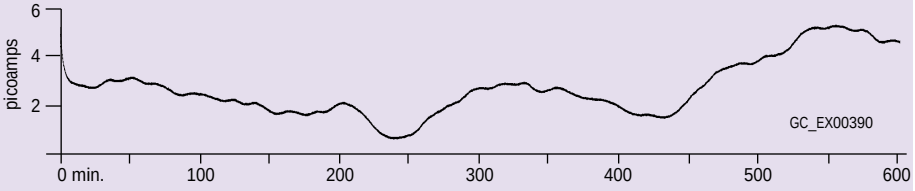


Figure 3

Using a high-purity gas generator and traps to remove contaminants can greatly improve FID baseline stability.

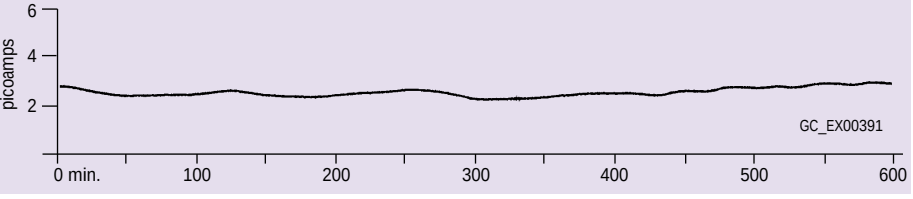
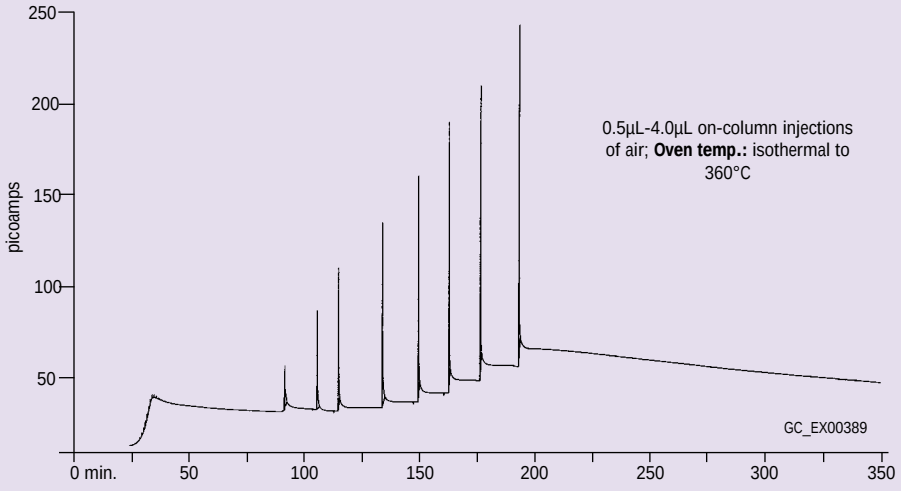


Figure 4

Trace amounts of oxygen in the carrier gas will cause an elevated baseline.



tures will result in higher bleed levels. To minimize column bleed, proper conditioning procedures must be followed when installing a new column. New columns generally are pre-conditioned by the manufacturer and should not require a significant amount of conditioning unless they are installed in highly sensitive detectors.

Conditioning columns at high temperatures actually can damage the column if the carrier gas contains trace amounts of oxygen or there are leaks in the carrier gas lines. These can oxidize the stationary phase and cause column bleed. To demonstrate this, we introduced increasing volumes of room air onto an Rtx®-5 capillary column at 360°C; notice the elevated baseline after every injection of air(Figure 4). After exposure to oxygen, several hours of continued conditioning with clean gas were needed to return the baseline to its original level. If trace amounts of oxygen are introduced continuously onto the column through carrier gas impurities or leaks, the baseline may never return to its original level. Therefore, it is imperative to use high-quality oxygen and moisture traps on all carrier gas lines and to thoroughly check for leaks using an electronic leak detector before conditioning the column.

Because oxidation of the phase is related to conditioning temperatures, keep them as low as possible. Condition columns at 20°C above the final temperature of the analysis or at the maximum operating temperature of the column, whichever is lower. For low-polarity phases such as Rtx®-1 (100% dimethylpolysiloxane) and Rtx®-5 (5% diphenyl/95% dimethyl polysiloxane) columns, oxygen will not begin to damage the phase until above 270°C. Therefore, longer conditioning times at lower temperatures may be more effective in reducing column bleed without risking damage from oxygen in the carrier gas.

Does sample contamination contribute to baseline rise?

High molecular weight contaminants in your samples can cause the baseline to rise during temperature programming. Conditioning the column usually can remove these contaminants, but prolonged bake-out at high temperatures increases the risk of phase oxidation. Solvent rinsing is an alternative that can remove high molecular weight contaminants without the need for high-temperature conditioning.

Summary

Controlling baseline rise is an important factor in achieving accurate quantitative and qualitative chromatographic analyses. The analytical column is not the only cause of this problem. The injector and detector also can contribute to baseline rise. Proper conditioning can reduce column bleed but, first, care must be taken to eliminate trace amounts of oxygen and leaks in the carrier gas line. Sample contamination in the analytical column should be reduced or eliminated by solvent rinsing. In addition, regular maintenance for the injector and detector are necessary for accurate analyses.

Questions?

Contact us at support@restekcorp.com. The industry's best technical service will be glad to help you with Plus 1™ service!

HPLC Analysis of Carboxylic Acids

Using Ultra Aqueous C18 Columns

by Terry Reid, HPLC Chemist

- ✔ Highly aqueous mobile phases provide maximum retention
- ✔ Proven reproducibility under harsh conditions

Small carboxylic acids can be difficult to retain using reversed phase high performance liquid chromatography (HPLC) and often require a highly aqueous mobile phase. Unfortunately, highly aqueous mobile phases are problematic for many C18 columns, leading to a reversible retention loss that is attributed to chain folding. Exposure to completely aqueous mobile phases at ambient pressure (no flow) accelerates the chain folding process.

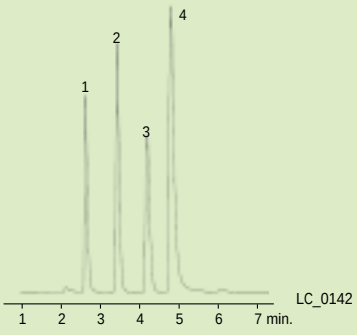
The Restek Ultra Aqueous C18 column, however, was designed to enhance retention of polar compounds and to provide completely stable retention—even when using 100% aqueous mobile phases. This column was compared to a conventional C18 column during continual exposure to a 100% aqueous mobile phase (phosphate buffer) over a three-day period of intermittent analysis and storage (Figure 1). While we do not recommend storing columns with buffer, this experiment demonstrates the complete stability of the Ultra Aqueous C18 column against retention loss caused by chain folding.

The analysis of four small carboxylic acids using an Ultra Aqueous C18 column and a completely aqueous mobile phase shows that these polar compounds can be successfully retained and resolved by reversed phase HPLC (Figure 2). Also, the reproducibility of this column is shown in the analysis of a tobacco extract. After 268 injections, the retention and peak shape are almost identical (Figure 3).

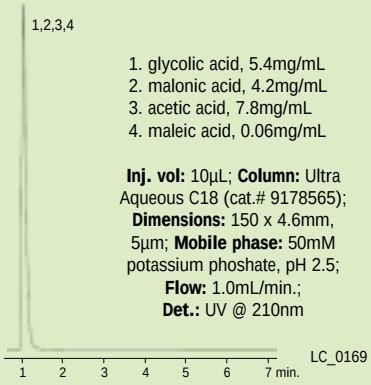
The unique characteristics of the Ultra Aqueous C18 column are advantageous for analyzing a wide range of polar compounds, including carboxylic acids. The ability to use highly aqueous mobile phases maximizes retention of polar compounds to provide enhanced resolution.

Figure 1

The Ultra Aqueous C18 column shows remarkable stability, even after 3 days of continuous exposure to a 100% aqueous mobile phase.



A conventional C18 column exhibits chain folding after exposure for 5 minutes with stopped flow.

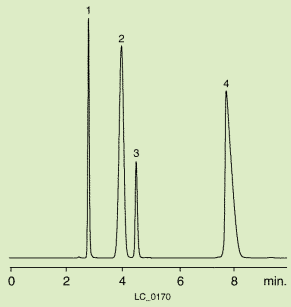


1. glycolic acid, 5.4mg/mL
2. malonic acid, 4.2mg/mL
3. acetic acid, 7.8mg/mL
4. maleic acid, 0.06mg/mL

Inj. vol: 10µL; **Column:** Ultra Aqueous C18 (cat.# 9178565); **Dimensions:** 150 x 4.6mm, 5µm; **Mobile phase:** 50mM potassium phoshate, pH 2.5; **Flow:** 1.0mL/min.; **Det.:** UV @ 210nm

Figure 2

The Ultra Aqueous C18 column successfully retains and resolves carboxylic acids.

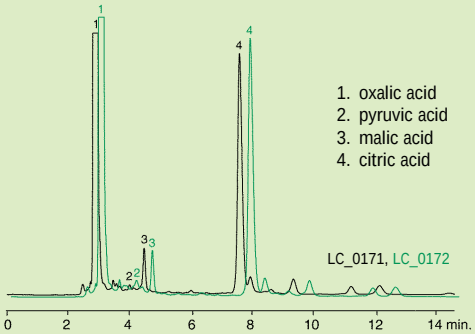


1. oxalic acid, 0.05mg/mL
2. pyruvic acid, 0.18mg/mL
3. malic acid, 0.42mg/mL
4. citric acid, 1.7mg/mL

Inj. vol: 10µL; **Column:** Ultra Aqueous C18 (cat.# 9178575); **Dimensions:** 250 x 4.6mm, 5µm; **Mobile phase:** 50mM potassium phoshate, pH 2.5; **Flow:** 1.0mL/min.; **Det.:** UV @ 210nm

Figure 3

The Ultra Aqueous C18 column demonstrates excellent reproducibility after 268 injections.



1. oxalic acid
2. pyruvic acid
3. malic acid
4. citric acid

Sample: tobacco extract; **Inj. vol.:** 20µL; **Column:** Ultra Aqueous C18 (cat.# 9178575) **Dimensions:** 250 x 4.6mm, 5µm; **Mobile phase:** 50mM potassium phosphate, pH 2.5; **Flow:** 1.0mL/min.; **Det.:** UV @ 210nm

✔ Ultra Aqueous C18 5µm HPLC Columns

For the complete HPLC columns offering, refer to Restek's Annual Chromatography Products Catalog *(lit. cat.# 59960).*

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
30mm	9178531	9178532	9178533	9178535
50mm	9178551	9178552	9178553	9178555
100mm	9178511	9178512	9178513	9178515
150mm	9178561	9178562	9178563	9178565
200mm	9178521	9178522	9178523	9178525
250mm	9178571	9178572	9178573	9178575

Analyzing the Heat Levels of Spicy Foods

Using an Ultra C18 HPLC Column

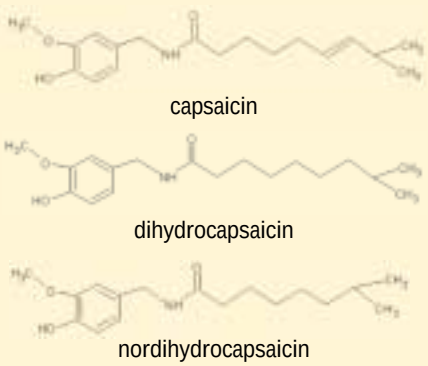
by Rebecca Wittrig, Ph.D., Food, Flavor, & Fragrance Innovations Group Leader

- ✔ Fast, reproducible separation of capsaicinoids
- ✔ Only minimal sample preparation needed

As interest in spicy foods grows, so does the need to test and classify products and raw materials for their heat levels. In 1912, Wilbur Scoville developed a method for quantitating the heat content of foods. A Scoville Heat Unit (SHU) is defined as the number of parts sugar water needed to neutralize the heat of one part sample extract (e.g., if the heat of a cayenne pepper is 30,000 SHU, that means 30,000 parts of sugar water are needed to dilute one part of cayenne pepper extract to the last point that hotness can be detected). However, this test is somewhat subjective because it relies on the tasters' palates and sensitivities. In addition, tasters can handle only a limited number of samples at one time before "fatiguing of the palate" occurs.[1] This makes it difficult to process a large number of samples in a reasonable amount of time, and can affect reproducibility of the tests.

Figure 1

Structures of three capsaicinoids commonly found in chili peppers



capsaicin

dihydrocapsaicin

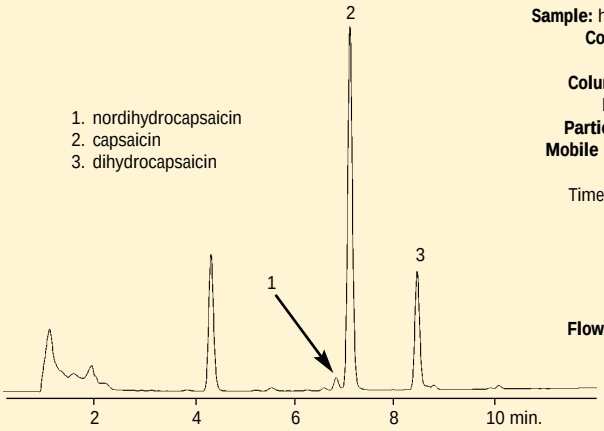
nordihydrocapsaicin

There are seven generally recognized capsaicinoids, three of which are shown in Figure 1. The capsaicinoid content, and thus the heat level, depends on the type of pepper, maturity, growing conditions, and processing methods. Starting in the 1970s, several analytical methods for heat level measurement were introduced. Of these, the HPLC procedures have provided the greatest specificity while requiring the least amount of sample preparation.[2] The American Spice Trade Association (ASTA) and the Association of Official Analytical Chemists (AOAC) have both published methods for the determination of capsaicinoids by HPLC.

AOAC Method 995.03[3] specifies the separation of the three target capsaicinoids responsible for the heat in chili peppers. This separation uses reversed phase HPLC on a C18 column followed by quantitation using either UV or fluorescence detection. The AOAC method is performed isocratically with a mobile phase consisting of 1% acetic acid in water:acetonitrile (60:40). Standardization is performed using synthetic capsaicin, N-vanillyl-*n*-nonanamide, and the relative amounts of nordihydrocapsaicin, capsaicin, and dihydrocapsaicin are calculated by applying the specified factors. Using the appropriate calculations where 1ppm of total capsaicinoids is approximately equal to 15 SHU, the heat index then can be calculated.

Figure 2

The Restek Ultra C18 Column achieves fast, reproducible separation of capsaicinoids.



Sample: habanero nugget extract; **Inj.:** 10µL;
Conc.: 0.8g sample in 200mL;
Solvent: ethanol;
Column: Ultra C18 (cat.# 9174565)
Dimensions: 150x4.6mm;
Particle size: 5µm; **Pore size:** 100Å
Mobile phase: A: 1% acetic acid in water
B: acetonitrile

Time (minutes):	% B
0	50
2	50
9	80
12	80
13	50

Flow: 1.0mL/min.; **Temp.:** ambient;
Det.: UV @ 280nm

Using a gradient elution program, an efficient separation of the three target capsaicinoids is achieved with the Restek Ultra C18 column (Figure 2). The high percentage of organic at the end of the run helps to elute any strongly adsorbing species present in the samples and decreases analysis time. This HPLC procedure provides an objective measurement of the heat level of a wide range of samples. By using a Restek Ultra C18 column and a gradient elution program, the analysis can be performed quickly and reproducibly with only minimal sample preparation. The selectivity and lot-to-lot reproducibility of the Ultra C18 column make it an excellent choice for heat level measurements.

For more detail on HPLC detection of heat level, including applications showing other products and a table comparing hotness rankings by HPLC for selected sauces, please request Applications Note lit. cat.#59199.

✔ Ultra C18 5µm HPLC Columns

For the complete HPLC column offering, refer to Restek's Annual Chromatography Products Catalog *(lit. cat.# 59960).*

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
30mm	9174531	9174532	9174533	9174535
50mm	9174551	9174552	9174553	9174555
100mm	9174511	9174512	9174513	9174515
150mm	9174561	9174562	9174563	9174565
200mm	9174521	9174522	9174523	9174525
250mm	9174571	9174572	9174573	9174575

for more info

Request Applications Note #59199

1. Bensinger, M. "How Hot is that 'Devil' Sauce?" Fiery Foods Magazine (1997), Sept/Oct.
3. Chiang, G.H., Journal of Food Science (1986) 51 (2), 499-503.
2. AOAC Official Methods of Analysis (2000), method 995.03.

Trident™ Direct Guard Column System

The Ultimate Combination of Convenience and Column Protection

by Greg France, HPLC Product Marketing Manager

- ✔ Direct connection eliminates connection tubing and extra dead volume
- ✔ Three levels of protection to fit your needs

Unlike other "one size fits all" guard systems, the Trident™ Direct system gives you the power to select the right level of protection for your analysis. The system offers three levels of protection and guard cartridges that are available in four dimensions with a variety of bonded phases to match your analytical column (Figure 1). The economical leak-free cartridge design provides an unprecedented combination of convenience, economy, and reliability.

The foundation of the Trident™ Direct system is a reusable direct connecting holder that easily attaches to any HPLC column using CPI- or Waters®-style end fittings.* The system is available in the following configurations to match different protection level needs: in-line filter (A), in-line filter with holder for 1cm guard cartridge (B), and in-line filter with holder for 2cm guard cartridge (C). The guard cartridges are available in both 2.1mm and 4.0mm IDs and are interchangeable with the appropriate length holder.

For protection against particulate matter only, use the Trident™ Direct high-pressure filter. For protection against particulate matter and sample impurities, use the Trident Direct 1cm holder and 1cm guard cartridges. This is the most popular configuration and is well suited for most applications. For protection against particulate matter and heavily contaminated samples, use the Trident Direct 2cm holder and 2cm guard cartridges.

Figure 1
The Trident™ Direct guard column system offers three levels of protection

(A) Trident™ Direct high-pressure filter
Protection against particulate matter

(B) Trident™ Direct 1cm guard cartridge holder with filter
Moderate protection against particulate matter and irreversibly-adsorbed compounds

(C) Trident™ Direct 2cm guard cartridge holder with filter
Maximum protection against particulate matter and irreversibly-adsorbed compounds

After you decide what level of protection and diameter guard cartridge is right for your application, you should choose the cartridge with a bonded phase that is the same or similar to your analytical column. The Allure™ and Ultra bonded phases are base-deactivated and compatible with virtually all silica-based analytical columns.

for more info

request Fast Facts # 59314.

✔ Trident™ Direct Guard Column System

Trident™ Direct	cat.#	ea.
High-pressure filter	25082	
1cm guard cartridge holder with filter	25084	
2cm guard cartridge holder with filter	25086	
Connection tip for Waters®-style end fittings	25088	

Replacement Frits for the Trident™ Filter	cat.#	5-pk.
Cap frits 4mm, 2.0µm	25022	
Cap frits 4mm, 0.5µm	25023	
Cap frits 2mm, 2.0µm	25057	

Guard Column Cartridges	(10 x 2.1mm) 3-pk.	(10 x 4.0mm) 3-pk.	(20 x 2.1mm) 2-pk.	(20 x 4.0mm) 2-pk.
Allure™ Acidix	916250212	916250210	916250222	916250220
Allure™ Basix	916150212	916150210	916150222	916150220
Allure™ C18	916450212	916450210	916450222	916450220
Allure™ PFP Propyl	916950212	916950210	916950222	916950220
Allure™ Silica	916050212	916050210	916050222	916050220
Ultra Amino	910750212	910750210	910750222	910750220
Ultra Aqueous C18	917850212	917850210	917850222	917850220
Ultra C1	910150212	910150210	910150222	910150220
Ultra C4	910250212	910250210	910250222	910250220
Ultra C8	910350212	910350210	910350222	910350220
Ultra C18	917450212	917450210	917450222	917450220
Ultra Cyano	910650212	910650210	910650222	910650220
Ultra IBD	917550212	917550210	917550222	917550220
Ultra PFP	917650212	917650210	917650222	917650220
Ultra Phenyl	910550212	910550210	910550222	910550220
Ultra Silica	910050212	910050210	910050222	910050220

**The standard PEEK® tip that comes with the Trident™ Direct systems is compatible with Parker, Swagelok, Upchurch, Valco, and other CPI-style fittings. To use the Trident™ Direct systems with Waters-style end fittings, the tip must be replaced with cat.# 25088.*

Restek's Environmental Commitment Remains Strong

You have probably seen our new packaging, and you may wonder if it is environmentally friendly. It sure is! The corrugated cardboard box and insert are completely recyclable, and we engineered the insert to virtually eliminate the use of styrofoam peanuts. Help us help the environment by recycling Restek packaging.

Analysis of PAHs

Using Rtx®-5Sil MS and Rtx®-CLPesticides2 Capillary Columns

by Gary Stidsen, Environmental Innovations Team Manager

- ✔ benzo(b)/benzo(k)flouranthene resolution
- ✔ Optimized conditions yield 18-minute analysis time

Analysis of polycyclic aromatic hydrocarbons (PAHs) is a very common method in environmental laboratories. US Environmental Protection Agency (EPA) Method 8100 requires gas chromatography/flame ionization detection (GC/FID) to quantitate PAHs found in extracts from soil, water, or biological samples. Confirmational analysis increases the confidence of proper identification and quantitation of the PAHs, and good resolution is necessary for proper quantitation. The most difficult compound pairs to resolve are benzo(b)/benzo(k)fluoranthene and indeno(1,2,3-cd)pyrene/dibenzo(a,h)anthracene. Short analysis time is another key consideration for most laboratories. By decreasing analysis time, sample throughput increases and the lab benefits from a cost savings.

For this analysis, the primary analytical stationary phase is a 5%diphenyl/95%dimethyl-polysiloxane polymer. The Restek Rtx®-5Sil MS column is an equivalent phase and is recommended for this analysis (Figure 1). The confirmational column recommended by Restek for this analysis is the Rtx®-CLPesticides2 column (Figure 2). Quantitative reliability for this analysis is maintained because the stationary phases differ in selectivity, resulting in retention time shifts of both PAHs and interference compounds.

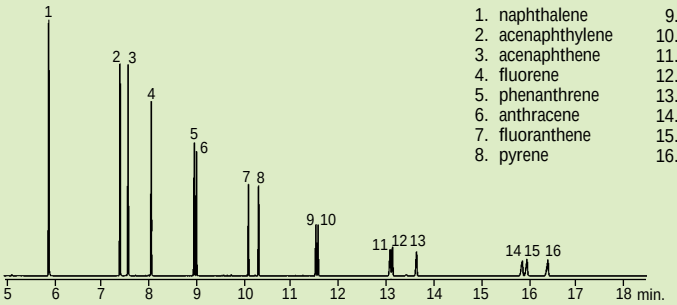
Resolution between benzo(b)fluoranthene and benzo(k)fluoranthene, and indeno(1,2,3,-cd)pyrene and dibenzo(a,h)anthracene is essential for quantitation when using an FID. To achieve excellent resolution of these peak pairs, the carrier gas, column flow rate, and temperature program must all be optimized. These three parameters should be optimized to increase throughput, too. To achieve even better quantitative reliability, it is recommended to clean sample extracts following EPA Method 3630 (silica gel) prior to analysis.

Optimizing the temperature program contributes to better resolution of closely eluting peak pairs and shortens analysis times. The temperature program and other conditions in Figures 1 and 2 achieve baseline resolution of indeno(1,2,3-cd)pyrene and dibenzo(a,h)anthracene, and excellent resolution of benzo(b)fluoranthene and benzo(k)fluoranthene, while still keeping the analysis time under 18 minutes. Because the temperature program for both columns is the same, the analysis can be run simultaneously on the primary and confirmation columns.

PAH analysis by US EPA Method 8100 can be improved by choosing the appropriate analytical columns and by optimizing the temperature program, carrier gas, and column flow rates. When operating under the conditions listed for Figures 1 and 2, the Rtx®-5Sil MS and the Rtx®-CLPesticides2 columns yield excellent resolution and short analysis times for PAHs.

Figure 1

The Rtx®-5Sil MS column exhibits excellent resolution of PAHs including benzo(b)/benzo(k)fluoranthene in less than 17 minutes.

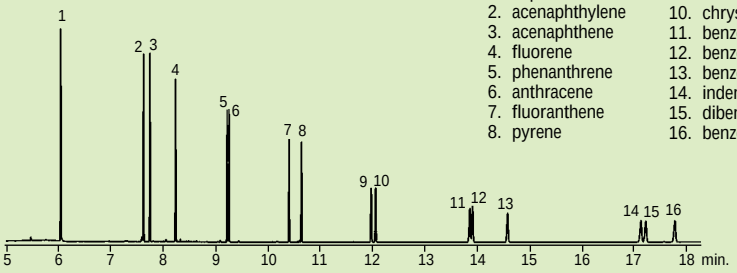


1. naphthalene
2. acenaphthylene
3. acenaphthene
4. fluorene
5. phenanthrene
6. anthracene
7. fluoranthene
8. pyrene
9. benzo(a)anthracene
10. chrysene
11. benzo(b)fluoranthene
12. benzo(k)fluoranthene
13. benzo(a)pyrene
14. indeno(1,2,3-cd)pyrene
15. dibenzo(a,h)anthracene
16. benzo(g,h,i)perylene

30m, 0.25mmID, 0.25µm Rtx®-5Sil MS (cat.# 12723); **Sample:** Method 610—Polynuclear Aromatic Hydrocarbons Mix (cat.# 31011); **Concentration:** 50ppm; **Solvent:** methylene chloride; **Sample size:** 1.0µL; **GC:** Thermo Trace 2000 Series; **Injector:** splitless @ 250°C; **Splitless hold time:** 2.0 min.; **Split vent flow:** 40cc/min.; **Carrier gas:** hydrogen (constant flow mode); **Column flow rate:** 4.0cc/min. @ 40°C; **Linear velocity:** 43cm/sec.; **Detector:** FID @ 340°C; **Make-up gas flow:** 40cc/min.; **Oven program:** 40°C (hold 2.0 min.) to 268° C @ 25°C/min. (hold 1.0 min.) to 330°C @ 5°C/min. (hold 10 min.)

Figure 2

The Rtx®-CLPesticides column is an excellent confirmational column for PAH analysis.



1. naphthalene
2. acenaphthylene
3. acenaphthene
4. fluorene
5. phenanthrene
6. anthracene
7. fluoranthene
8. pyrene
9. benzo(a)anthracene
10. chrysene
11. benzo(b)fluoranthene
12. benzo(k)fluoranthene
13. benzo(a)pyrene
14. indeno(1,2,3-cd)pyrene
15. dibenzo(a,h)anthracene
16. benzo(g,h,i)perylene

30m, 0.25mm ID, 0.25µm Rtx®-CLPesticides2 (cat.# 11323); **Sample:** Method 610—Polynuclear Aromatic Hydrocarbons Mix (cat.# 31011); **Concentration:** 50ppm; **Solvent:** methylene chloride; **Sample size:** 1.0µL; **GC:** Trace 2000 Series; **Injector:** splitless @ 250°C; **Splitless hold time:** 2.0 min.; **Split vent flow:** 40cc/min.; **Carrier gas:** hydrogen (constant flow mode); **Column flow rate:** 4.0cc/min. @ 40°C; **Linear velocity:** 43cm/sec.; **Detector:** FID @ 340°C; **Make-up gas flow:** 40cc/min.; **Oven program:** 40°C (hold 2.0 min.) to 268°C @ 25°C/min. (hold 1.0 min.) to 330°C @ 5°C/min. (hold10 min.)

for more info

Request Applications Note # 59196.

For the complete Rtx®-5Sil MS and Rtx®-CLPesticides2 columns offering, refer to Restek's Annual Chromatography Products Catalog *(lit. cat.# 59960).*

✔ Rtx®-5Sil MS Columns

Temp limits: -60 to 330/350°C

ID (mm)	df (µm)	30-Meter
0.25	0.25	12723
0.32	0.25	12724

✔ Rtx®-CLPesticides2 Columns

Temp limits: -60 to 310/330°C

ID (mm)	df (µm)	30-Meter
0.25	0.20	11323
0.32	0.25	11324

Rtx®-OPPesticides2

GC Column for the Analysis of Organophosphorous Pesticides

by Frank Dorman, Ph.D., Environmental Innovations Team Chemist

- ✔ No coelutions between compounds with similar mass spectra—column can be used alone with MS
- ✔ Confirmational column for Rtx®-OPPesticides

Several years ago Restek introduced the Rtx®-OPPesticides column, which was the first column designed specifically for the analysis of organophosphorus pesticides according to US Environmental Protection Agency (EPA) Method 8140. While this column was clearly superior to any other commercially available stationary phase for this separation, the need for confirmational analysis still made the choice of column pair unclear.

The Rtx®-OPPesticides column was designed using early versions of computer modeling techniques and, since then, column requirements have changed as the EPA has added more target compounds.[1] EPA Method 8141A now requires 53 compounds to be completely resolved using a two-column system. Additionally, for laboratories preferring to use gas chromatography/mass spectrometry (GC/MS) for this analysis, it is important to have no coelution between compounds with similar mass spectra.

To address these requirements, Restek developed a new column using a more sophisticated modeling technique (i.e., a computer program helps determine optimum phase structure and column dimensions based on the specific separation requirements). This new Rtx®-OPPesticides2 column has a 330°C maximum operating temperature, low bleed, and excellent inertness. It completely separates all of the target compounds in US EPA Method 8141A when used in combination with the Rtx®-OPPesticides column (Figure 1). This analysis results in only two coelutions on the Rtx®-OPPesticides2 column: thionazin and tributyl phosphate, and parathion-ethyl and trichloronate. However, these compounds do not coelute on the Rtx®-OPPesticides column, so a complete separation is attainable using the column pair.

When using a nitrogen phosphorus detector (NPD) or a flame photometric detector (FPD), the 0.32mm ID Rtx®-OPPesticides and Rtx®-OPPesticides2 column pair is optimal; use direct injection via a single injection port and split the sample into the two columns with a glass 'Y' PressTight® connector. For GC/MS analysis, the Rtx®-OPPesticides2 column can be used alone because the two coelutions have dissimilar mass spectra, so different quantitation ions can be used to resolve them.

In summary, the Rtx®-OPPesticides and Rtx®-OPPesticides2 column pair is ideal for the analysis of organophosphate pesticides. These columns were designed using computer-modeling techniques to be superior to any other commercially available stationary phases for this separation. If you are involved in the separation of organophosphate pesticides and would like more information, please contact the Restek Technical Service Team at 814-353-1300 or 800-356-1688, ext. 4.

References
1. Frank L. Dorman, Paul D. Schettler, Christopher M. English, and Michael J. Feeney, LC GC, 18 (9), 928-934, 2000.
References not available from Restek.

for more info

Request the *Rtx®-OPPesticides2 Column* brochure (lit. cat.# 59275).

✔ Rtx®-OPPesticides Columns

Temp limits: -20 to 310/330°C

ID (mm)	df (µm)	20-Meter	30-Meter
0.18	0.25	56898	—
0.25	0.40	—	55239
0.32	0.50	—	11239
0.53	0.83	—	11240

✔ Rtx®-OPPesticides2 Columns

Temp limits: -20 to 310/330°C

ID (mm)	df (µm)	20-Meter	30-Meter
0.18	0.20	11244	—
0.25	0.25	—	11243
0.32	0.32	—	11241
0.53	0.50	—	11242

✔ "Y" Press-Tight® Connectors

Type	ea.	3-pk.
straight	20405	20406
angled	20403	20404

✔ 8140/8141 OP Pesticide Calibration Mix A

azinphos methyl	fenthion
bolstar (sulprofos)	merphos
chlorpyrifos	methyl parathion
coumaphos	mevinphos
demeton, O and S	naled
diazinon	phorate
dichlorvos	ronnel
disulfoton	stirofos
ethoprop	tokuthion (prothiofos)
fensulfothion	trichloronate

200µg/mL each in hexane/acetone (95%/5%), 1mL/ampul

Ea.	5-pk.	10-pk.
32277	32277-510	—
with data pack		
32277-500	32277-520	32377

✔ 8141 OP Pesticide Calibration Mix B

dimethoate	parathion
EPN	sulfotepp
malathion	TEPP
monocrotophos	

200µg/mL each in hexane/acetone (95%/5%), 1mL/ampul

Ea.	5-pk.	10-pk.
32278	32278-510	—
with data pack		
32278-500	32278-520	32378

✔ Method 8140/8141 Internal Standards & Surrogates

1,000µg/mL each in acetone, 1mL/ampul

NPD Detector

Internal Standard: *1-bromo-2-nitrobenzene*
Surrogate: *4-chloro-3-nitrobenzotrifluoride*

1-bromo-2-nitrobenzene Standard

Ea.	5-pk.	10-pk.
32279	32279-510	—
with data pack		
32279-500	32279-520	32379

4-chloro-3-nitrobenzotrifluoride Standard

Ea.	5-pk.	10-pk.
32282	32282-510	—
with data pack		
32282-500	32282-520	32382

FPD Detector

Internal Standard: *none recommended*
Surrogate: *tributylphosphate and triphenylphosphate*

Tributylphosphate Standard

Ea.	5-pk.	10-pk.
32280	32280-510	—
with data pack		
32280-500	32280-520	32380

Triphenylphosphate Standard

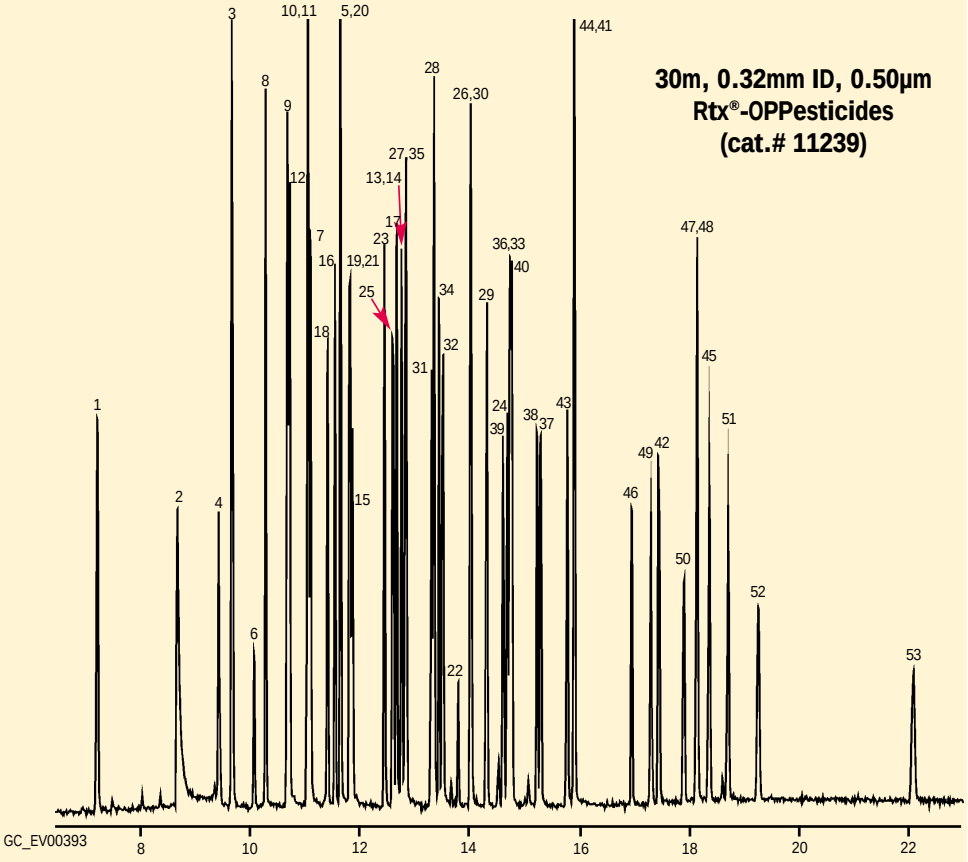
Ea.	5-pk.	10-pk.
32281	32281-510	—
with data pack		
32281-500	32281-520	32381

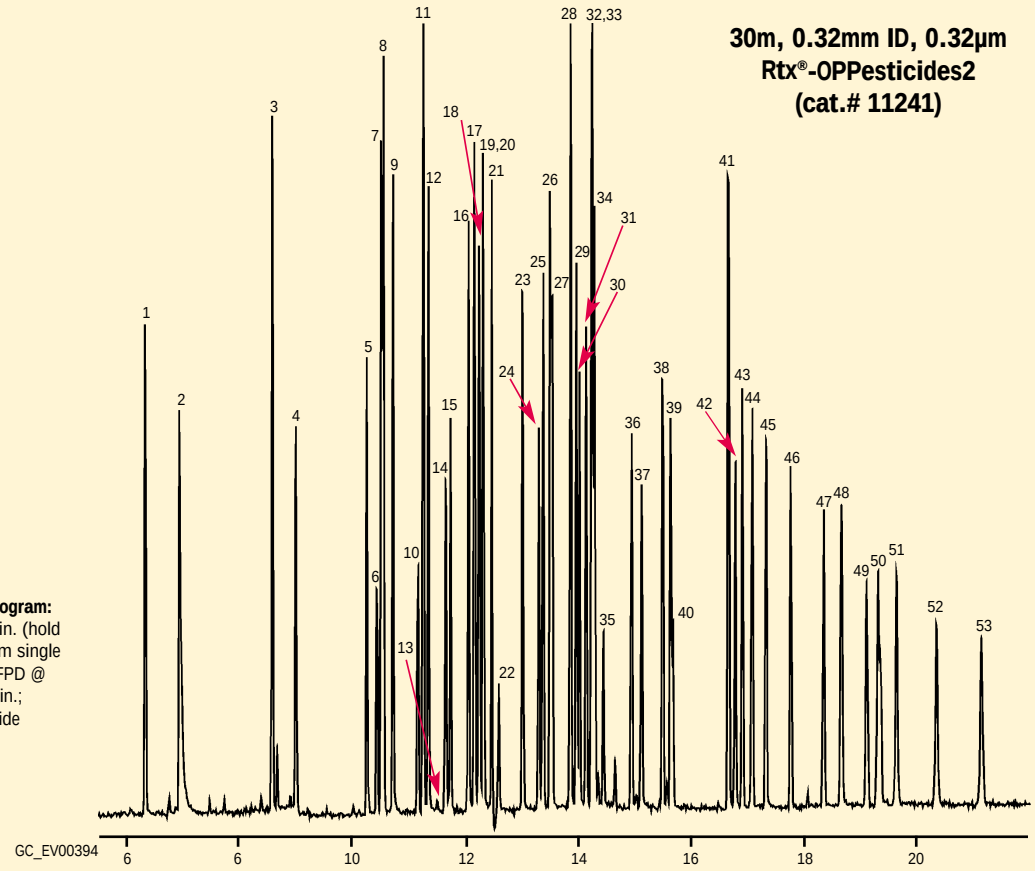
Figure 1

The Rtx®-OPPesticides and Rtx®-OPPesticides2 column pair completely separates all target compounds in US EPA Method 8141A.

dual-column injector
Detector: FPD @ 250°C;
Dead time: @ 80°C = 0.98 min.

1. dichlorvos
2. hexamethylphosphoramide
3. mevinphos
4. trichlorfon
5. TEPP
6. demeton-*o*
7. tributyl phosphate (SS)
8. thionazin
9. ethoprop
10. naled
11. sulfotepp
12. phorate
13. dicrotophos
14. monocrotophos
15. demeton-s
16. terbufos
17. dimethoate
18. diazinon
19. dioxathion
20. fonophos
21. disulfoton
22. phosphamidon isomer (breakdown product)
23. dichlorofenthion
24. phosphamidon
25. chlorpyrifos methyl
26. parathion-methyl
27. ronnel
28. aspon
29. fenitrothion
30. malathion
31. chlorpyrifos
32. trichloronate
33. parathion-ethyl
34. fenthion
35. merphos
36. chlorfenvinphos
37. crotoxyphos
38. stirofos
39. tokuthion
40. merphos oxon (breakdown product)
41. ethion
42. fensulfothion
43. bolstar
44. carbophenothion
45. famphur
46. triphenyl phosphate (SS)
47. EPN
48. phosmet
49. leptophos
50. tri-*o*-cresyl phosphate
51. azinphos-methyl
52. azinphos-ethyl
53. coumaphos





GC: splitless, purge on 1.0 min.; **Oven program:** 80°C (hold 0.5 min.) to 280°C @ 12°C/min. (hold 10 min.); **Injector:** 200°C, **Inlet liner:** 4mm single gooseneck Siltek™ inlet liner; **Detector:** FPD @ 250°C; **Dead time:** @ 80°C = 1.03 min.; **Injection:** 1µL, 100ng/mL OP Pesticide Calibration Mix

Analytical Reference Materials

For Volatile Organic Compounds such as US EPA Method 8260B

by Christopher Cox, R&D Manager

✔ Fewest Mixtures needed for calibration
✔ Mixtures divided for maximum stability
✔ Contains the most commonly run compounds

502.2 Calibration Mix # 1

bromomethane
chloroethane
chloromethane
dichlorodifluoromethane
trichlorofluoromethane
vinyl chloride

2000µg/mL each in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30042	30042-510	—
with data pack		
30042-500	30042-520	30142

VOA Calibration Mix # 1 (ketones)

acetone
2-butanone
2-hexanone
4-methyl-2-pentanone

5,000µg/mL each in P&T methanol
1mL per ampule

Ea.	5-pk.	10-pk.
30006	30006-510	—
with data pack		
30006-500	30006-520	30106

1,2-Dichlorotetrafluoroethane Mix (Freon® 114)

2000ppm in P&T methanol
1mL per ampule

Ea.	5-pk.	10-pk.
30476	30476-510	—
with data pack		
30476-500	30476-520	30576

CA Oxygentates Mix

diisopropyl ether	2000µg/mL
ethyl-*tert*-butyl ether	2000µg/mL
tert-amyl methyl ether	2000µg/mL
tert-butyl alcohol	10,000µg/mL
methyl-*tert*-butyl ether	2000µg/mL

in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30465	30465-510	—
with data pack		
30465-500	30465-520	30565

Ethanol Mix

ethanol

10,000µg/mL in deionized water
1mL per ampul

Ea.	5-pk.	10-pk.
30466	30466-510	—
with data pack		
30466-500	30466-520	30566

Acrolein Mix

10,000µg/mL in P&T methanol

Ea.	5-pk.	10-pk.
30478	30478-510	—
with data pack		
30478-500	30478-520	30578

8260B Acetate Mix

vinyl acetate
ethyl acetate
isopropyl acetate
n-propyl acetate
n-butyl acetate

2000µg/ml each in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30477	30477-510	—
with data pack		
30477-500	30477-520	30577

8260A Surrogate Mix

4-bromofluorobenzene
dibromofluoromethane
1,2-dichloroethane-d4
toluene-d8

2500µg/mL each in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30240	30240-510	—
with data pack		
30240-500	30240-520	30340

8260A Internal Standard Mix

chlorobenzene-d5
1,4-dichlorobenzene-d4
fluorobenzene

2500µg/mL each in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30241	30241-510	—
with data pack		
30241-500	30241-520	30341

4-Bromofluorobenzene Mix (Tuning Mix)

2500µg/mL in P&T methanol
1mL per ampule

Ea.	5-pk.	10-pk.
30067	30067-510	—
with data pack		
30067-500	30067-520	30167

8260B Matrix Spike Mix

1,1-dichloroethene
trichloroethylene
chlorobenzene
toluene
benzene

2500µg/mL in P&T methanol
1mL per ampule

Ea.	5-pk.	10-pk.
30479	30479-510	—
with data pack		
30479-500	30479-520	30579

for *more* info

8260B Calibration Mix # 1

1,1-dichloroethene
carbon disulfide
1,1,2-trichlorotrifluororethane (Freon® 113)
iodomethane
allyl chloride
methylene chloride
trans-1,2-dichloroethene
1,1-dichloroethane
acetonitrile
acrylonitrile
cis-1,2-dichloroethene
2,2-dichloropropane
bromochloromethane
chloroform
carbon tetrachloride
tetrahydrofuran
methyl acrylate
1,1,1-trichloroethane
1,1-dichloropropene
propionitrile
benzene
methacrylonitrile
1,2-dichloroethane
isobutyl alcohol
trichloroethene
dibromomethane
1,2-dichloropropane
bromodichloromethane
methyl methacrylate
2-chloroethyl-vinyl-ether
chloroprene
cis-1,3-dichloropropene
toluene
tetrachloroethene
trans-1,3-dichloropropene
2-bromo-1-chloropropane
1,1,2-trichloroethane
ethyl methacrylate
dibromochloromethane
1,3-dichloropropane
1,2-dibromoethane
chlorobenzene
ethylbenzene
1,1,1,2-tetrachloroethane
m-xylene
p-xylene
o-xylene
stryrene
bromoform
isopropylbenzene
cis-1,4-dichloro-2-butene
bromobenzene
1,4-dichlorobutane
n-propylbenzene
1,1,2,2-tetrachloroethane
2-chlorotoluene
1,2,3-trichloropropane
1,3,5-trimethylbenzene
trans-1,4-dichloro-2-butene
4-chlorotoluene
tert-butylbenzene
pentachloroethane
1,2,4-trimethylbenzene
sec-butylbenzene
p-isopropyltoluene
1,3-dichlorobenzene
1,4-dichlorobenzene
n-butylbenzene
1,2-dichlorobenzene
1,2-dibromo-3-chloropropane
nitrobenzene
hexachlorobutadiene
1,2,4-trichlorobenzene
naphthalene
1,2,3-trichlorobenzene
1,4-dioxane
2-chloroethanol
diethyl ether
2-nitropropane

2000µg/mL each in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30475	30475-510	—
with data pack		
30475-500	30475-520	30575

Request *US EPA Method 8260B Standards* Fast Facts for short lists (lit. cat.# 59332).

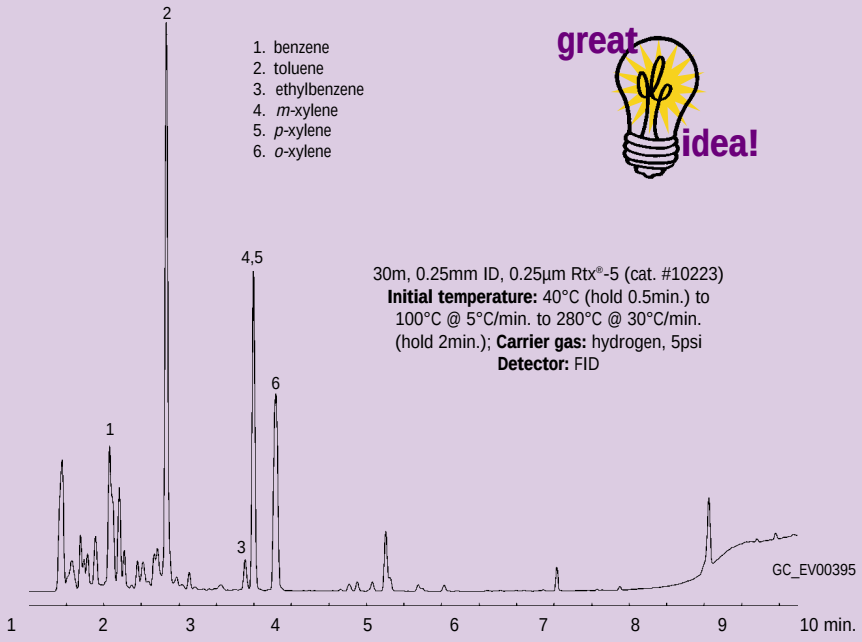
MESI—Membrane Extraction Sorbent Interface

by Rick Morehead, Innovations Chemist

MESI is a new sample preparation technique that uses a permeable membrane to extract volatile organic compounds (VOCs) from various matrices and a sorbent trap to concentrate these compounds prior to analysis by gas chromatography. MESI has been designed to be an easy to use "solventless" extraction technique. It can be used for fast, routine analyses or in long-term or continuous monitoring operations.

MESI extraction is based on the partitioning of VOCs across a non-polar membrane approximately 25µm thick. A small section of membrane material is mounted in a holder that allows one side of the membrane to be exposed to the sample matrix. The other side of the membrane is continuously swept with a carrier gas. As volatile compounds come in contact with the membrane surface, migration through the membrane begins. Compounds are then picked up by the carrier gas on the other side of the membrane and passed along to an adsorbent trap. After the sampling or extraction period is complete, the trap is very rapidly heated to desorb all of the compounds that have been collected on the adsorbent material. Desorbed analytes are picked up in the flow of the carrier gas and are transferred to the inlet of the GC analytical column for separation.

Figure 1
MESI used to determine hydrocarbons at low ppb levels.



Adsorbent traps used for MESI are made from small diameter Silcosteel® tubing packed with traditional trapping materials like Carboxen®, Tenax® or other divinylbenzene resins. The capacity of the trap is related to the type of adsorbent used as well as the amount of material used. Traps are desorbed by passing a short pulse of DC current across the trap and resistively heating the trap.

Figure 1 shows the analysis of a soil sample for the presence of gasoline range organics (GROs). A 1gm soil sample was spiked with 1ppm of gasoline and sampled for 2 minutes. Analytes were extracted by the membrane and concentrated on the trap, then were desorbed and analyzed on an Rtx®-1 column. Good peak shape and response can be achieved by optimizing the sampling time and the desorption conditions.

MESI can be used in a variety of sampling applications. Short turnaround time can be obtained for higher concentration applications. The cycle time for heating and cooling the trap can be as short as thirty seconds, so samples can be collected and desorbed in rapid succession to perform on-line monitoring of processes or streams. For lower concentration samples, sampling time can be extended in order to maximize the response for the analytes of interest. MESI can be used as a bench-top accessory for most GCs or, because of its compact design, can be used as part of a field portable unit for on-site analysis.

MESI also can be used with a variety of sample types. Gas, liquid, and solid samples can be analyzed by exposing the sample to the membrane using minor variations in the hardware that holds the sample.

MESI is a low-cost alternative to other sample preparation techniques. It is easy to use and automate, while being sensitive enough for most applications.

This product is still in development. If you would like to discuss potential applications, please call Technical Service at 800-356-1688 or 814-353-1300, ext. 4, or contact your local Restek representative.

Questions?

Contact us at
support@restekcorp.com.
The industry's best technical service will be glad to help you with Plus 1™ service!

Peak Performers

New GC Accessories to Make Your Analyses Easier and More Reproducible

by Gary Barone, GC Accessories Product Marketing Manager

Thermal Gas Purifiers

- ✔ Removes oxygen, water, carbon monoxide, carbon dioxide and hydrocarbons
- ✔ Purity in ppb levels
- ✔ Mass Spec. purity carrier gas produced
- ✔ Dual-tube model purifiers double capacity at less than double the price
- ✔ Welded end-fittings on getter tubes eliminate leaks
- ✔ Packed with reactor-grade, pure getter material for maximum efficiency and no contamination

Introducing Restek's line of re-engineered thermal gas purifiers. This line of purifiers works by producing a chemical reaction between impurities in the carrier gas stream and the getter material. Because the reaction is non-reversible, there is no possibility of contaminants breaking through the thermal gas purifier.

Gas purification is very economical when using a thermal gas purifier. After initial installation cost, getter tubes only require changing every year; heavy use and very impure feed gas may require more frequent getter tube replacement.

Restek Single-Tube Thermal Gas Purifier, 110 Volt:

⅛" Fittings: cat.# 21496, (ea.)
¼" Fittings: cat.# 21497, (ea.)

Restek Dual-Tube Thermal Gas Purifier, 110 Volt:

⅛" Fittings: cat.# 21498, (ea.)
¼" Fittings: cat.# 21499, (ea.)

Replacement Straight Getter Tubes:

⅛" Fittings: cat.# 21661, (ea.)
¼" Fittings: cat.# 21660, (ea.)

Introducing Super-Clean™ gas filters

- ✔ Full glass/metal design
- ✔ High-purity output (six-9s=99.9999% purity)
- ✔ Features "quick connect" for quick cartridge changes

Special promotional offer to get you started with Super-Clean™ Gas Filters!

Buy an oxygen/moisture/hydrocarbon triple filter and receive a mounting baseplate with ⅛" inlet and outlet fittings for no additional charge.

(Offer good through July 31, 2001): cat.# 22024, (kit)

This line of Super-Clean gas filters is the latest in cartridge gas filtration. Cartridge systems make changing gas filters quick and easy. The system works using a baseplate that allows cartridges to be exchanged without introducing oxygen. The spring-loaded check valves seal when a filter is removed and open only when a new filter has been locked in place. There is no longer a need for loosening and tightening fittings every time a trap is changed.

The Triple Filter model is ideal for carrier gas purification. This trap contains the oxygen, moisture, and hydrocarbon scrubber in one cartridge. The gas purity of your carrier gas through the Triple Filter is better than six-9s, ideal for mass spectrometry and for protecting your columns against damage.

The Fuel Gas Filter cartridge is perfect for purifying FID fuel gases, removing both moisture and hydrocarbons. Using the Fuel Gas Filter for FID hydrogen and air will produce a stable baseline, improving overall FID reproducibility and sensitivity.

Single filters for oxygen, moisture, and hydrocarbon; and 2- or 3-position baseplates are available from stock.

Carrier Gas Cleaning Kit
Includes mounting baseplate, ⅛" inlet/outlet fittings, and oxygen/moisture/hydrocarbon triple filter: cat.# 22019, (kit)
Replacement oxygen/moisture/hydrocarbon triple filter: cat.# 22020, (ea.)

Fuel Gas Purification Kit
Includes mounting baseplate, ⅛" inlet/outlet fittings, and hydrocarbon/moisture filter: cat.# 22021, (kit)
Replacement fuel gas hydrocarbon/moisture filter: cat.# 22022, (ea.)
Replacement O-rings for baseplate (contains 10 lg. & 10 sm. o-rings): cat.# 22023, (20-pk.)
To adapt baseplates for ¼" fittings use a ⅛" to ¼" tube-end union: cat.# 21833, (5-pk.)

Brass capillary nuts for Agilent 5890/6890 GCs

- ✔ Eliminates sticking or crossed threads
- ✔ Use for either compact or standard 1/16" ferrules

For use with "short" Agilent-style ferrules: cat.# 21878, (2-pk.) $25
For use with standard 1/16" -type ferrules: cat.# 21879, (2-pk.)

Sapphire scribe

- ✔ Cuts fused silica tubing
- ✔ Results in a good, square and clean cut

cat.# 20182, (ea.)

FIX IT laboratory Swiss knife

- ✔ Every GC analyst should have one!
- ✔ Genuine Swiss Army quality with 5-year warranty
- ✔ Includes a magnifying glass to check column end cuts
- ✔ 15-function tool including inlet liner remover, screwdrivers, scissors, blade, and tweezers

cat.# 23013, (ea.)

Inlet liner removal tool

- ✔ Easily remove liners from injectors
- ✔ Made from high-temperature silicone
- ✔ Won't chip or crack the liner

cat.# 20181, (3-pk.)

GC accessory organizer

- ✔ Ideal for organizing GC accessories and supplies
- ✔ Built-in syringe and vial holders
- ✔ Mounts on the GC for easy access
- ✔ Includes all mounting hardware

For Agilent 5890/6890 GCs: cat.# 22681, (qty.)
For Varian GCs: cat.# 22682, (qty.)

Connect Fused Silica Capillary Columns with New MXT® Unions

- ✔ Low-dead-volume, leak-free connection
- ✔ Reusable
- ✔ Silcosteel® treatment ensures maximum inertness
- ✔ Ideal for connecting guard columns and transfer lines
- ✔ Usable to oven temperatures of 350°C
- ✔ Available in union and "Y" configurations

Previously only metal tubing could benefit from an easy-to-use MXT® connector. Now the MXT® connector can be used with fused silica capillary columns because of the Valcon polyimide 1/32" one-piece fused silica adaptor. This unique graphite-reinforced composite allows capillary columns to slide and be locked into place simply by loosening and tightening the MXT® union 1/32" fitting.

MXT®-Union Connector Kits

Each kit contains the MXT® union, 2- 1/32" nuts and 2 one-piece fused silica adaptors

For 0.53mm columns: cat.# 21384, (kit)
For 0.32mm columns: cat.# 21385, (kit)
For 0.25mm columns: cat.# 21386, (kit)

MXT® "Y"-Union Connector Kits

Each kit contains the MXT® union, 3- 1/32" nuts and 3 one-piece fused silica adaptors

For 0.53mm columns: cat.# 21387, (kit)
For 0.32mmcolumns: cat.# 21388, (kit)
For 0.25mmcolumns: cat.# 21389, (kit)

Replacement One-Piece Fused Silica Adaptors for Fused Silica Capillary Columns

For 0.25mm columns: cat.# 20137, (5-pk.)
For 0.32mm columns: cat.# 20140, (5-pk)
For 0.53mm columns: cat.# 20141, (5-pk)
Replacement 1/32" nuts: cat.# 20389, (5-pk)

Expanded Line of Stock Sulfinert™ and Silcosteel® Fittings

A full line of 1/16", 1/8" and 1/4" fittings is now available Sulfinert™ or Silcosteel® treated direct from Restek. Because of expanding applications for these coatings, we have received many requests for a broader product offering. If you still do not see everything you need, contact us for information on custom coating services.

Fitting	Size	Sulfinert™	Silcosteel®
 **Unions**	1/16"	22520	20510
	1/8"	22521	20511
	1/4"	22522	20512
 **Tees**	1/16"	22526	20513
	1/8"	22527	20514
	1/4"	22528	20515
 **Reducing Fittings**	1/16" to 1/8"	22523	20519
	1/16" to 1/4"	22524	20520
	1/8" to 1/4"	22525	20521
 **Elbows**	1/16"	22529	20516
	1/8"	22530	20517
	1/4"	22531	20518
 **Plug**	1/16"	21539	21518
	1/8"	21540	21519
	1/4"	21541	21520
 **Cross**	1/8"	21542	21521
	1/4"	21543	21522
 **Tube End Reducer**	1/8" to 1/16"	21544	21523
	1/4" to 1/16"	21545	21524
	1/8" to 1/4"	21546	21525
	1/4" to 1/8"	21547	21526
 **Port Connectors**	1/8"	21548	21527
	1/4"	21549	21528
	1/8" to 1/4"	21550	21529
 **Compression to NPT Male Connectors**	1/8" to 1/8" NPT	21551	21530
	1/4" to 1/4" NPT	21552	21531
	1/16" to 1/8" NPT	21553	21532
	1/8" to 1/4" NPT	21554	21533
	1/4" to 1/8" NPT	21555	21534
 **Compression to NPT Female Connectors**	1/8" to 1/8" NPT	21556	21535
	1/4" to 1/4" NPT	21557	21536
	1/4" to 1/8" NPT	21558	21537
	1/8" to 1/4" NPT	21559	21538

Sulfinert™ Tubing

For the complete Sulfinert™ and Silcosteel® tubing offering, refer to Restek's Annual Chromatography Products Catalog *(lit. cat.# 59960).*

Sulfinert™ Tubing (304 Welded Stainless Steel)		***Price-per-foot by length***			
ID, OD	**cat.#**	**5-24 ft.**	**25-199 ft.**	**200-399 ft.**	**>400 ft.**
0.040" (1.02mm), 1/16" (1.59mm)	22505				
0.085" (2.16mm), 1/8" (3.18mm)*	22506				
0.210" (5.33mm), 1/4" (6.35mm)*	22507				

Sulfinert™ Tubing (316 Seamless Stainless Steel)		***Price-per-foot by length***			
ID, OD	**cat.#**	**5-24 ft.**	**25-199 ft.**	**200-399 ft.**	**>400 ft.**
0.055" (1.40mm), 1/8" (3.18mm)**	22508				
0.180" (4.57mm), 1/4" (6.35mm)**	22509				

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

RESTEK
Behind the Scenes

Restek-
The Company
Chromatographers Trust

You may have noticed our new advertisements describe Restek as, "The Company Chromatographers Trust." What does this mean to you, our customer? Well, it's fairly simple. Restek wants to supply you with products that perform better than expected, and service that is responsive and knowledgeable. You can trust what we say and what our products do. That's part of our Plus 1™ customer service commitment as well.


CIS Team


Customer Service Team

Restek is employee-owned. We are responsible to our customers and our employees, not to an impersonal parent company or external shareholders. This allows our employees the freedom to make decisions based on the needs of our customers.


Technical Service Team


Shipping & Assembly Team

While other chromatography suppliers are slowing down, Restek is growing because we have the internal strength and agility to adapt to your needs. Where else can you find such a strong Research and Development department? These scientists design new HPLC and GC columns, new lab accessories, new applications; and they take turns answering customer questions as part of our real-world technical service team. Where else can you find a sales force that has such chromatography experience and dedication to your satisfaction? Where else can you find chromatography products and service you can trust? Only from Restek.

If you've had an exceptional Restek experience, please let us know. And, likewise, if you can suggest an improvement, please let us know that, too.

Restek Ordering Online

Go to **www.restekcorp.com** to order your chromatography supplies without picking up the phone or leaving your desk! Don't forget we also offer technical service and literature online, too.

New Literature

Restek has an extensive library of free technical literature. Call 800-356-1688 or 814-353-1300, ext. 4. We'll be glad to help you out!

Applications Notes

Analysis of Vanillin & Ethyl Vanillin by HPLC (59186)
Optimizing Volatiles by GC/MS using a Rtx-VMS (59191)
EPA 8100 Analysis of PAHs on a Rtx-5Sil MS (59196)
Optimizing CG Analysis of Ethylene Glycol in Water (59187)
Separating *m*- and *p*-Xylene Isomers by US EPA 8360 (59190)

Minicatalogs

Analyzing Foods, Flavors, & Fragrances by GC and HPLC (59260)
Packed Columns (59986A)
HPLC Columns & Accessories (59241)
Genuine Restek Replacement Parts (59627B)

Fast Facts: At a Glance Product Info.

Air Standards (59276)

New Product Brochures, misc.

GC Wall Chart (59668A)
Chiral GC Columns (59242)
Sulfinert Metal Passivation (59203)

New Restek Wizards

Join us in welcoming the following new members to the Restek family:

Randy Emel, Applied Technology Technician
Stacy Finefrock, Accountant
Rick Fleck, Customer Service Representative
Wesley Heaton, Incoming Inspection
Marian Koch, Customer Service Representative
Maggie McCartin, HR Administrative Assistant
Jean Voigt, Technical Service Specialist
Dan Watson, Quality Systems Coordinator

ISO 9001 REGISTERED

Lit. Cat.# 59418

Please direct your comments on this publication to Kristin Dick, Editor, at kristind@restekcorp.com or call Restek, ext. 2313.

RESTEK

the RESTEK Advantage
Innovators of High Resolution Chromatography Products

Pinnacle II™

A New Line of HPLC Columns from Restek

new

by Greg France, HPLC Product Marketing Manager

- ✔ Featuring Restek silica—manufactured, bonded, and QA tested in-house.
- ✔ Wide range of applications: environmental, pharmaceutical, nutraceutical, foods, flavors, and fragrances.
- ✔ Currently available with C18, C8, phenyl, and cyano stationary phases.

Pinnacle II™ columns are manufactured using Restek silica, which features a controlled particle size distribution and spherical shape. It is available in 5µm particle size, with a pore diameter centered at 110Å, and a surface area of ~180m²/g. These silica particles address the growing demand for narrower columns and faster flow rates in an attempt to decrease analyses times. The phases offer similar selectivity to the original line of Pinnacle™ columns.

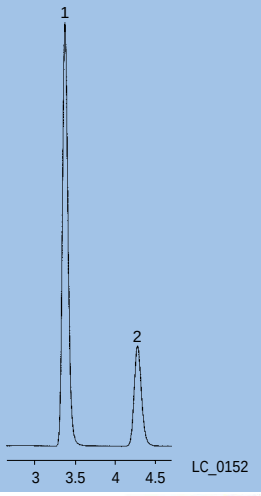
With the design of Pinnacle II™ columns, Restek achieves complete control over the HPLC column manufacturing chain. We can go a step further than most to ensure consistent quality and reproducibility because we manufacture the silica, perform the stationary phase bonding and column packing in-house, and apply our stringent quality testing throughout the whole process. The ability to consistently manufacture silica at a specified pore volume, surface area, pore size distribution, and particle size is part of the equation in providing columns that achieve reproducible analyses.

Pharmaceutical Applications

Notice how a Pinnacle II™ C18 column provides good peak shape for acetaminophen and codeine phosphate (Figure 1). You'll get this level of quality in your results, from analysis to analysis.

Figure 1

The separation of acetaminophen and codeine phosphate on a Pinnacle II™ C18 column shows symmetrical peak shape.



Peak List	**Conc.**
1. acetaminophen	1200µg/mL
2. codeine phosphate	246µg/mL

Sample:	
Inj.:	1.0µL
Solvent:	mobile phase
Column:	**Pinnacle II™ C18**
Catalog #:	9214565
Dimensions:	150x4.6mm
Particle size:	5µm
Pore size:	110Å
Conditions:	
Mobile phase:	10mM potassium phosphate monobasic with 0.1% v/v TEA, pH 3.0:acetonitrile (90:10)
Flow:	1.0mL/min.
Temp.:	27°C
Det.:	UV @ 210nm

in this issue

RESTEK
www.restekcorp.com

Summer'01
INTERNATIONAL

Pinnacle II™

New HPLC Columns, cont.

RESTEK Exclusive!

Nutraceutical and Food Applications

The Institute for Nutraceutical Advancement (INA) has published a series of methods for the determination of active compounds in nutraceutical products. Pinnacle II™ HPLC columns exhibit excellent peak shapes and reproducibility for natural product analyses (Figure 2).

As society's taste in spicy foods grows, so does the need to test and classify products and raw materials for their heat levels. AOAC Method 995.03 specifies the separation of the three target capsaicinoids responsible for the heat in chili peppers. Using a gradient elution program, an efficient separation of these compounds is achieved with the Pinnacle II™ C18 column (Figure 3).

Many New Phases Coming Soon!

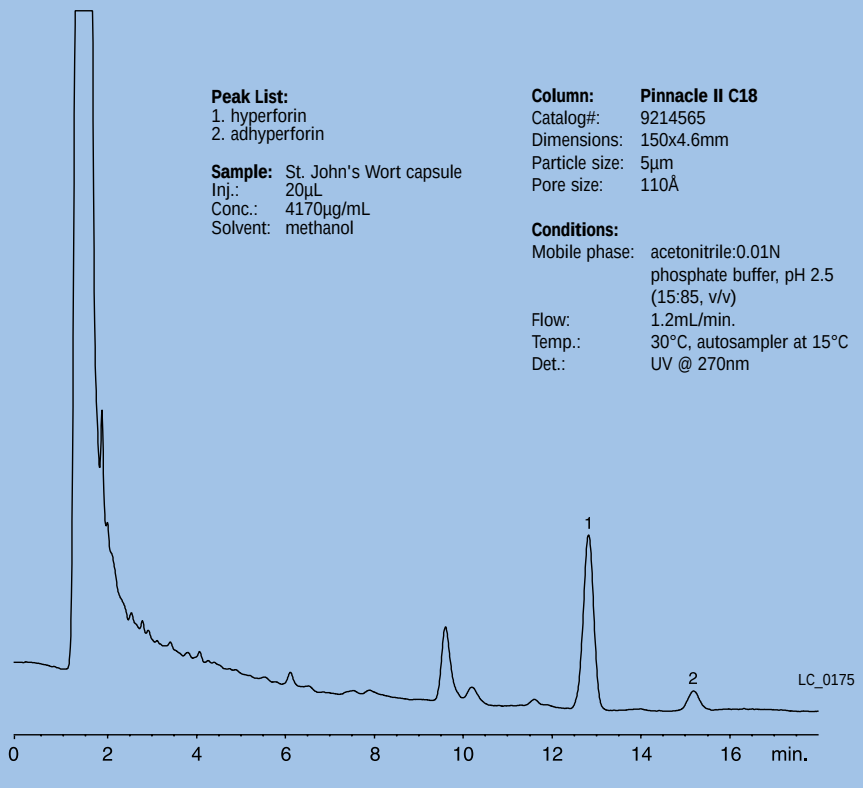
Additional stationary phases will be joining this family of products.

Base-Deactivated Columns Coming Soon!

A line of columns that offer the structural integrity of Type A silica, combined with an improved treatment for base deactivation. Excellent for analyzing basic compounds.

Figure 2

The Pinnacle II™ C18 column achieves excellent separation of hyperforin and adhyperforin when analyzing a St. John's Wort capsule.



Peak List:
1. hyperforin
2. adhyperforin

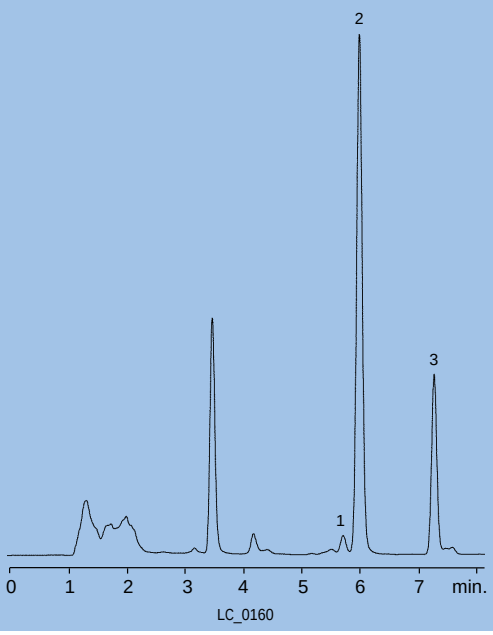
Sample: St. John's Wort capsule
Inj.: 20µL
Conc.: 4170µg/mL
Solvent: methanol

Column: Pinnacle II C18
Catalog#: 9214565
Dimensions: 150x4.6mm
Particle size: 5µm
Pore size: 110Å

Conditions:
Mobile phase: acetonitrile:0.01N phosphate buffer, pH 2.5 (15:85, v/v)
Flow: 1.2mL/min.
Temp.: 30°C, autosampler at 15°C
Det.: UV @ 270nm

Figure 3

The Pinnacle II™ C18 column achieves good resolution of three target capsaicinoids.



Peak List:
1. nordihydrocapsaicin
2. capsaicin
3. dihydrocapsaicin

Sample: habanero nugget extract
Inj.: 10µL
Conc.: 4000µg/mL
Solvent: ethanol

Column: Pinnacle II C18
Catalog#: 9214565
Dimensions: 150x4.6mm
Particle size: 5µm
Pore size: 110Å

Conditions:
Mobile phase: A: 1% acetic acid in water
B: acetonitrile

Time (min.):	% B
0	50
2	50
9	80
12	80
13	50

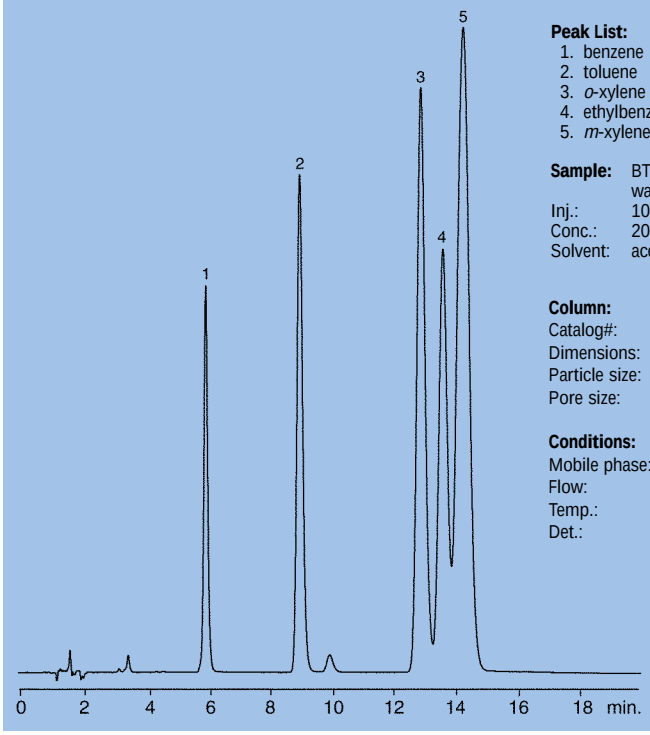
Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 280nm

Restek offers the **Trident Direct™ guard cartridge system**. All of our Pinnacle™ II phases are available in guard columns. The Trident Direct™ system features three levels of protection against many of the contaminated samples that analysts inject into a system.

Restek also offers a full line of HPLC accessories—everything from PEEK™ fittings to syringes.

Figure 4

A Pinnacle II™ C18 column resolves all 5 components of a BTEX standard in 15 minutes.



Peak List:
1. benzene
2. toluene
3. *o*-xylene
4. ethylbenzene
5. *m*-xylene & *p*-xylene

Sample: BTEX standard (cat.# 30213) was diluted 1:10
Inj.: 10µL
Conc.: 200µg/mL
Solvent: acetonitrile

Column: **Pinnacle II C18**
Catalog#: 9214565
Dimensions: 150x4.6mm
Particle size: 5µm
Pore size: 110Å

Conditions:
Mobile phase: water:acetonitrile (50:50, v/v)
Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 254nm

✔ Pinnacle II™ C8 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
50mm	9213551	9213552	9213553	9213555
100mm	9213511	9213512	9213513	9213515
150mm	9213561	9213562	9213563	9213565
250mm	9213571	9213572	9213573	9213575

✔ Pinnacle II™ C18 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
50mm	9214551	9214552	9214553	9214555
100mm	9214511	9214512	9214513	9214515
150mm	9214561	9214562	9214563	9214565
250mm	9214571	9214572	9214573	9214575

✔ Pinnacle II™ Phenyl 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
50mm	9215551	9215552	9215553	9215555
100mm	9215511	9215512	9215513	9215515
150mm	9215561	9215562	9215563	9215565
250mm	9215571	9215572	9215573	9215575

✔ Pinnacle II™ Cyano 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
50mm	9216551	9216552	9216553	9216555
100mm	9216511	9216512	9216513	9216515
150mm	9216561	9216562	9216563	9216565
250mm	9216571	9216572	9216573	9216575

Many New Phases Coming Soon!

Check our web site for additional stationary phases in this family of products.

Environmental Applications

Pinnacle™ II HPLC columns provide a cost-effective tool for many traditional methods used in the environmental industry, including US Environmental Protection Agency (EPA) Methods 8310 and 8330, as well as methods for analyzing BTEX (Figure 4) and carbamates.

Restek controls Pinnacle™ II raw material quality from the very beginning of the silica manufacturing process. Add our phase bonding and column packing experience to this high level of quality control, and you benefit from even better column-to-column and analysis-to-analysis reproducibility.

for more info

request the Pinnacle™ II product flyer (lit. cat.# #59281).

Hot Tech Tip

.High backpressure is one of the most common problems encountered when performing HPLC analysis. Normal column backpressure is observed after a new column has been installed and equilibrated with the mobile phase. Unfortunately, this pressure will often increase with use because of particles collecting on the column inlet frit.

These particles can be sample impurities, mobile phase contaminants, or materials from the injector or autosampler rotor seal. The presence of particles can result in increased backpressure, split peaks, tailing, and eventually over-pressure shut-down. In some circumstances, these problems can be corrected by back-flushing the column. However, in many cases the result is an unusable column.

To reduce backpressure problems, all samples and mobile phase solvents must be filtered before use, and rotor seals should be changed on a routine basis. Along with these preventative measures, it is advisable to use column prefilters such as the Trident™ Direct column protection system. Particles build up on the inexpensive, replaceable frit in the prefilter, instead of the permanent frit at the head of the column.

For more information on the Trident™ Direct guard column system and a complete product offering, request lit. cat.# 59314.

Pesticide Reference Materials

For Food Testing Now Available

by Christopher Cox, R&D Manager

new

✔ For laboratories performing food testing.
✔ Ensures accurate proficiency testing.

Laboratories testing food quality and safety are encouraged to routinely perform proficiency tests. Proficiency testing is an external check of quality. It provides an independent and unbiased assessment of the performance of all aspects of the laboratory, both human and hardware. Each participating laboratory is encouraged to use its normal analytical method, thereby simulating the testing of a routine laboratory sample as closely as possible. While the outcome of the analysis may be dependent upon the choice of method, it also could be affected by the performance of the laboratory equipment or the competence of the analyst. Using proficiency testing, those laboratories performing well can ensure high standards are maintained and those performing unsatisfactorily can implement corrective action rapidly. In an environment where analytical laboratories now compete intensively for work, proficiency testing provides the means by which external customers can compare competence in carrying out specific tests. Together with laboratory accreditation and the use of validated methods, proficiency tests are an important requirement of the EU Additional Measures Directive 93/99/EEC applying to laboratories entrusted with the official control of food.

The FAPAS® (Food Analysis Performance Assessment Scheme) program is run by the proficiency testing group of the Central Science Laboratory, an executive agency of the UK Department for Environment, Food and Rural Affairs (DEFRA). The FAPAS program was established in 1990 to improve the quality of analytical data on certain foodstuffs from UK analytical laboratories, and it was soon expanded worldwide. It is now the largest food chemistry proficiency testing scheme in the world, with the widest range of matrices and analytes. The program is designed to test real food samples, including canned meats, canned fish, soft drinks, fruit, vegetables, alcoholic drinks, and more.

Restek introduces calibration mixes that can be used with the FAPAS® Series 5 organochlorine pesticides analyses, and the FAPAS® Series 9 organophosphorus pesticides analyses. Use of Restek calibration mixtures by laboratories participating in the FAPAS® program is voluntary and no endorsement of any Restek product has been made by the Central Science Laboratory.

To obtain further information regarding the FAPAS® program or to participate, contact fapas@csl.gov.uk.

For technical information regarding the mixtures listed here, contact Restek Technical Service or your local Restek distributor.

✔ Series V Organochlorine Pesticide Mix #1—*Suitable for GC/MS Methods*

α-BHC	oxychlordane
β-BHC	α-endosulfan I
γ-BHC	β-endosulfan II
hexachlorobenzene	endosulfan sulfate
heptachlor	endrin
heptachlor epoxide (isomer B)	4,4'-DDE
aldrin	4,4'-DDD
dieldrin	2,4'-DDT
α-*cis*-chlordane	4,4'-DDT
γ-*trans*-chlordane	

100µg/mL each in acetone
1mL per ampule

Ea.	5-pk.	10-pk.
32412	32412-510	—
	with data pack	
32412-500	32412-520	32512

✔ Series V Organochlorine Pesticide Mix #2—*Suitable for GC/ECD Methods*

α-BHC	10 µg/mL
β-BHC	10
γ-BHC	10
hexachlorobenzene	10
heptachlor	10
heptachlor epoxide (isomer B)	10
aldrin	10
dieldrin	20
α-*cis*-chlordane	10
γ-*trans*-chlordane	10
oxychlordane	10
α-endosulfan I	10
β-endosulfan II	20
endosulfan sulfate	20
endrin	20
4,4'-DDE	20
4,4'-DDD	20
2,4'-DDT	20
4,4'-DDT	20

Each compound at concentration listed, in acetone
1mL per ampule

Ea.	5-pk.	10-pk.
32414	32414-510	—
	with data pack	
32414-500	32414-520	32514

✔ Series IX Organophosphorus Pesticide Standard

Suitable for GC/FPD, GC/NPD, and GC/MS Methods

chlorpyriphos	chlorpyriphos-methyl
diazinon	dichlorvos
etrimphos	fenitrothion
malathion	methacriphos
phosphamidon	pirimiphos-methyl

100µg/mL in acetone
1 mL per ampule

Ea.	5-pk.	10-pk.
32413	32413-510	—
	with data pack	
32413-500	32413-520	32513

Order a FREE Foods, Flavors, and Fragrances Catalog! This 52-page document includes important analysis tips, and chromatograms for the analysis of fats and oils, carbohydrates, vitamins, amino acids, organic acids, preservatives, flavors and fragrances, essential oils, and chiral separations. Retention time indices and complete product listings for all the relevant GC and HPLC products also are included (lit. cat.# 59260). Also, request the soon-to-be-released Applications Note detailing food packaging testing (lit. cat.# 59348).

Plus 1™ Restek's Customer Commitment

You will be seeing the Plus 1™ symbol throughout our catalog, on our packaging, and on our website. Plus 1™ Service means we will surpass your expectations every time you contact us! You'll get Plus 1™ service when you ask our experienced Technical Service Team to help solve a difficult analytical problem. Our helpful, efficient Customer Service Team provides Plus 1™ service even when you place a late-day order. Plus 1™ customer service is what has made Restek unique. If special attention was paid to your request or if our employees went out of their way for you, let us know. Contact us with your Restek success stories today!

plus 1™

Analytical Reference Materials & Columns

ISO/DIS 9377-4 Water Quality Testing for Total Petroleum Hydrocarbons (TPH)

by Christopher Cox, R&D Manager

new

✔ For GC analysis of TPHs in water.
✔ Environmentally safer method than those performed previously.

ISO/DIS 9377-4 describes a gas chromatography/flame ionization detection (GC/FID) method to analyze total petroleum hydrocarbons (TPHs) in drinking, surface, waste, and treated waste water. Previous methods used Freon® extraction, which was harmful to the environment. This new method uses less harmful solvents such as pentane, hexane, or cyclohexane for sample extraction.

Restek now offers mixtures for ISO/DIS 9377-4 analyses. Florisil® cleanup to remove polar compounds is accomplished using a 150-250µm (60/100 mesh) sample preparation column. The analytical column suggested is either an Rtx®-1 or an Rtx®-5 column with dimensions of 10-25m, 0.25-0.53mm ID, and 0.25-1.0µm film thickness.

✔ Standard Mixture Stock Solution

diesel #2 (additive free)
mineral oil (additive free [i.e. USP grade] bp 325–460 or C18–C32 retention time range)
5,000 µg/mL each in cyclohexane, 1mL per ampule (prepares 8mL of 1.25µg/µL calibration curve high point)
Total hydrocarbon concentration 10,000 µg/mL

Ea.	5-pk.	10-pk.
31630	31630-510	—
with data pack		
31630-500	31630-520	31730

✔ Quality Control Standard Mixture

diesel #2 (additive free)
mineral oil (additive free [i.e. USP grade] bp 325–460 or C18–C32 retention time range)
500µg/mL each in acetone, 1mL per ampule (enough to spike one quality control sample)
Total hydrocarbon concentration 1000µg/mL

Ea.	5-pk.	10-pk.
31631	31631-510	—
with data pack		
31631-500	31631-520	31731

✔ Florisil® Cartridge Quality Control Standard Mixture

Diesel #2 (additive free)
mineral oil (additive free [i.e. USP grade] bp 325–460 or C18–C32 retention time range)
1000µg/mL each in cyclohexane, 10mL per ampule (enough to check one Florisil® cartridge)
Total hydrocarbon concentration 2000µg/mL

Ea.	5-pk.	10-pk.
31632	31632-510	—
with data pack		
31632-500	31632-520	31732

✔ Standard Mixture of *n*-alkanes for System Performance Test

n-decane	*n*-hexacosane
n-dodecane	*n*-octacosane
n-tetradecane	*n*-triacontane
n-hexadecane	*n*-dotriacontane
n-octadecane	*n*-tetratriacontane
n-eicosane	*n*-hexatriacontane
n-docosane	*n*-octatriacontane
n-tetracosane	*n*-tetracontane

50µg/mL each in cyclohexane, 1mL per ampule

Ea.	5-pk.	10-pk.
31633	31633-510	—
with data pack		
31633-500	31633-520	31733

✔ Extraction Solvent Stock Solution #1

n-decane	20µL/L
n-tetracontane	20mg/L

in cyclohexane, 5mL per ampule (makes 50mL of extraction solvent, enough for 1 sample)

Ea.	5-pk.	10-pk.
31634	31634-510	—
with data pack		
31634-500	31634-520	31734

✔ Extraction Solvent Stock Solution #2

n-decane	20µL/L
n-tetracontane	20mg/L

in cyclohexane, 20mL per ampule (makes 200mL of extraction solvent, enough for 4 samples)

Ea.	5-pk.	10-pk.
31635	31635-510	—
with data pack		
31635-500	31635-520	31735

✔ Stearyl Stearate Test Solution

stearyl stearate
2,000 µg/mL in cyclohexane, 10mL per ampule, (enough to check one Florisil® cartridge)

Ea.	5-pk.	10-pk.
31636	31636-510	—
with data pack		
31636-500	31636-520	31736

Columns

✔ Rtx®-1 (fused silica)

Crossbond® 100% dimethyl polysiloxane

ID	df (µm)	temp. limits	15-Meter
0.25mm	0.25	-60 to 330/350°C	10120
0.25mm	0.50	-60 to 330/350°C	10135
0.32mm	0.25	-60 to 330/350°C	10121
0.32mm	0.50	-60 to 330/350°C	10136
0.53mm	0.25	-60 to 320/340°C	10122
0.53mm	0.50	-60 to 310/330°C	10137
0.53mm	1.00	-60 to 310/330°C	10152

✔ MXT®-1 (Silcosteel®)

Crossbond® 100% dimethyl polysiloxane

ID	df (µm)	temp. limits	15-Meter
0.25mm	0.25	-60 to 360°C	70120
0.25mm	0.50	-60 to 350°C	70135
0.28mm	0.25	-60 to 360°C	70121
0.28mm	0.50	-60 to 330°C	70136
0.53mm	0.25	-60 to 360°C	70122
0.53mm	0.50	-60 to 330°C	70137
0.53mm	1.00	-60 to 320°C	70152

✔ Rtx®-5 (fused silica)

Crossbond® 5% diphenyl/95% dimethyl polysiloxane

ID	df (µm)	temp. limits	15-Meter
0.25mm	0.25	-60 to 330/350°C	10220
0.25mm	0.50	-60 to 330/350°C	10235
0.32mm	0.25	-60 to 330/350°C	10221
0.32mm	0.50	-60 to 330/350°C	10236
0.53mm	0.25	-60 to 320/340°C	10222
0.53mm	0.50	-60 to 310/330°C	10237
0.53mm	1.00	-60 to 310/330°C	10252

✔ MXT®-5 (Silcosteel®)

Crossbond® 5% diphenyl/95% dimethyl polysiloxane

ID	df (µm)	temp. limits	15-Meter
0.25mm	0.25	-60 to 360°C	70220
0.25mm	0.50	-60 to 350°C	70235
0.28mm	0.25	-60 to 360°C	70221
0.28mm	0.50	-60 to 330°C	70236
0.53mm	0.25	-60 to 360°C	70222
0.53mm	0.50	-60 to 330°C	70237
0.53mm	1.00	-60 to 325°C	70252

Always in stock!

Restek has Rtx®-1, Rtx®-5, & Rtx®-Wax columns in stock for you! There's no waiting for the columns you need.

Super-Clean™ Gas Filters

Economical Gas Purification Made Easy**

by Gary Barone, GC Accessories Product Marketing Manager

✔ High-purity output (99.9999% purity).
✔ Features a "quick connect" for fast and simple cartridge changes.
✔ Full glass/metal design.

This line of Super-Clean™ gas filters is the latest in cartridge gas filtration. Cartridge systems make changing gas filters quick and easy. The system works using a baseplate that allows cartridges to be exchanged without introducing oxygen. The spring-loaded check valves seal when a filter is removed and open only when a new filter has been locked in place. There is no longer a need for loosening and tightening fittings every time a trap is changed.

The Triple Filter model is ideal for carrier gas purification. This trap contains the oxygen, moisture, and hydrocarbon scrubbers in one cartridge. The gas purity of your carrier gas through the Triple Filter is better than six-9s, ideal for mass spectrometry (MS) and electron capture detection (ECD), and for protecting your columns against damage.

The Fuel Gas Filter cartridge is perfect for purifying flame ionization detector (FID) fuel gases, and removing moisture and hydrocarbons. Using the Fuel Gas Filter for FID hydrogen and air will produce a stable baseline, improving overall reproducibility and sensitivity.

Also available are 2- and 3-position baseplates. By using the 2- and 3-position baseplates you can purify all GC gas streams at one physical location with multiple Super-Clean™ filter cartridges. Figure 1 shows some possible filter cartridge combinations using these baseplates. Any combination of filters is possible because all the Super-Clean™ filter cartridges can be used with any baseplate.

Special offer!

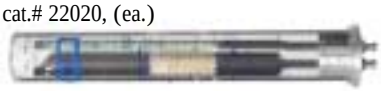
Carrier Gas Filter Kit: Buy one Triple Filter and receive one single-position baseplate free!* cat.# 22024, (kit)

buy this

get this too!

*Offer good until 9/31/01

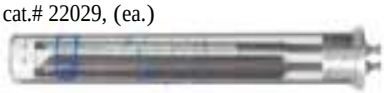
Replacement Triple Filter: removes oxygen/moisture and hydrocarbon impurities.
cat.# 22020, (ea.)



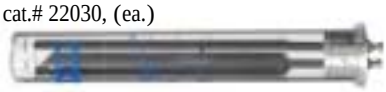
Replacement Fuel Gas Filter: removes moisture and hydrocarbon impurities
cat.# 22022, (ea.)

Ultra-High Capacity Moisture Filter
cat.# 22028, (ea.)

Ultra-High Capacity Oxygen Filter
cat.# 22029, (ea.)



Ultra-High Capacity Hydrocarbon Filter
cat.# 22030, (ea.)



Filter Bundle Kit: two Fuel Gas Filters for FID fuel gases and one Triple Filter for carrier gas.
cat.# 22031, (kit)

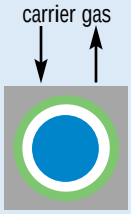
FID Fuel Gas Filter Kit: one Fuel Gas Filter and one single-position baseplate.
cat.# 22021. (kit)

Replacement O-Rings: for Super-Clean Filter cartridge baseplates. cat.# 22023, (2-pk.)

All Super-Clean™ filter cartridges feature **easy-to-read indicators.** The indicator code is shown on every trap so there is no confusion determining when a trap needs to be replaced.

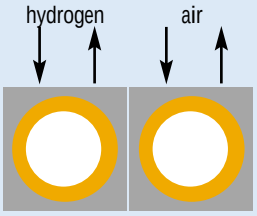
Figure 1
Configurations for different methods.

Triple Kit for MS, ECD, NPD



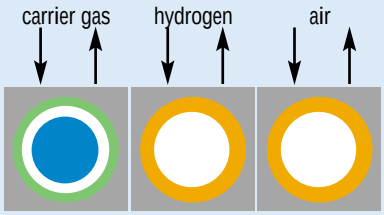
Triple Filter
hydrocarbons/
moisture/oxygen

Burner Gas Kit for FID



Fuel Gas Filter hydrocarbons/ moisture
Fuel Gas Filter hydrocarbons/ moisture

Complete Filter Bundle Kit for FID



Triple Filter hydrocarbons/ moisture/oxygen
Fuel Gas Filter hydrocarbons/ moisture
Fuel Gas Filter hydrocarbons/ moisture

Single-Position Baseplate
cat.# 22025, (ea.)

2-Position Baseplate
cat.# 22026, (ea.)

3-Position Baseplate
cat.# 22027, (ea.)

**These filters should last 10 months when used in a typical application.

New Reference Texts

For GC, GC/MS, HPLC, & More!

by Jack Crissman, Training & Education Manager

Atmospheric Chemistry and Physics
J. H. Seinfeld and S. N. Pandis, John Wiley, 1997, 1326pp., ISBN 0-471-17815-2
cat.# 20470

Chromatographic Analysis of Environmental and Food Toxicants
T. Shibamoto, Marcel Dekker, 1998, 331pp., ISBN 0-8247-0145-3
cat.# 21085

Chromatographic Analysis of Pharmaceuticals, 2nd Ed.
J. A. Adamovics, Marcel Dekker, 1997, 527pp., ISBN 0-8247-9776-0
cat.# 21089

GC/MS. A Practical User's Guide
M. McMaster and C. McMaster, John Wiley, 1998, 167pp., ISBN 0-471-24826-6
cat.# 20496

GC/MS Guide to Ignitable Liquids
R. Newman, M. Gilbert and K. Lothridge, CRC Press LLC, 1998, 750pp., ISBN 0-8493-3107-2
cat.# 20471

Handbook of HPLC
E. Katz, R. Eksteen, P. Schoenmakers, and N. Miller, Eds., Marcel Dekker, 1998, 989pp., ISBN 0-8247-9444-3
cat.# 21087

Hawley's Condensed Chemical Dictionary, 13th Ed.
R. J. Lewis, John Wiley, 1997, 1229pp., ISBN 0-471-29205-2
cat.# 21369

Introduction to Analytical Gas Chromatography, 2nd Ed.
R. P. W. Scott, Marcel Dekker, 1998, 397pp., ISBN 0-8247-0016-3
cat.# 21084

Liquid Chromatography/Mass Spectrometry, 2nd Ed.
W. M. A. Niessen, Marcel Dekker, 1999, 634pp., ISBN 0-8247-1936-0
cat.# 21086

On-Column Injection in Capillary Gas Chromatography, 2nd Ed.
K. Grob, Wiley-VCH, 1998, 591pp., ISBN 3-7785-2055-5
cat. # 20453

A Practical Guide to the Care, Maintenance, and Troubleshooting of Capillary Gas Chromatographic Systems, 3rd Ed.
D. Rood, Wiley-VCH, 1999, 323pp., ISBN 3-527-29750-2
cat.# 20450

Quantitative Chromatographic Analysis
T. E. Beesley, B. Buglio and R. P. W. Scott, Marcel Dekker, 2001, 378pp., ISBN 0-8247-0503-3
cat.# 21093

Solid Phase Extraction. Principles and Practice
E. M. Thurman and M. S. Mills, John Wiley, 1998, 344pp., ISBN 0-471-61422
ct.# 20494

Solid Phase Extraction. Principles, Techniques, and Applications
N. J. K. Simpson, Marcel Dekker, 2000, 514pp., ISBN 0-8247-0021
cat.# 21091

Split and Splitless Injection for Quantitative Gas Chromatography, 4th Ed.
K. Grob, John Wiley, 2001, 460pp., ISBN 3-527-29879-7
cat.# 20451

Static Headspace–Gas Chromatography, Theory and Practice
B. Kolb and L. S. Ettre, Wiley-VCH, 1998, 298pp., ISBN 0-471-19238-4
cat.# 20495

Visit our website at **www.restekcorp.com** for a complete list of reference texts and details on other educational materials.

Restek On-the-Road

GC, HPLC, & Environmental Training Coming to a Location Near You!

Restek "On-the-Road" presents a nationwide, three-month tour to spread chromatographic knowledge! We are offering three different courses this year: *Comprehensive GC*, *HPLC*, *and Environmental GC*. Each full-day course is presented in an engaging multimedia format. We teach key chromatographic concepts, tricks of the trade, and little known secrets that are of benefit to the novice and the seasoned veteran. We are chromatographers talking about chromatography, presenting the facts on how to help improve your chromatographic analyses. This is a great opportunity to learn tips for improving the efficiency and effectiveness of your laboratory.

These courses will help you:

- Improve chromatographic efficiencies.
- Identify and adjust variables to optimize your system.
- Increase sample throughput.
- Identify and troubleshoot problems with your analysis and instrument.

Courses start in early September and seats fill quickly, so register today!

only $199 per person

materials, refreshments, and lunch included.

For more information or to register, call 800-356-1688 or 814-353-1300, ext. 3, or go online at ***www.restekcorp.com***

SilcoCan® Canisters

Ideal for Low-Level (1ppb-20ppb) Reactive Sulfur Compounds

by Dave Shelow, Air Monitoring Innovations Chemist

✔ Stable, long-term storage of sulfur VOCs.
✔ More accurate sampling.
✔ Accessories available.

The analysis of low-level sulfur volatile organic compounds (VOCs) has become important because of odor complaints near manufacturing sites and refineries. Collection and measurement of these compounds in the atmosphere is very difficult because of their low concentration and high reactivity. These sulfur compounds not only can react with each other but also with the vessels in which they are collected. This results in low recoveries of compounds such as hydrogen sulfide (H_2S), methyl mercaptan (CH_3SH), ethyl mercaptan (C_2H_3SH), and dimethyl disulfide (CH_3SSCH_3).

Tedlar bags traditionally have been used for collecting sulfur VOCs; however, the stability of low-level (100ppbv) sulfur VOCs is poor within 24 hours of sampling.[1] Electropolished canisters (e.g., SUMMA® canisters) are excellent for storing VOCs in ambient air; however, the sulfur compounds react with the metal surface so these canisters are unsuitable for collecting and storing low-level sulfur VOCs.[2] SilcoCan™ air monitoring canisters, which feature a Silcosteel®-treated surface, increase the storage stability of low-level sulfur VOCs. We evaluated the stability of sulfur VOCs within SilcoCan™ canisters at very low levels (1–20ppbv) for six days. A comparison study of dry vs. humidified standards demonstrates the ability of SilcoCan™ canisters to store low-level sulfur VOCs in real-world conditions. The results showed excellent stability of each of the low-level sulfur VOCs in the dry and the humidified standards when using SilcoCan™ canisters. However, the electropolished canisters exhibit rapid degradation of hydrogen sulfide, methyl mercaptan, and ethyl mercaptan during a similar study (Figure 1).

When you need to perform sensitive air monitoring analyses, use a SilcoCan™ canister to collect and store your samples.

for more info

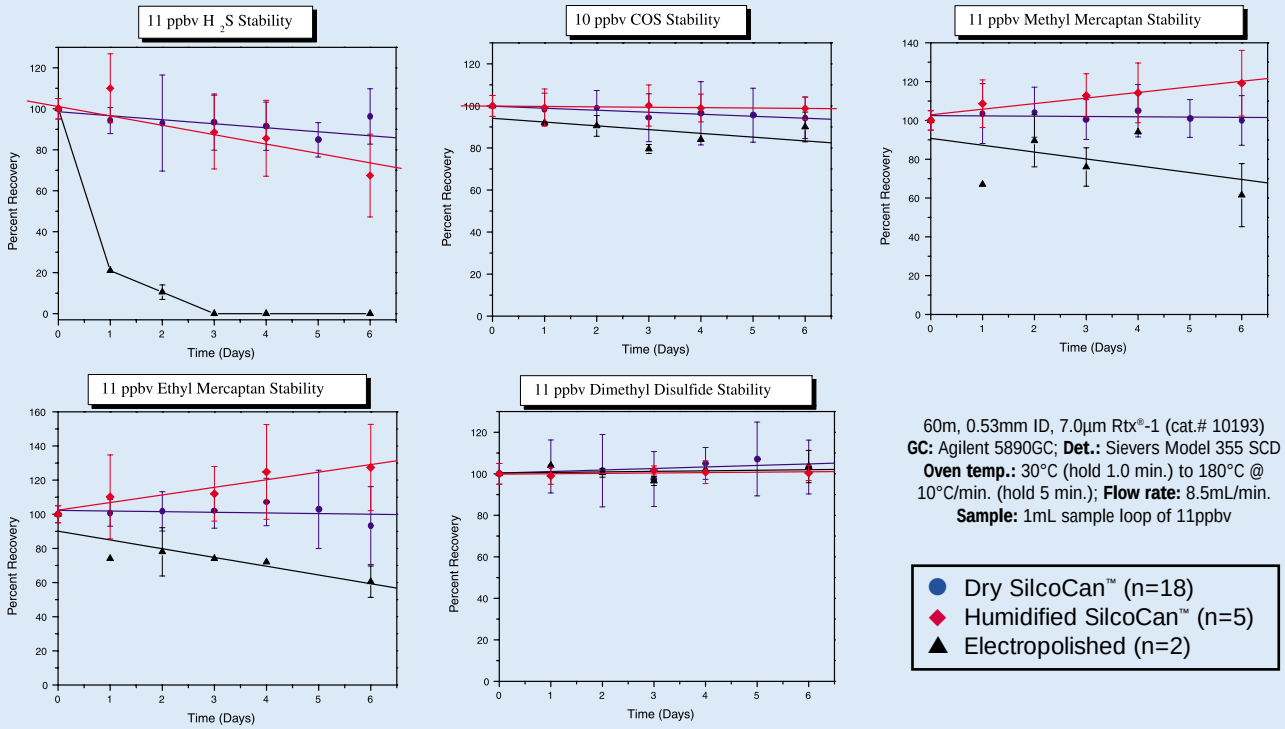
For complete details on the analytical system, request lit. cat.# 59347.

✔ SilcoCan™ Canisters

size (L)	cat.#
1	24112
3	24113
6	24114
15	24115

Figure 1

Stability data demonstrates the effectiveness of using SilcoCan™ canisters to store low-level organic sulfur compounds in real-world conditions.



Standards: Dry standards were made by taking 2mL of a 100ppm stock sulfur standard and adding it to each precleaned and evacuated canister, then pressurizing to 30psig with ultra-pure nitrogen. The resultant concentrations are listed in Applications Note #59347. Humidified standards were made by injecting the evacuated canisters with 100µL of deionized water prior to adding the 2mL aliquot of stock standard. This resulted in 50% RH.

References

1. Quang Tran, You-Zhi Tang; Stability of Reduced Sulfur Compounds in Whole Air Samplers, 1994 AWMA/EPA International Symposium of Measurement of Toxic and Related Air Pollutants.
2. Hoyt, Steven; Longacre, Vivian; and Stroupe, Michale; Measurement of Oxygenated Hydrocarbons and Reduced Sulfur Gases by Full Scan GC/MS: EPA TO-14; Sampling and Analysis of Airborne Pollutants, Eric Winegar, Lawrence Keith.

References not available from Restek.

Improved Passive Air Sampling Kits

Better Performance at a Better Value

by Dave Shelow, Air Monitoring Innovations Chemist

✔ New design eliminates leaks at the filter.
✔ Silcosteel®-treated components result in a very inert surface.
✔ Excellent for 8-, 12-, 24-, or 48-hour grab sampling.

Restek introduces a newly-designed passive air sampling kit that incorporates all of the hardware necessary to successfully collect air samples. These kits are excellent for 8-, 12-, 24-, and 48-hour or grab sampling, and are easy to assemble for field sampling.*

This most recent improvement incorporates the particulate filter inside the critical orifice fittings. The original design used an in-line filter before the critical orifice. By eliminating the in-line filter, the number of potential leak sites was greatly reduced. Also, reducing the amount of components in the kit has allowed us to reduce the price, while improving the quality and ease of use.

The new passive air sampling kit is available in a wide range of sampling flow ranges, and in stainless steel or Silcosteel® coating. The stainless steel passive air sampling kit is ideal to partner with the Restek TO-Can™ air sampling canister for TO-14A and TO-15 methods. The Silcosteel®-coated version should be used with the Restek SilcoCan® air sampling canister when collecting low-level volatile sulfur compounds (see previous page).

Canister Volume (L) 1	3	6	15	Flow	Orifice	Silcosteel® Complete Sampling Kits	Non-Silcosteel® Complete Sampling Kits
4 hour	12 hour	24 hour	60 hour	2-4	.0012	cat.# 24160	cat.# 24165
2 hour	6 hour	12 hour	30 hour	4-8	.0016	cat.# 24161	cat.# 24166
1 hour	4 hour	8 hour	20 hour	8-20	.0020	cat.# 24162	cat.# 24167
—	2 hour	3 hour	8 hour	20-40	.0030	cat.# 24163	cat.# 24168
—	—	1 hour	3 hour	40-80	.0060	cat.# 24164	cat.# 24169

*Air sampling canisters sold separately.

✔ SilcoCan™ Canisters

size (L)	cat.#
1	24112
3	24113
6	24114
15	24115

✔ TO-Can™ Canisters

size (L)	cat.#
1	24150
3	24152
6	24153
15	24154

✔ SilcoCan™ Canisters with Vacuum/Pressure Guage

size (L)	cat.#
1	24116
3	24117
6	24118
15	24119

✔ TO-Can™ Canisters with Vacuum/Pressure Guage

size (L)	cat.#
1	24155
3	24156
6	24157
15	24158

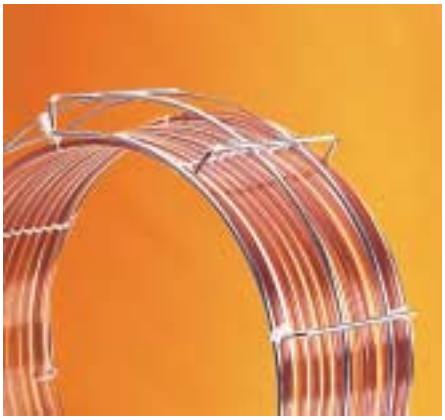
Questions?

Contact us at support@restekcorp.com. The industry's best technical service will be glad to help you with Plus 1™ service!

Rtx®-5 Amine GC Column

Ideal for Trace-Level Analyses of Basic Compounds

by Neil Mosesman, GC Columns Product Marketing Manager



- ✔ Deactivated tubing reduces sample adsorption.
- ✔ Eliminates costly derivatization procedures.
- ✔ Offers low bleed and excellent peak shape.

Analyzing ppm levels of basic compounds, such as amines, by gas chromatography (GC) often requires derivatization of the sample or column priming to improve peak shape and sensitivity. Restek's innovative Rtx®-5 Amine capillary column features a unique deactivation technology that reduces adsorption and peak tailing for a wide variety of amines and other basic compounds without derivatizing or priming, even at low ppm concentrations.

Many drug compounds are basic because they contain amine functional groups. The Rtx®-5 Amine column can analyze many of these drug compounds successfully without costly derivatization. The ion trap GC/MS analysis of underivatized sympathomimetic amines on the Rtx®-5 Amine column shows excellent peak shapes and low column bleed, making it ideal for trace-level analyses (Figure 1).

Ethyleneamines are used as chemical intermediates and solvents, and in the manufacture of chelating and emulsifying agents. Analyzing trace impurities of these basic compounds is difficult because most capillary columns will adsorb them. The Rtx®-5 Amine column is uniquely deactivated to be ideal for the analysis of ppm-level basic impurities in ethyleneamines and piperazines (Figure 2). Notice that tailing is minimal and exceptional response is achieved.

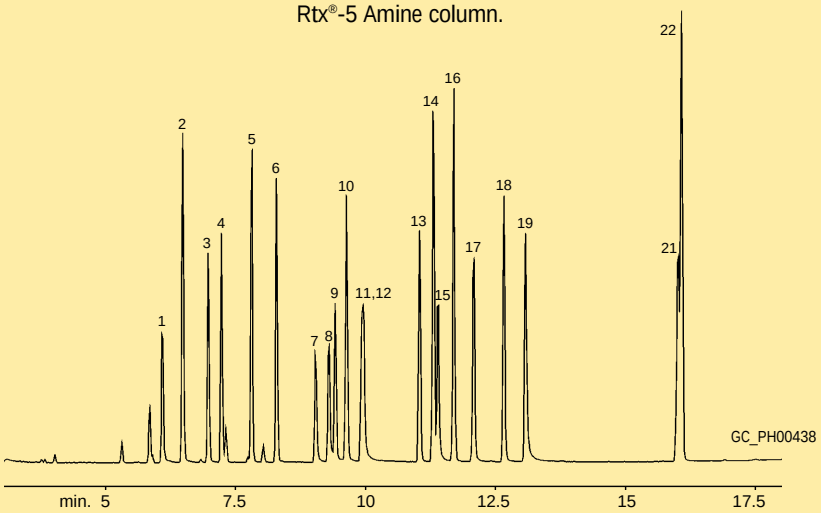
Because some adsorption of basic compounds can occur in the injection port, further improvements to the peak shape and response of amines can be obtained by using base-deactivated inlet liners. Base-deactivated inlet liners are available for most GC configurations. This ensures a completely inert sample pathway from injection port to detector. For consistently successful amine response, use this base-deactivated system and the Rtx®-5 Amine column.

Questions?

Contact us at
support@restekcorp.com.
The industry's best technical service will be glad to help you with Plus 1™ service!

Figure 1

Achieve good peak shape and response of sympathomimetic amines using an Rtx®-5 Amine column.

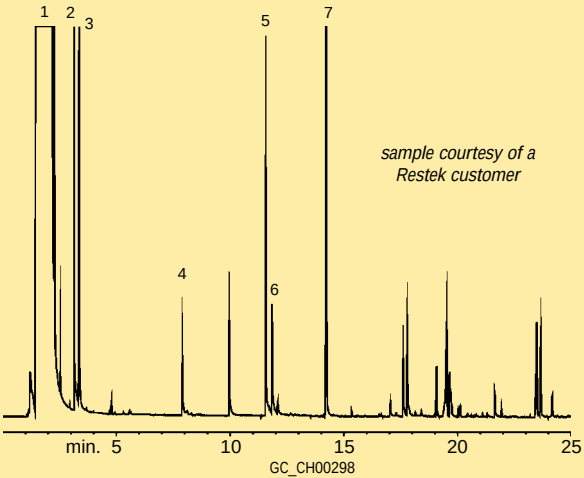


1. phenylethylamine
2. amphetamine
3. phentermine
4. methamphetamine
5. fenfluramine
6. mephentermine
7. cathinone
8. phenylpropanolamine
9. methcathinone
10. nicotine
11. ephedrine
12. pseudoephedrine
13. phenmetrazine
14. phendimetrazine
15. methylenedioxyamphetamine
16. diethylpropion
17. methylenedioxymethamphetamine
18. methylenedioxyethylamphetamine
19. 4-methyl-2,5-dimethoxyamphetamine
20. phenylephrine
21. caffeine
22. benzphetamine

30m, 0.25mm ID, 0.50µm Rtx-5 Amine (cat.# 12338); Injections performed in the split mode with a split vent flow rate of 45mL/min.; Analysis performed using a Varian 3400 GC coupled with a Varian Saturn 2000 MS detector; **Conc.:** 1mg/mL; **Oven temp.:** 100°C to 310°C @ 10°C/min.; **MS scan range:** 40-600 amu. Ionization was performed in the EI Auto mode.

Figure 2

The Rtx®-5 Amine column accurately analyzes trace impurities in ethyleneamines and piperazines.



1. isopropanol
2. monoethanolamine
3. ethylenediamine
4. piperazine
5. diethylenetriamine
6. aminoethylethanolamine
7. aminoethylpiperazine

remaining impurities consist of ethyleneamine and piperazine derivatives

30m, 0.25mm ID, 0.50µm Rtx-5 Amine (cat.# 12338); **Sample:** 3.0µL split injection of ethyleneamine industrial sample with on-column concentration ~5-80ng.; **Oven temp.:** 40°C (hold 4 min.) to 315°C @ 10°C/min. (hold 5 min.); **Inj. & det. temp.:** 315°C; **Carrier gas:** hydrogen; **Linear velocity:** 43cm/sec. set @ 40°C; **FID sensitivity:** 6.4 x 10^{-11} AFS; **Split ratio:** 20:1

✔ Rtx®-5 Amine (fused silica) Crossbond® 5% diphenyl/95% dimethylpolysiloxane; Stable to 340°C

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.50	-60 to 300/315°C	12335	12338
	1.00	-60 to 300/315°C	12350	12353
0.32mm	1.00	-60 to 300/315°C	12351	12354
	1.50	-60 to 290/305°C	12366	12369
0.53mm	1.00	-60 to 290/305°C	12352	12355
	3.00	-60 to 280/295°C	12382	12385

✔ Base-Deactivated Inlet Liners for amines and basic compounds

Restek stocks the most requested base-deactivated liners for immediate delivery. However, if you do not see the liner you need, orders can be placed by adding the appropriate suffix number: each (-210.1), 5-packs (-210.5), and 25-packs (-210.25). For base-deactivated liners packed with base-deactivated wool: each (-211.1), 5-packs (-211.5), and 25-packs (-211.25).

Base-Deactivated Inlet Liners for Agilent GCs			
	each	*5-pk.*	*25-pk.*
4mm Split Straight with Wool	20781-211.1	20782-211.5	20783-211.25
Cyclosplitter®	20706-210.1	20707-210.5	—
4mm Splitless Straight	20772-210.1	20773-210.5	—
2mm Gooseneck	20795-210.1	20796-210.5	20797-210.25
4mm Gooseneck	20798-210.1	20799-210.5	20800-210.25

Base-Deactivated Inlet Liners for Varian GCs		
	each	*5-pk.*
Splitter with Wool	20792-211.1, (ea.)	20793-211.5, (5-pk.)
Frit Splitter	20715-210.1, (ea.)	20716-210.5, (5-pk.)

Inlet Supplies Guide

We've updated and expanded the convenient, pocket-size guide. This highly-requested booklet details Restek's extensive selection of liners and other inlet supplies—organized by GC manufacturer. Request your free copy today! (lit. cat.# 59893A)

ChromGas Hydrogen & N_2 LC/MS Generators

The Best Value in Laboratory Gas Generators

by Gary Barone, GC Accessories Product Marketing Manager

✔ No high-pressure cylinders means improved lab safety.
✔ Ultra-high purity for better chromatography.

Restek has developed a marketing alliance with Parker Hannifin Corporation, Filtration and Separation Division—formerly Whatman—to offer gas generators world-wide. Parker Hannifin now provides the best value in laboratory gas generators.

The ChromGas fuel-grade hydrogen generator is a hazard-free alternative to high-pressure gas cylinders. The ChromGas generators use an exclusive solid polymer electrolyte to produce hydrogen on demand. Deionized water and an electrical supply is all that is needed to generate hydrogen for weeks of continuous operation.

With an output capacity up to 500cc/min., this generator can supply 99.9995% pure hydrogen to over ten gas chromatographs (GCs). Based on cylinder gas savings alone, a ChromGas hydrogen generator pays for itself in less than a year.

The Parker ChromGas hydrogen generators are certified for laboratory use by Canadian Standards Association (CSA), Underwriters Laboratories (UL), and International Electrotechnical Commission (IEC) 1010. A built-in sensing circuit shuts the generator down if a hydrogen leak is detected.

Nitrogen Generator and Air Compressor All in One—Ideal for LC/MS!

The Parker N2-15 generator incorporates an oilless air compressor and membrane nitrogen generator assembled as a package specifically to feed LC/MS systems. The N2-15 produces 15 liters-per-minute at 99% purity. The N2-15 generator is an ideal, safe, and less expensive long-term alternative than nitrogen cylinders or bulky dewars.

The N2-15 generator is a convenient, inexpensive solution to high-volume nitrogen generation. Previously a compressor and generator would have to be purchased separately. Often the end-users were responsible for installing and interfacing the compressor and generator. The cost of a reasonable system was well in excess of $20,000. Now the N2-15 generator solves the installation and set-up difficulties at a very attractive price.

✔ Hydrogen Generators

Model #	Capacity	cat.#
A9090	90 cc/min	22033
A9150	150 cc/min	22034
B9200	250 cc/min	22035
B9400	500 cc/min	22036

✔ Replacement deionizer bags and desiccant cartridge for Models A9090, A9150, B9200, and B9400

Deionizer bag: cat.# 21670, (ea.)
Desiccant cartridge: cat.# 21671, (ea.)

✔ Model N2-15 generator

cat.# 22037, (ea.)

Trace Explosives Analysis

Using an Rtx®-1 GC Column

by Gordon McMillen and Ann Irwin, Forensic Science Northern Ireland**

Background

For more than 30 years the forensic science laboratory in Northern Ireland has undertaken explosives analysis to cope with the continual use of explosives by terrorists. In many terrorist cases explosives and firearms are used together, so the laboratory developed methods to collect and examine both types of evidence. The trace residue from the discharge of firearms (cartridge discharge residue [CDR]) comprises two components: the primer residue (e.g., inorganic metallic particles) and organic residue (e.g., nitroglycerine from the propellant).

Collection procedure

Two types of laboratory swabbing kits[1] are manufactured for collecting CDR and explosive residue: one for suspects and one for scene locations. They each contain a ball of acrilan fiber moistened with isopropanol and heat-sealed in foil envelopes. Gloves and a disposable boiler suit also are included to prevent cross-contamination from the analyst during the collection process. After use, each swab is placed in a custom-made plastic swab holder resembling a miniature syringe body, which is then capped at both ends. Items in the laboratory, such as clothing, are sampled with balls of acrilan fiber or, more likely, vacuum sampled using a 25mm diameter deldrin filter holder (Gelman cat.# 1109) and a 0.5µm pore size Fluoropore® membrane filter (Millipore cat.# FHLP02500).

Extraction procedure

The swabs and filter samples can be examined for explosives only, or for particulate CDR residue and explosives. An extract is produced for explosives analysis by centrifuging the samples twice, once after the addition of an internal standard (1,3-dinitrobenzene) and once after the addition of an aliquot of diethyl ether. The final 1mL extract is collected in a 1.5mL gas chromatography (GC) autosampler vial.

The inorganic extract for particulate CDR examination is produced by ultrasonicating the swabs or filter residue in petroleum ether 140-165 for 30 minutes. The suspension then is decanted through a 25µm wire mesh filter in a Swinnex® holder (Millipore cat.# SX 0001300) to remove heavy debris, then through a 12.5mm diameter Swinnex® holder containing a 1µm pore size Fluoropore® membrane filter (Millipore cat.# FALP01300). After filtration, the 1µm filter is removed and placed on a 12.5mm diameter aluminium stub, carbon-coated and examined by automated scanning electron microscopy/energy dispersive x-ray (SEM/EDX) analysis.

GC analysis for organic explosives

The ether extracts are analyzed by GC fitted with a thermal energy analyzer (TEA®, Thermo Orion Model 543). An Agilent 5890 Series II GC, which has the pyrolyzer for the TEA® fitted through a hole cut in the left side of the oven, was used. The pyrolyzer and TEA® have been modified following some of the suggestions by Douse.[1] A Restek Rtx®-1 capillary column (cat.# 10120, 15m 0.25mm ID 0.25µm df) is butt-connected to a length of deactivated, 0.25mm ID fused silica tubing (Restek cat.# 10012) using a universal Press-Tight® connector (Restek cat.# 20400). This passes through the pyrolyzer directly into the reaction chamber of the TEA® detector, alongside a similar length of uncoated fused silica tubing originating directly from the ozone outlet. The second inlet on the reaction chamber is sealed with a blanking plug.

Five microliters of sample and standard are injected from the autosampler into an injector port fitted with a cup-split liner containing a small amount of fused silica wool (Restek cat.# 20790). The system is calibrated to assess performance and response with a combined standard solution (Figure 1). Casework samples are analysed without further cleanup and those samples with peaks lying within a specified retention time window are subject to confirmation.

HPLC Confirmation analysis

Positive results indicated on the GC/TEA® system must be confirmed by a second analytical system before the results can be reported in a statement to the police. The method of choice is high performance liquid chromatography (HPLC) with electrochemical detection (ECD) at a pendant mercury drop electrode using an autosampler.[2] Prior to HPLC analysis, the samples are cleaned using SPE extraction[3] and a column containing 40mg of a mixture of Chromosorb® 104 and Amberlite® XAD®-4 in the ratio 3:1.

Conclusion

The GC/TEA® system has been the Forensic Science Northern Ireland "workhorse" for many years, processing many thousands of samples a year during our busiest periods. We have been using Restek columns and accessories for a number of years and have found the products to be very reliable, producing a consistent analysis. There also is a first-rate customer backup service, provided by Restek Ireland.

References

1. *Improved method for the trace analysis of explosives by silica capillary column gas chromatography with thermal energy analysis detection.* J.M.F. Douse, Journal of Chromatography, 410 (1987) 181-189.
2. *Automated method for the analysis of organic explosive residues by HPLC with a pendant mercury drop electrode detector.* WJ McKeown and SJ Speers, Science & Justice 1996: 36: 15-20.
3. *Evaluation of improved methods for the recovery and detection of organic and inorganic cartridge redsidues.* Speers SJ, Doolan K, McQuillan J and Wallace JS, Journal of Chromatography 1994; A, 674: 319-327.

References not available from Restek.

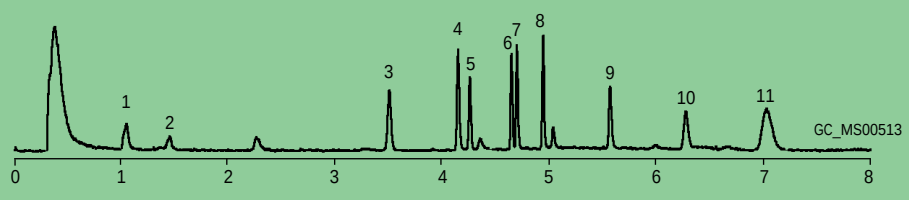
✔ **Rtx®-1** (fused silica)
Crossbond® 100% dimethyl polysiloxane

ID	df (µm)	temp. limits	15-Meter
0.25mm	0.25	-60 to 330/350°C	10120

For a complete Restek GC column offering, refer to the annual chromatography products guide, lit. cat.# 59960.

Figure 1

An Rtx-1® column is ideal for assessing performance and response for explosives standards.



Oven temp: 100°C (hold 3 min.) to 165°C (hold 3 min.) at 35°C/min. to 195°C (hold 3 min.) at 30°C/min.; **Carrier gas:** helium, 2.5mL/min.; **Split ratio:** 5.6:1; **Inj.:** 198°C; **TEA® conditions:** *Interface temp.:* 250°C; *Pyrolyzer temp.:* 800°C; The signal output of the TEA® detector is fed to a chromatography data system, which comprises a 4-channel A/D converter and Atlas software from Thermo Labsystems Ltd., a Thermo Electron Corporation Company

1. nitrobenzene (NB)
2. orthonitrotuluene (ONT)
3. nitroglycerine (NG)
4. 1,3-dinitrobenzene (1,3-DNB) (internal standard)*
5. 2,6-dinitrotoluene (2,6-DNT)
6. 2,3-dinitrotoluene (2,3-DNT)
7. 2,4-dinitrotoluene (2,4-DNT)
8. 3,4-dinitrotoluene (3,4-DNT)
9. trinitrotoluene (TNT)*
10. penta erythritol tetranitrate (PETN)*
11. cyclo trimethylene trinitramine (RDX)*

Standards are injected at a concentration of 0.1ng/µL, except for those marked *, which are 0.2ng/µL.

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Rt-QPLOT™ GC Column

New Deactivation Improves Response

by Neil Mosesman, GC Columns Product Marketing Manager

✔ Improved peak shape of polar solvents.
✔ Not sensitive to moisture—can be used for direct aqueous injections.
✔ Unique particle immobilization process eliminates detector spikes.

Porous layer open tubular (PLOT) columns commonly are used for the analysis of gases, low molecular weight compounds, and volatile solvents. In specific, PLOT columns coated with divinylbenzene (e.g., Porapak® Q or HayeSep® Q polymers) often are used for analyzing permanent gases such as air, CO, CO_2, and CH_4; C1 to C5 hydrocarbons; polar solvents; and C1 to C6 free fatty acids. While most compounds exhibited symmetrical peak shapes on the original Rt-QPLOT™ column, polar compounds such as alcohols often resulted in tailing peaks. Additionally, active compounds such as free fatty acids could display poor response on Rt-QPLOT™ columns that were not properly deactivated.

New deactivation techniques were developed recently to improve the performance of Restek's Rt-QPLOT™ columns. As a result, peak tailing of polar solvents has been reduced significantly. The analysis of 11 alcohols on an Rt-QPLOT™ column proves that the enhanced inertness results in more symmetrical peak shapes of alcohols and other polar solvents (Figure 1).

Peak tailing and poor response often occur when analyzing low molecular weight free fatty acids from C1 to C5 on a divinylbenzene PLOT column. Therefore, a highly inert PLOT column is required to obtain good peak shape and response. The improved deactivation on the Rt-QPLOT™ column results in excellent peak shape and response for low molecular weight free fatty acids (Figure 2).

The Rt-QPLOT™ column is not sensitive to moisture and, therefore, can be used for direct aqueous injections. To eliminate particle generation, which can cause detector spiking, a special bonding process immobilizes the stationary phase to produce strong, uniform particle adherence to the inside of the capillary tube. The Rt-QPLOT™ columns are available in 0.32 and 0.53mm internal diameters and in 15- and 30-meter lengths. The 0.32mm ID column has a 10µm particle coating, and the 0.53mm ID column has a 20µm particle coating. The maximum operating temperature for the Rt-QPLOT™ column is 250°C.

PLOT columns are useful for the analysis of a wide range of volatile compounds. Because tailing and poor response of active compounds can occur on improperly deactivated PLOT columns, it is critical to select a column with adequate inertness. The new and improved Rt-QPLOT™ column is specially deactivated to produce excellent peak shape and response for active polar compounds such as alcohols and free fatty acids. Also, the unique particle immobilization process eliminates detector spikes. Call the Restek Technical Service Team at ext. 4 with questions.

✔ **Rt-QPLOT™** (fused silica PLOT)
divinylbenzene Stable to 250°C

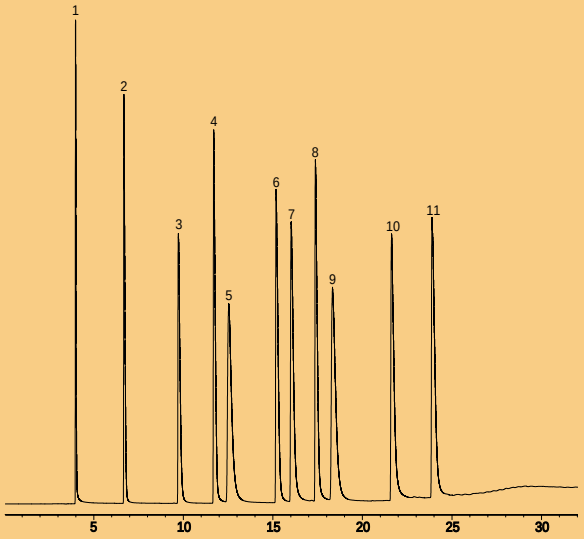
ID	df (µm)	15-Meter	30-Meter
0.32mm	10	19717	19718
0.53mm	20	19715	19716

✔ **MXT®-QPLOT** (metal Silcosteel®-coated PLOT)
divinylbenzene Stable to 250°C

ID	df (µm)	15-Meter	30-Meter
0.53mm	20	79715	79716

Figure 1

Obtain improved peak shapes for alcohols and other polar solvents on the new Rt-QPLOT™ column.



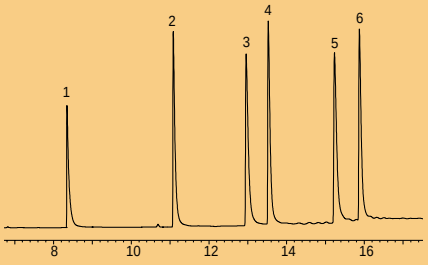
GC_CH00495

1. methanol
2. ethanol
3. 2-propanol
4. 1-propanol
5. tert-butanol
6. 2-butanol
7. isobutyl alcohol
8. 1-butanol
9. 2-methyl-2-butanol
10. 3-methyl-1-butanol
11. 4-methyl-2-pentanol

30m, 0.32mm ID, 10µm Rt-QPLOT™ (cat.# 19718); **Sample:** alcohol mix; **Conc.:** 1% of each compound in water; **Sample size:** 1.0µL; **GC:** Agilent 6890; **Oven program:** 100°C to 240°C @ 5°C/min. (hold 10min.); **Injector:** split @ 250°C; **Carrier gas:** helium (constant pressure mode); **Head pressure:** 18.0psi; **Column flow rate:** 1.1cc/min. @ 100°C; **Linear velocity:** 31cm/sec. @ 100C; **Split ratio:** 70:1; **Detector:** FID @ 270°C; **Make-up gas flow:** 45cc/min.; **Inlet liner:** 4mm single gooseneck (cat.# 20798)

Figure 2

Excellent peak shapes and response for free fatty acids are achieved with the improved Rt-QPLOT™ column.



GC_FF00496

1. acetic acid
2. propionic
3. isobutyric acid
4. butyric acid
5. isovaleric acid
6. valeric acid

30m, 0.32mm ID, 10µm Rt-QPLOT™ (cat.# 19718); **Sample:** atty acids mix; **Conc.:** 5% of each compound in water; **Sample size:** 0.5µL; **GC:** TRACE 2000; **Oven program:** 75°C to 240°C @ 10°C/min. (hold 15min.); **Injector:** split @ 240°C; **Carrier gas:** hydrogen (constant pressure mode); **Head pressure:** 12.0psi; **Column flow rate:** 1.1cc/min. @ 100°C; **Split ratio:** 100:1; **Detector:** FID @ 240°C; **Make-up gas flow:** 40cc/min.; **Inlet liner:** 3mm split w/glass wool (cat.# 20936-202.1)

Peak Performers

New GC Accessories to Make Your Analyses Easier and More Reproducible

by Gary Barone, GC Accessories Product Marketing Manager

Replacement Inlet Seals for Agilent 5890/6890 and 6850 GCs

- ✔ Special grade of stainless steel that is softer and compresses more easily, ensuring a completely leak-free seal.
- ✔ Increases column lifetime because oxygen cannot permeate into the carrier gas.
- ✔ Reduced noise benefits high-sensitivity detectors (e.g. ECDs, MSDs).
- ✔ Silcosteel®-treated seal offers the inertness of glass.

(0.8mm ID stainless steel inlet seal is equivalent to Agilent part #18740-20880.)
(0.8mm ID gold-plated inlet seal is equivalent to Agilent part #18740-20885.)

Single-Column Installation, Opening Size 0.8mm ID		0.25/0.32mm ID Dual-Column Installation, Opening Size 1.2mm ID		0.53mm ID Dual-Column Installation, Opening Size 1/16-Inch	
2-pk.	*10-pk.*	*2-pk.*	*10-pk.*	*2-pk.*	*10-pk.*
Stainless Steel Inlet Seal					
21315	21316	20390	20391	20392	20393
Gold-Plated Inlet Seal					
21317	21318	21305	21306	—	—
Silcosteel® Inlet Seal					
21319	21320	21307	21308	—	—

Cross Disk for Agilent 5890/6890 GCs

(Similar to Agilent part #5182-9652.)

0.8mm ID Cross Disk Inlet Seal for Agilent GCs	2-pk	10-pk.
Gold-Plated	20477	20476
Silcosteel®-Treated	20475	20474
1.2mm ID Cross Disk Inlet Seal for Agilent GCs	**2-pk.**	**10-pk.**
Gold-Plated	21009	21010
Silcosteel®-Treated	21011	21012

Thermolite® Septa

- ✔ Premium, low-bleed septa
- ✔ Lowest bleed on FIDs, ECDs, & MSDs*
- ✔ Usable to 340°C inlet temperatures
- ✔ Excellent puncturability
- ✔ Preconditioned and ready to use
- ✔ Do not adhere to hot metal surfaces

Septum Diameter	25-pk.	50-pk.	100-pk.
5mm (3/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9mm	20354	20358	20362
9.5mm (3/8")	20359	20360	20361
10mm	20378	20379	20380
11mm (7/16")	20363	20364	20365
11.5mm	22385	22386	22387
12.5mm (1/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374

Restek Leak Detective

The most-powerful, lowest-cost thermal conductivity leak detector available!

- ✔ Compact, lightweight, hand-held design.
- ✔ Contamination-free leak detection.
- ✔ Detects helium or hydrogen trace leaks at ≥3 x 10-4cc/sec. or ≥200ppm.
- ✔ Audible alarm and LED readout.
- ✔ Responds in less than 2 seconds to trace leaks of gases.**
- ✔ Two 9-volt batteries provide 10-12 hours of continuous operation.
- ✔ Unit can also be used with an AC adaptor (both included).

(110 VAC): cat.# 21607, (ea.)
(220 VAC): cat.# 21609, (ea.)
(220 VAC with UK plug): cat.# 21382, (ea.)

"Y" Vu-Union® Connecotrs

So unique, they're patented!
(patent #5,487,569)

- ✔ Combines the benefits of Press-Tight® connectors and metal unions.
- ✔ Leak-free connections.
- ✔ Will not unexpectedly disconnect.
- ✔ Glass window allows visual confirmation of seal.
- ✔ Usable at temperatures up to 400°C.

"Y" Vu-Union connector: cat.# 20432, (ea.)
Replacement "Y" inserts: cat.# 20433, (ea.)
cat.# 20434, (3-pk.)

*Refer to *A Guide to Minimizing Septa Problems* (lit. cat.# 59886).
**Not designed for use in explosive atmospheres.

New Thermal Gas Purifiers

- ✔ Remove oxygen, water, carbon monoxide, carbon dioxide and hydrocarbons.
- ✔ Purity in ppb levels.
- ✔ Mass Spec. purity carrier gas produced.
- ✔ Dual-tube model purifiers double capacity at less than double the price.
- ✔ Welded end-fittings on getter tubes eliminate leaks.
- ✔ Packed with reactor-grade, pure getter material for maximum efficiency and no contamination.

Introducing Restek's line of re-engineered thermal gas purifiers. This line of purifiers works by producing a chemical reaction between impurities in the carrier gas stream and the getter material. Because the reaction is non-reversible, there is no possibility of contaminants breaking through the thermal gas purifier.

Gas purification is very economical when using a thermal gas purifier. After initial installation cost, getter tubes only require changing once every year; heavy use and very impure feed gas may require more frequent getter tube replacement.

Restek Single-Tube Thermal Gas Purifier, 110 Volt:
⅛" Fittings: cat.# 21496, (ea.)
¼" Fittings: cat.# 21497, (ea.)

Restek Dual-Tube Thermal Gas Purifier, 110 Volt:
⅛" Fittings: cat.# 21498, (ea.)
¼" Fittings: cat.# 21499, (ea.)

Replacement Straight Getter Tubes:
⅛" Fittings: cat.# 21661, (ea.)
¼" Fittings: cat.# 21660, (ea.)

New Inlet Liners

For TRACE GCs
- ✔ Fully deactivated.
- ✔ Designed to meet original equipment specifications.

1mm ID: cat.# 21114, (ea.) cat.# 21115, (5-pk.)
2mm ID: cat.# 21116, (ea.) cat.# 21117, (5-pk.)

For Agilent GCs
- ✔ Designed for use with EPC-equipped Agilent 6890 GCs.
- ✔ Siltek™ deactivated.
- ✔ Low internal dead volume.

Each: cat.# 21390-214.1
5-pack: cat.# 21391-214.5

HPLC Piston Seal Insertion Tool

- ✔ Simplify your pump maintenance.
- ✔ One end removes old piston seal, and the other easily and securely installs new seal.

cat.# 21356, (ea.)

Connect Fused Silica Capillary Columns with New MXT® Unions

- ✔ Low-dead-volume, leak-free connection.
- ✔ Reusable.
- ✔ Silcosteel® treatment ensures maximum inertness.
- ✔ Ideal for connecting guard columns and transfer lines.
- ✔ Usable to oven temperatures of 350°C.
- ✔ Available in union and "Y" configurations.

Previously only metal tubing could benefit from an easy-to-use MXT® connector. Now the MXT® connector can be used with fused silica capillary columns because of the Valcon polyimide 1/32" one-piece fused silica adaptor. This unique graphite-reinforced composite allows capillary columns to slide and be locked into place simply by loosening and tightening the MXT® union 1/32" fitting.

MXT®-Union Connector Kits
Each kit contains the MXT® union, 2- 1/32" nuts and 2 one-piece fused silica adaptors
For 0.53mm columns: cat.# 21384, (kit)
For 0.32mm columns: cat.# 21385, (kit)
For 0.25mm columns: cat.# 21386, (kit)

MXT® "Y"-Union Connector Kits
Each kit contains the MXT® union, 3- 1/32" nuts and 3 one-piece fused silica adaptors
For 0.53mm columns: cat.# 21387, (kit)
For 0.32mm columns: cat.# 21388, (kit)
For 0.25mm columns: cat.# 21389, (kit)

Replacement One-Piece Fused Silica Adaptors for Fused Silica Capillary Columns
For 0.25mm columns: cat.# 20137, (5-pk.)
For 0.32mm columns: cat.# 20140, (5-pk)
For 0.53mm columns: cat.# 20141, (5-pk)
Replacement 1/32" nuts: cat.# 20389, (5-pk)

RESTEK
Behind the Scenes

Contact the representative nearest you for personal, Plus 1™ service!

Restek's field representatives are trained to assist customers in choosing the right chromatography supplies, optimizing method and instrument performance, and troubleshooting lab problems. As part of our Plus 1™ service commitment, we strive to surpass your expectations every time you contact us. Come to a Restek seminar (see pg. 7) to meet your rep, or contact the one nearest you from the map below.

Lonestar Instruments welcomes Pat to their team. Pat, Steve, and Anne are your contacts for the Gulf Coast region. Contact them for Plus 1™ service today!

New Sales Manager!

Restek's own Christine Vargo was promoted to Sales Manager early in 2001. Christine began at Restek in 1988 as the first Applications Chemist. She then became involved in technical marketing, moving into Field Support Chemist and Sales Representative positions for the New Jersey area. Most recently, Christine handled Corporate Accounts Management and Marketing of the GC Column product line. She brings hands-on laboratory experience, extensive Restek product knowledge, and an intense customer commitment to her new role.

New Literature

Application Notes

Determination of Oxygenates in Gasoline Using an Rtx®-VGC Column (59345)
Analyzing the Heat Levels of Peppers and Hot Sauces Using an Ultra C18 HPLC Column (59199)
Analyzing Glucosinolates Using HPLC (59335)
Fast Analysis of Dioxin and Related Compounds Using an Rtx®-5MS Column (59343)
Analysis of Phenylpropanolamine in Cold Medicine (59339)
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Rtx®-5 Amine GC Columns (59330)

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Guide to Preparing and Analyzing Semivolatile Organic Compounds (59411)

Fast Facts: At-a-Glance Product Info

ASTM D6042-69 Plastics in Packaging (59279)
Palladium Diffusion Hydrogen Generators (59331)
SGT Super-Clean™ Filters (59344)

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RESTEK

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the RESTEK Advantage

Innovators of High Resolution Chromatography Products

Low-Bleed, High-Temperature Column for ASTM Method D-6352

MXT®-1HT Sim Dist GC Column

by Neil Mosesman, GC Columns Product Marketing Manager, and Dinesh V. Patwardhan, Ph.D., Senior Research Chemist

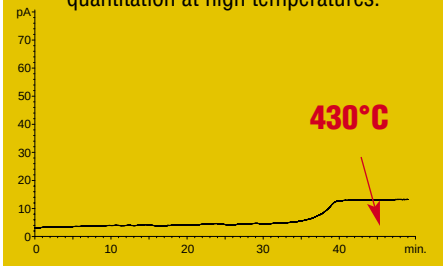
new

- ✔ Durable metal tubing and high-temperature polymer allows analysis of hydrocarbons up to C110.
- ✔ Low bleed at high temperatures help achieve accurate quantitation.
- ✔ Meets all criteria of ASTM Method D-6352.

The American Society for Testing and Materials (ASTM) is an organization that publishes consensus standards for materials, products, and services. Because ASTM methods are developed by consensus from the laboratories following the method, they are recognized worldwide. ASTM Method D-6352 is a gas chromatography (GC) method developed for the determination of petroleum distillates with a boiling point range of 174°C to 700°C. Often referred to as high-temperature simulated distillation or Sim Dist, this method requires a capillary column capable of withstanding high GC oven temperatures, up to 430°C. This presents many challenges for analysts because most capillary

Figure 1

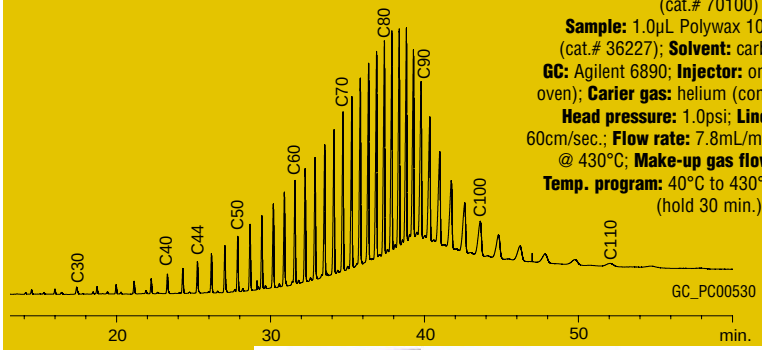
The MXT®-1HT Sim Dist column exhibits low bleed, which gives accurate quantitation at high temperatures.



5m, 0.53mm ID, 0.10µm MXT®-1HT (cat.# 70100)
GC: Agilent 6890; **Injector:** on-column (track oven);
Carier gas: helium (constant pressure);
Head pressure: 1.0psi; **Linear velocity:** 60cm/sec.;
Flow rate: 7.8mL/min.; **Detector:** FID @ 430°C;
Make-up gas flow: 40cc/min.; **Temp. program:** 40°C to 430°C @ 10°C/min. (hold 30 min.)

Figure 2

The MXT®-1HT Sim Dist column allows analysis of hydrocarbons up to C110 in less than one hour.



5m, 0.53mm ID, 0.10µm MXT®-1HT Sim Dist (cat.# 70100)
Sample: 1.0µL Polywax 1000 standard (cat.# 36227); **Solvent:** carbon disulfide;
GC: Agilent 6890; **Injector:** on-column (track oven); **Carier gas:** helium (constant pressure);
Head pressure: 1.0psi; **Linear velocity:** 60cm/sec.; **Flow rate:** 7.8mL/min.; **Detector:** FID @ 430°C; **Make-up gas flow:** 40cc/min.;
Temp. program: 40°C to 430°C @ 10°C/min. (hold 30 min.)

in this issue

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800-356-1688 ★ 814-353-1300
www.restekcorp.com

Fall 2001

columns are manufactured using polyimide-coated fused silica tubing. At temperatures above 380°C, even the best polyimide coating becomes brittle, which leads to very short column lifetimes. In addition, the methyl silicone stationary phase recommended in the method also must survive these high temperatures.

Extensive research by scientists at Restek has led to a major improvement in columns for high-temperature simulated distillation. By combining a new proprietary polymer synthesis technology, Siltek™ deactivation chemistry, and rugged Silcosteel® tubing, we developed a capillary column that meets all the criteria of ASTM Method D-6352. Because the MXT®-1HT Sim Dist column is coated with a 100% dimethyl polysiloxane polymer, it will give the correct retention time/boiling point curve. The MXT®-1HT Sim Dist column exhibits low bleed at 430°C and excellent peak shape due to the unique polymer synthesis and Siltek™ deactivation (Figure 1).

The rugged Silcosteel® tubing will hold up indefinitely to temperatures in excess of 430°C, so column lifetime is not limited by the tubing. To demonstrate the utility of this innovative product, we analyzed Polywax® 1000 using the MXT®-1HT Sim Dist column (Figure 2). Notice the excellent peak shape of hydrocarbons up to C110.

To maintain the low bleed and high performance of the MXT®-1HT Sim Dist column, it is critical to prevent oxygen from entering the column. This can be achieved by electronically leak checking your entire system. We also recommend the use of graphite ferrules; Vespel® or Vespel®/graphite ferrules are more likely to loosen over time.

The MXT®-1HT Sim Dist column is available in a 5m, 0.53mm ID, 0.1µm film to conform to the requirements of ASTM Method D-6352. It exceeds the criteria for resolution, peak shape, and bleed for hydrocarbons ranging up to C110.

MXT®-1HT SimDist (metal column)

Length (m)	ID (mm)	df(µm)	Temp. Limits	cat.#
5	0.53	0.10	-60 to 430°C	70100

***Please note:** For high-temperature analyses such as simulated distillation, Restek strongly recommends the use of the following accessories to ensure low bleed and maintain high performance.*

Capillary Graphite Ferrules

For 1/16" compression-type fittings

Ferrule ID (mm)	Fits Column ID (mm)	10-pack	50-pack
0.8	0.53	20202	20224

For Agilent GCs (compact ferrules)

Ferrule ID (mm)	Fits Column ID (mm)	10-pack	50-pack
0.8	0.53	20252	20253

For M4 fittings for QCQ ThermoQuest 8000 & Trace GCs

Ferrule ID (mm)	Fits Column ID (mm)	2-pack	10-pack
0.8	0.53	20284	20285

Standard Graphite Ferrules

For 1/4" fittings

Fitting Size (")	Ferrule ID (")	10-pack
1/4	1/4	20210

Leak Detective™ Electronic Leak Detector

- Compact, lightweight, hand-held design.
- Lowest-cost thermal conductivity leak detector available.
- Contamination-free leak detection.
- Detects helium or hydrogen trace leaks at ≥3 x 10^{-4}cc/sec. or ≥200ppm.
- Audible alarm and LED readout.
- Responds in less than 2 seconds to trace leaks of gases.*
- Operates on two 9-volt batteries or AC adaptor, both included.

(110 VAC): cat.# 21607, (ea.)
(220 VAC): cat.# 21609, (ea.)
European 2-prong plug (220 VAC): cat.# 21382 (ea.)
*Not designed for use in explosive atmospheres.

Hot Tech Tip

Many GC problems can be avoided by electronic leak checking the system during the plumbing process. Thorough leak checking will prevent loss of GC gases, damage to capillary columns, and increased detector maintenance. Oxygen can move into the system via a leak due to the Venturi effect, and irreversible damage can occur if a column is exposed to oxygen at high temperatures, such as those needed for simulated distillation. Also, some detectors are very sensitive to oxygen. Leak checking the instrument before column installation and conditioning prevents column degradations indicated by high bleed and short lifetimes. Leak checking should be performed on the entire gas system and GC. Begin by checking all fittings inside the GC. Next check the external fittings along the carrier gas lines, all the way to the tanks. Never use liquid leak detectors that contain soap or surfactants because liquids can be drawn inside the fitting at the site of the leak and contaminate the system.

D6352-98 Polywax® Standards

These high molecular weight hydrocarbon waxes are useful for simulated distillation and other high-temperature GC work.

Ea.	cat.#	qty.
Polywax 500	36224	1 gram
Polywax 655	36225	1 gram
Polywax 850	36226	1 gram
Polywax 1000	36227	1 gram

Super-Clean™ SGT Gas Filters

- High-purity output (99.9999% purity).
- Features a "quick connect" for fast and simple cartridge changes.
- Full glass/metal design with easy-to-read indicators.

Ultra-High Capacity Oxygen Filter:
cat.# 22029, (ea.)

Single-Position Baseplate:
cat.# 22025, (ea.)

Trace-Level Analysis of Explosives by HPLC

Pinnacle II™ C18 & Cyano Columns

by Greg France, HPLC Product Marketing Manager

- ✔ Strictly controlled silica manufacturing ensures reproducible chromatography.
- ✔ Sharp peak shape and excellent efficiency for explosives.
- ✔ Economically priced.

Pinnacle II™ high performance liquid chromatography (HPLC) stationary phases were designed to function well under the difficult matrices encountered in environmental samples. The original Pinnacle™ columns served as benchmarks for the selectivity and efficiency of these new Pinnacle II™ columns. While striving to create columns with characteristics similar to Pinnacle™ columns, Restek designed them using Restek silica. Now, we can go a step further in providing consistent quality and reproducibility by controlling the manufacturing process back to the raw material stage.

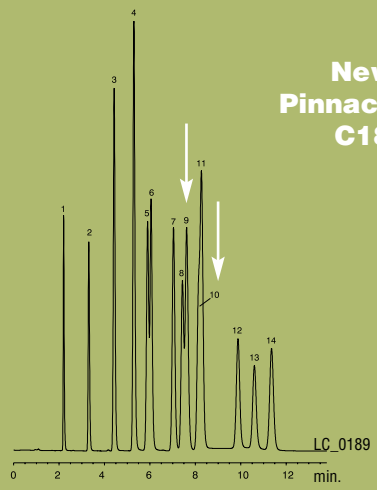
The new Pinnacle II™ C18 and Cyano columns function as primary and confirmation columns (respectively) to efficiently separate explosives according to US Environmental Protection Agency (EPA) Method 8330A. Environmental methods frequently employ a confirmation column for two reasons. First, many environmental methods require scanning for a large number of related compounds. Because of their similarities, analysts often will encounter coelutions when using a single type of stationary phase. Second, the matrices encountered in many environmental samples can contain components that may interfere or obscure the analytes of interest. By using two columns with different selectivities, analysts can more accurately identify the analytes of interest.

Selectivity for the 14 explosives of interest listed in Method 8330A are similar on the original Pinnacle™ and the new Pinnacle II™ C18 columns (Figure 1). On these C18 columns, there are closely eluting peaks or coelutions for the following compounds: tetryl/nitrobenzene; 2-amino-4,6-dinitrotoluene/4-amino-2,6-dinitrotoluene; and 2,6-dinitrotoluene/2,4-dinitrotoluene. Closer examination shows that the new Pinnacle II™ C18 column achieves better resolution for two of these pairs. This may be caused by the slightly higher surface area and carbon load, and the smaller pore size on the Pinnacle II™ column (110Å) as compared to the Pinnacle™ column (120Å). The higher carbon load of 13% for the Pinnacle II™ column, versus 11% for the Pinnacle™ column, translates into longer compound retention, and better resolution and column lifetime.

According to Method 8330A, these 14 compounds also need to be analyzed on a Cyano column for confirmation (Figure 2). Changing from a reversed phase C18 column to a normal phase Cyano column is fairly easy. The method recommends using the same mobile phase for both columns, which allows a quick changeover from the primary analysis to the confirmation analysis. Because the mobile phase is a simple mixture of water and methanol, the process of switching from the C18 to the Cyano column is only a matter of removing the primary column and installing the confirmation column on the same HPLC system. There is relatively little downtime, only that required for the system to re-equilibrate.

Figure 1

The new Pinnacle II™ C18 column achieves better resolution for difficult-to-analyze pairs of explosive compounds.

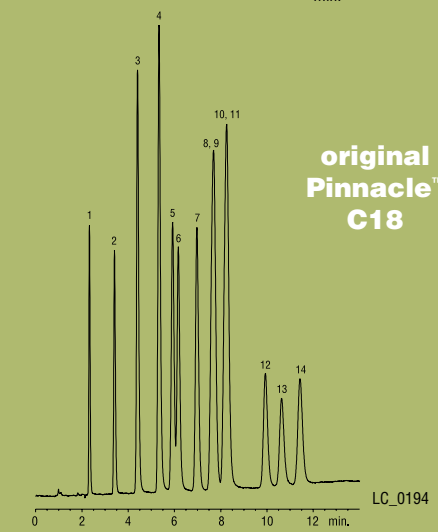


Column: Pinnacle II™ C18
Catalog #: 9214575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 110Å

Conditions:
Mobile phase: water:methanol (50:50, v/v)
Flow: 1.5 mL/min
Temp.: 27°C
Det.: UV @ 254nm

Sample:
Inj.: 3µL
Conc.: 500µg/mL each component
Solvent: acetonitrile

Restek 8330 Calibration mix #1 (cat. # 31450)
Restek 8330 Calibration Mix #2: (cat. # 31451)



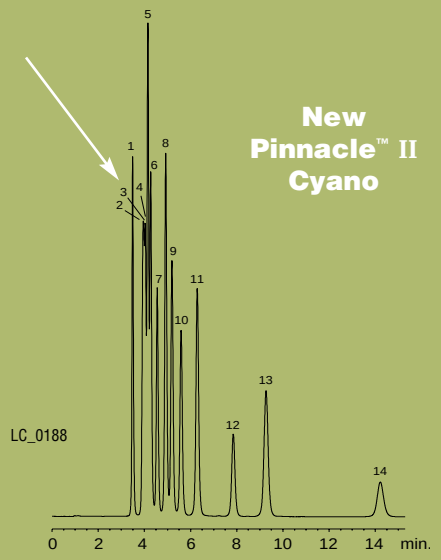
Column: Pinnacle™ C18
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 120Å

Peak List for Figure 1

1. HMX
2. RDX
3. 1,3,5-trinitrobenzene
4. 1,3-dinitrobenzene
5. tetryl
6. nitrobenzene
7. 2,4,6-trinitrotoluene
8. 2-amino-4,6-dinitrotoluene
9. 4-amino-2,6-dinitrotoluene
10. 2,6-dinitrotoluene
11. 2,4-dinitrotoluene
12. 2-nitrotoluene
13. 4-nitrotoluene
14. 3-nitrotoluene

Figure 2

The Pinnacle II™ Cyano column shows better resolution than the original Pinnacle™ Cyano column for explosives compounds, and is an excellent confirmational column to the Pinnacle II™ C18 for this analysis.



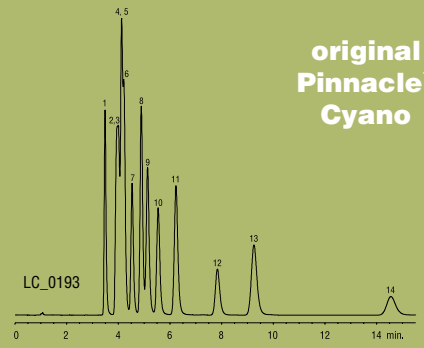
New Pinnacle™ II Cyano

Column: Pinnacle II™ Cyano
Catalog #: 9216575
Dimensions: 250 x 4.6mm
Particle Size: 5µm
Pore Size: 110Å

Conditions:
Mobile phase: water:methanol (50:50, v/v)
Flow: 1.5mL/min
Temp.: 27°C
Det.: UV @ 254nm

Sample:
Inj.: 3µL
Conc.: 500 µg/mL each component
Solvent: acetonitrile

Restek 8330 Calibration mix #1 (cat.# 31450)
Restek 8330 Calibration Mix #2 (cat.# 31451)



original Pinnacle™ Cyano

Column: Pinnacle™ Cyano
Catalog #: 9116575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 120Å

Notice that all of the coeluting pairs from the C18 column are resolved from one another on the Cyano column. There is a cluster of compounds: 2-nitrotoluene, 3-nitrotoluene, 4-nitrotoluene, 1,3,5-trinitrotoluene, and 1,3 dinitrotoluene on the Cyano column, but these compounds are well resolved on the C18 column. Again, the selectivity between the original Pinnacle™ Cyano and the new Pinnacle II™ Cyano columns is similar, but the Pinnacle II™ column shows better resolution.

Restek controls the raw material quality from the very beginning of the silica manufacturing process. Add our phase bonding and column packing experience to this high level of quality control, and you benefit from even better column-to-column reproducibility. Because of this and their economical production, Pinnacle II™ HPLC columns provide a cost-effective analytical tool for many traditional methods used in the environmental industry.

Peak List for Figure 2

1. nitrobenzene
2. 2-nitrotoluene
3. 4-nitrotoluene
4. 3-nitrotoluene
5. 1,3-dinitrobenzene
6. 1,3,5-trinitrobenzene
7. 2,6 dinitrotoluene
8. 2,4-dinitrotoluene
9. 2,4,6-trinitrotoluene
10. 4-amino-2,6-dinitrotoluene
11. 2-amino-4,6-dinitrotoluene
12. RDX
13. tetryl
14. HMX

Pinnacle II™ C18 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
50mm	9214551	9214552	9214553	9214555
100mm	9214511	9214512	9214513	9214515
150mm	9214561	9214562	9214563	9214565
250mm	9214571	9214572	9214573	9214575

Pinnacle II™ Cyano 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
50mm	9216551	9216552	9216553	9216555
100mm	9216511	9216512	9216513	9216515
150mm	9216561	9216562	9216563	9216565
250mm	9216571	9216572	9216573	9216575

Trident™ HPLC Guard Column Cartridges

Guard Column Cartridge	10 x 2.1mm 3-pk.	10 x 4.0mm 3-pk.	20 x 4.0mm 2-pk.
Pinnacle II™ C18	921450212	921450210	921450220
Pinnacle II™ Cyano	921650212	921650210	921650220

Trident™ Direct Guard Column System*

Description	qty.	cat.#
High pressure filter	each	25082
1cm guard cartridge holder with filter	each	25084
2cm guard cartridge holder with filter	each	25086
PEEK® connection tip for Waters®-style end fittings	each	25088
Replacement cap frits: 4mm, 2.0µm	5-pack	25022
Replacement cap frits: 4mm, 0.5µm	5-pack	25023
Replacement cap frits: 2mm, 2.0µm	5-pack	25057

The Trident™ Direct guard column system offers three levels of protection:

Trident™ Direct high-pressure filter
Protection against particulate matter

Trident™ Direct 1cm guard cartridge holder with filter
Moderate protection against particulate matter and irreversibly-adsorbed compounds

Trident™ Direct 2cm guard cartridge holder with filter
Maximum protection against particulate matter and irreversibly-adsorbed compounds

*The standard PEEK® tip in Trident™ Direct systems is compatible with Parker, Upchurch®, Valco®, and other CPI-style fittings. To use Trident™ Direct systems with Waters®-style end fittings, the tip must be replaced with cat.# 25088.

Fast Semivolatile Analysis by GC/MS Using Performance-Based Measurement Systems

Rtx®-5Sil MS Columns and Uniliner® Liners

by Gary Stidsen, Innovations Team Manager

- ✔ Decrease analysis time to 22 minutes for increased lab throughput.
- ✔ Resolve key analytes.
- ✔ Analytical conditions can be used for all MS detectors.

Restek has developed a fast GC/MS method for analyzing semivolatile compounds [e.g., US Environmental Protection Agency (EPA) Method 8270] that will help increase productivity in the lab. The changes include modification of the final extract volume, use of the DI Uniliner with a hole, shorter GC analysis time, and a modification of the calibration curve to offset the increased extract volume. Following is an explanation of each modification. For more detailed information, please request application note #59125.

1) Increase the extract volume

Increase the final extract volume from 1mL to 5mL. This will reduce preparation time and the amount of low-boiling compounds lost from evaporation. Also, one-fifth the amount of matrix imterferences will be injected into the GC. The reduction of matrix interferences will allow the instrument to stay calibrated for more sample windows.

2) Use a Uniliner® DI injection port liner with a hole

This unique inlet liner can be used for direct and splitless injection. The column is fixed via a press-tight connection at the bottom of the liner, eliminating any sample contact with metal parts below the liner. In order for the carrier gas to be routed through the split vent line, a hole has been drilled into the side of the liner. This hole allows the carrier gas to be vented through the split vent line during the split operation of the injection port. The Uniliner® liner with a hole provides a more inert sample pathway that minimizes injection port discrimination and active compound loss.

3) Use a thin-film Rtx®-5Sil MS column

Lower concentration standards allow the use of a thinner-film column, which can reduce analysis time. A thin-film Rtx®-5Sil MS column can achieve improved resolution of difficult-to-analyze compounds in less than 22 minutes. This column features a silarylene stationary phase and optimized dimensions that are ideal for the analysis of semivolatile compounds (Figure 1).

4) Oven temperature programming

Adjusting the oven temperature program of the GC will help optimize the separation of critical pairs. Use of an initial hold time resolves early-eluting compounds; then a fast ramp rate can be used through non-critical areas, and a lower ramp rate used to elute later compounds.

5) Calibration curve

We used 1/5 the recommended concentration level of Method 8270—1µL injection of 4, 10, 16, 24, and 32ppm standard. Notice how the 4ppm (4ng on-column) injection shows excellent signal-to-noise ratio, and low column bleed and injection port discrimination (Figure 1).

Conclusion

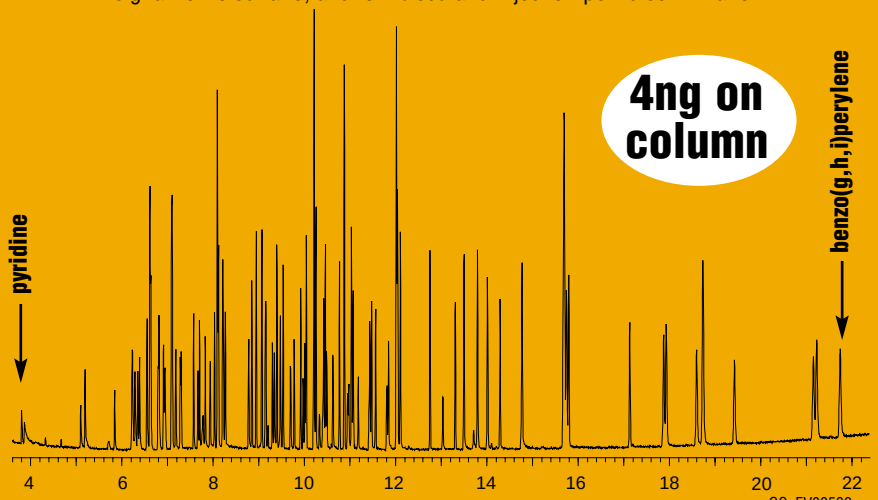
A number of techniques can be used to increase sample throughput for the analysis of semivolatile compounds according to US EPA Method 8270. Increasing extract volume will reduce preparation time and injection port contamination. Using a Uniliner® injection port liner with a hole results in a more inert sample pathway and eliminates injection port discrimination. In addition, the use of a thin-film column reduces analysis time.

for more info

on the analysis of semivolatile compounds, request lit. cat.# #59411, *A Guide to Preparing and Analyzing Semivolatile Organic Compounds.*

Figure 1

Fast analysis of Method 8270 semivolatile compounds with a Uniliner® liner achieves excellent signal-to-noise ratio, and low bleed and injection port discrimination.



30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)

Inj.: 1µL splitless injection of 8270 Calibration Mix #1 (cat.# 31618), 8270 Calibration Mix #2 (cat.# 31619), 8270 Calibration Mix #3 (cat.# 31620), 8270 Calibration Mix #4 (cat.# 31621), 8270 Calibration Mix #5 (cat.# 31622), BN Surrogate (cat.# 31024), Acid Surrogate (cat.# 31025), SU Internal Standard (cat.# 31006); **Flow rate:** helium, constant flow @ 1mL/min.; **Oven temp.:** 35°C (hold 2 min.) to 260°C @ 20°C/min. to 330°C @ 6°C/min. (hold 1 min.); **Inj. temp.:** 300°C; **Detector:** Agilent 6890 w/ 5973 MS; **MS transfer temp.:** 280°C; **Inlet liner:** 4.0mm ID Uniliner® w/hole (cat.# 21054)

Rtx®-5Sil MS (fused silica)

Length (m)	ID (mm)	df(µm)	Temp. Limits	cat.#
30	0.25	0.25	-60 to 330/350°C	12723
30	0.25	0.50	-60 to 330/350°C	12738

Uniliner® Liner with Hole

4.0mm ID, 6.3mm OD, 78.5mm length

each	5-pk.
21054, $60	21055

Analyzing Trace Sulfur Compounds in CO_2

Rt-XLSulfur™ Packed Column and Sulfinert™ System

by Neil Mosesman, GC Columns Product Marketing Manager, and Barry Burger, Innovations Chemist

✔ Detects sulfur compounds at low ppbv levels.
✔ Thermal stability to 300°C for longer column lifetime.

The taste and aroma of a carbonated beverage can be affected by trace impurities from the carbonation process. Therefore, gas producers go to great lengths to purify carbon dioxide (CO_2). Carbon dioxide, a by-product of oil refining, fermentation, and power generating facilities, must be extremely pure to be suitable for a beverage additive. The beverage industry has spent much research time and money monitoring the impurities in CO_2.

The most common impurities in CO_2 are hydrocarbons, alcohols, permanent gases, and sulfur compounds. Sulfur impurities are the predominant problem, adding unwanted taste and odor to beverages. The most common volatile sulfur compounds (VSC) impurities are targeted for monitoring by the International Society of Beverage Technologists (ISBT) (Table I). Of this group, hydrogen sulfide, carbonyl sulfide, sulfur dioxide, dimethyl sulfide, and methyl mercaptan are the ones most commonly found in beverage-grade CO_2. ISBT guidelines specify Total Sulfur Content* (TSC) as 0.1ppm (v/v) maximum, excluding sulfur dioxide; the maximum level of sulfur dioxide must not exceed 1ppm v/v maximum.

The ability to measure reactive sulfur compounds at these levels requires a highly inert chromatography system. The Restek Rt-XLSulfur™ micropacked column is a powerful analytical tool that can detect sulfurs in CO_2 at levels of 20ppbv, far below the ISBT guideline for total sulfur content. This column also achieves the critical separation of hydrogen sulfide, carbonyl sulfide, and sulfur dioxide as defined in ISBT Procedure 14.0. The Rt-XLSulfur™ micropacked column contains a modified divinyl benzene polymer packed into Sulfinert™ tubing, which is a metal tubing specially deactivated for monitoring ppbv levels of active sulfur compounds. Other features of the Rt-XLSulfur™ column include low bleed and thermal stability up to 300°C.

Sample introduction into the column is another critical aspect of obtaining accurate analytical results for sulfur compounds. The sample is introduced onto the column using a Valco® six-port sampling valve, fitted with a 1mL sampling loop (cat. #22845). When the valve, sample loop, and all other surfaces in the sample pathway are deactivated using the Sulfinert™ process, the analyst will see improved response compared to systems using conventional deactivations. We suggest connecting your bulk CO_2 via this system (Figure 1). The specialized inertness of the Sulfinert™ process is critical for the system to achieve detection limits of 50ppbv for sulfur dioxide and the other target sulfur impurities.

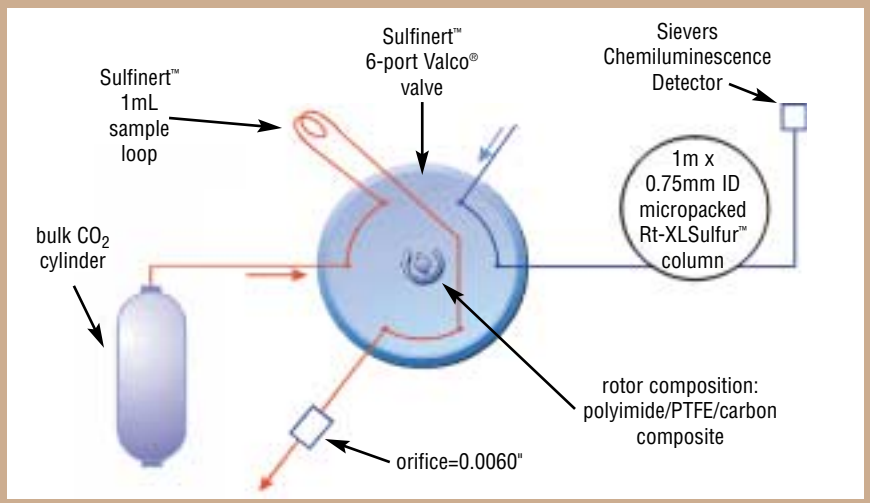
We evaluated the effectiveness of the RT-XLSulfur™ column and Sulfinert™ sampling system by analyzing bulk CO_2 and CO_2 spiked with a sulfur standard (Figure 2). Notice how even low ppbv of sulfurs can be detected. We also sampled and measured the TSC* of two top brands of cola and a domestic beer (Figures 3 and 4). The colas show no sulfur content, verifying that the CO_2 used for carbonation was clean. The beer sample shows sulfur compounds that naturally occur during the fermentation process.

This system is sensitive enough to monitor the levels of sulfur in CO_2 during the carbonation process, or in the headspace of the beverage after carbonation. The TSC* generated from headspace sampling of these products demonstrates the ability of the RT-XLSulfur™ column and the Sulfinert™-deactivated GC system to easily detect sulfur compounds at the 20ppbv level. The combination of the Rt-XLSulfur™ micropacked column and a Sulfinert™-deactivated sample introduction system provide a state-of-the-art, robust sampling and analysis technique for ppb levels of VSCs in beverage-grade CO_2.

Table I
Sulfur compounds can affect the taste and aroma of beer.

hydrogen sulfide	isopropyl mercaptan
carbonyl sulfide	methyl ethyl sulfide
methyl mercaptan	*n*-propyl mercaptan
ethyl mercaptan	*tert*-butyl mercaptan
sulfur dioxide	*sec*-butyl mercaptan
dimethyl sulfide	diethyl sulfide
dimethy disulfide	isobutyl mercaptan
carbon disulfide	*n*-butyl mercaptan
	tert-amyl mercaptan

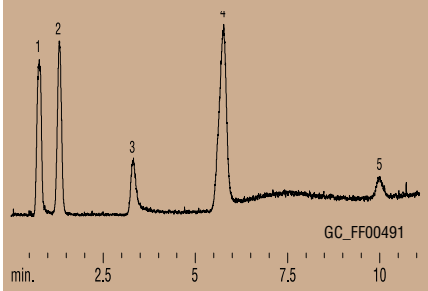
Figure 1
A sampling system was designed to optimize trace-level sulfur analysis without adsorption.



*Total Sulfur Content seen with an asterisk indicates it is without SO_2.

Figure 2

Easily achieve 20ppbv detection limits of reactive sulfur compounds using the Rt-XLSulfur™ packed column and Sulfinert™-treated sample pathways.

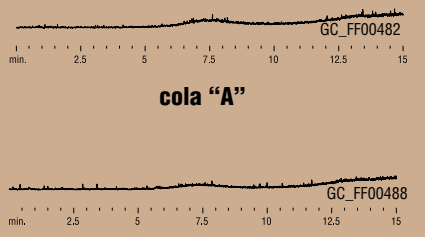


1. hydrogen sulfide
2. carbonyl sulfide
3. methyl mercaptan
4. ethyl mercaptan and/or dimethyl sulfide
5. dimethyl disulfide

1m, 0.75mm ID Sulfinert™ tubing
Rt-XLSulfur™ 100/120 mesh (cat.# 19806)
Oven temp.: 60°C to 260°C @ 15°C/min. (hold 5 min.);
Det. temp.: 800°C; **Carrier gas:** He;
Flow rate: 10mL/ min. @ ambient temp.; **Detector sensitivity:** SCD (Sievers Chemiluminescence Detector)
Attn. x 1; **Inj.:** 1cc sample loop; **Inj. method:** 6-port Valco® valve; **Std. concentration:** sulfur standard @ 20ppb each in CO_2

Figure 3

Two samples of popular colas show no sulfur compounds.



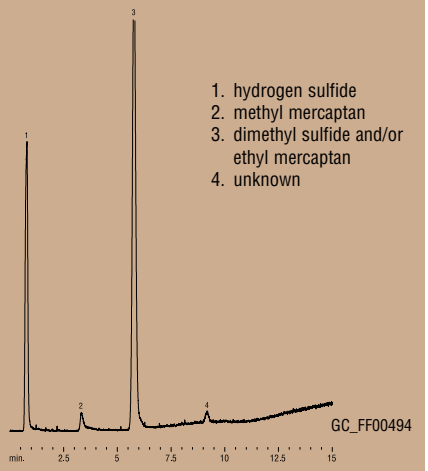
1m, 0.75mm ID Sulfinert™ tubing
Rt-XLSulfur™ 100/120 mesh (cat.# 19806)
Oven temp.: 60°C to 260°C @ 15°C/min. (hold 5 min.);
Det. temp.: 800°C; **Carrier gas:** He;
Flow rate: 10mL/ min. @ ambient temp.; **Detector sensitivity:** SCD (Sievers Chemiluminescence Detector)
Attn. x 1;**Inj.:** 1cc sample loop; **Inj. method:** 6-port Valco® valve; **Std. concentration:** head space of colas

for more info

on Restek's complete line of packed and micropacked columns, and bulk packing materials, request the *Packed Column Catalog* (lit. cat.# 59986A).

Figure 4

A sample of domestic (US) beer contains ppbv levels of hydrogen sulfide, dimethyl sulfide and/or ethyl mercaptan, and methyl mercaptan.



1. hydrogen sulfide
2. methyl mercaptan
3. dimethyl sulfide and/or ethyl mercaptan
4. unknown

1m, 0.75mm ID Sulfinert™ tubing
Rt-XLSulfur™ 100/120 mesh (cat.# 19806)
Oven temp.: 60°C to 260°C @ 15°C/min. (hold 5 min.);
Det. temp.: 800°C; **Carrier gas:** He;
Flow rate: 10mL/ min. @ ambient temp.; **Detector sensitivity:** SCD (Sievers Chemiluminescence Detector)
Attn. x 1; **Inj.:** 1cc sample loop; **Inj. method:** 6-port Valco® valve; **Std. concentration:** head space of a domestic (US) beer sample

Sulfinert™ Sample Loops

size	cat.#
5µL	22840
10µL	22841
20µL	22842
25µL	22843
50µL	22844
100µL	22845
250µL	22846
500µL	22847
1cc	22848
2cc	22849
5cc	22850

6-Port Valco® Valve

The 6-port Valco® valve was coated with Sulfinert™ treatment on a custom basis. For custom Sulfinert™ quotes, call customer service at ext. 3, or contact your local Restek representative.

** Please include configuration suffix number when ordering.*

***Installation kit must be purchased with column when using for valve applications.*

for more info

on Sulfinert™ coating's features and benefits, and a detailed product listing, *Sulfinert™ Flyer* (lit. cat.# 59203).

Rt-XLSulfur™ Packed and Micropacked Columns

OD (in.)	ID (mm)	1-Meter	2-Meter
1/16**	1.0	19804	19805
0.95mm**	0.75	19806	19807
1/8**	2.0	80484*	80485*
3/16	3.2	80482*	80483*

Installation Kits

	for 0.75mm ID col.	for 1mm ID col.	for 2mm ID col.
For valve applications	21062	21065	21067
For split applications	21063	—	—
For all Agilent GCs	21064	—	—
For direct injections	—	21066	—

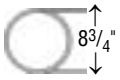
Packed Column Configurations

Custom configurations are available. Please contact Customer Service (ext. 3) or your local Restek representative.

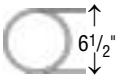
General Configuration: **Suffix -800**

Agilent 5880, 5890, 5987: **Suffix -810**

Varian 3700, Vista Series, FID: **Suffix -820**



PE 900-3920, Sigma 1,2,3: **Suffix -830**



PE Auto System 8300, 8400, 8700 (Not On-Column): **Suffix -840**

Sulfinert™ Tubing (Price-per-foot by length)

ID, OD	cat.#	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
316 Seamless Stainless Steel Tubing (0.035" wall thickness)					
0.011" (0.28mm), .022" (0.56mm),	22500				
0.021" (0.53mm), .029" (0.74mm)	22501				
0.010" (0.25mm), 1/16" (1.59mm)	22502				
0.020" (0.51mm), 1/16" (1.59mm)	22503				
0.030" (0.76mm), 1/16" (1.59mm)	22504				
0.040" (1.02mm), 1/16" (1.59mm)	22505				
0.085" (2.16mm), 1/8" (3.18mm)*	22506				
0.210" (5.33mm), 1/4" (6.35mm)*	22507				
316 Seamless Stainless Steel Tubing (0.035" wall thickness)					
0.055" (1.40mm), 1/8" (3.18mm)	22508				
0.180" (4.57mm), 1/4" (6.35mm)	22509				

Reduced Endrin Breakdown for Chlorinated Pesticides Analysis

Stx™-CLPesticides GC Column Pairs

by Lydia Nolan, Environmental Innovations Chemist, and
Gary Stidsen, Innovations Team Manager

- ✔ Siltek™-deactivated analytical columns decrease endrin breakdown.
- ✔ Same selectivity as Rtx®-CLPesticides column pairs—no modification to analysis parameters.
- ✔ Flow adjustments improve separation.

Many laboratories performing gas chromatography (GC) analysis of chlorinated pesticides struggle with endrin breakdown caused by the compound adsorbing to active sites throughout the analytical system, especially in the injection port and the analytical column. Restek Siltek™ technology—used successfully to passivate injection port liners and guard columns—is now available in Stx™-CLPesticides and Stx™-CLPesticides2 capillary columns. The combination of a properly deactivated injection system and inert analytical columns provides the lowest possible level of endrin breakdown.

Method Requirements

Chlorinated pesticide analyses following US Environmental Protection Agency (EPA) Methods 8081, 608, 505 and 508 recommend dual-column confirmation using electron capture detection (ECD). The most common analytes of interest are analyzed in Figure 1. As in all analytical methods, the instrument used for quantitative analysis must be calibrated to ensure accurate results are reported. For chlorinated pesticides this usually entails a calibration curve of three to five points and check standards injected at specified time intervals during sample analysis. In addition, performance standards containing endrin are analyzed periodically to ensure system inertness. Typically, endrin breaks down to endrin aldehyde and endrin ketone when there are active sites in the sample pathway, and their presence must also be verified.

Endrin Breakdown

Maintaining a low breakdown level for endrin is necessary for laboratories analyzing chlorinated pesticides. Reduction of endrin breakdown generally focuses on improving the inertness of the injection port (see sidebar on page 9). Recently, liners treated with Siltek™ deactivation were designed to prevent endrin breakdown in the injection port. This innovative deactivation technology also was incorporated into capillary guard tubing so that the entire sample introduction pathway is as inert as possible.

Endrin Response

In addition to breakdown, endrin response can be reduced by irreversable adsorption onto active sites in the sample pathway. To minimize on-column adsorption of endrin, we incorporated Siltek™ deactivation technology into the analytical columns.

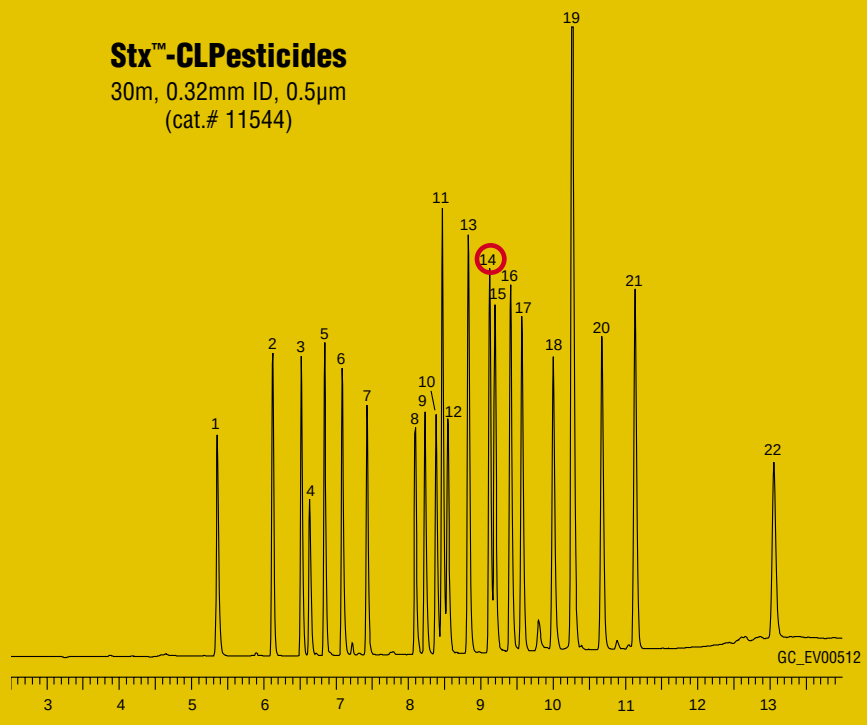
Combining Siltek™ deactivation with the unique selectivity of Restek CLPesticides phases results in the new Stx™-CLPesticides and Stx™-CLPesticides2 columns, and a significant improvement in endrin response compared to "Rtx" columns (Figure 1). Using the Stx™-CLPesticides and Stx™-CLPesticides2 columns, the endrin peak response is now higher than the analytes eluting in the same region—something not seen consistently in columns using traditional deactivations.

Column Installation and Optimizing Resolution

The Stx™-CLPesticides2 column is the ideal confirmational column to the Stx™-CLPesticides column. It was designed to achieve resolution of the chlorinated pesticides using the same flowrate and oven temperature program. The columns can be installed in parallel using a universal Siltek™ Press-Tight® "Y" connector or a metal MXT® "Y" connector. This parallel set-up reduces downtime caused by maintenance of multiple injection ports. Additionally, these columns can be installed in separate injection ports and mounted in the same GC oven.

Figure 1

Highly inert Stx™-CLPesticides and Stx™-CLPesticides2 columns provide excellent response for active pesticides such as endrin, DDT, and methoxychlor so you get accurate quantitation.



Oven temp.: 110°C (hold 1 min.) to 245°C @ 20°C/min. to 300°C @ 6°C/min.; **Inj. & det. temp.:** 210°C / 310°C; **Carrier gas:** helium; **Dead time:** 0.8min. @ 120°C; **Inlet liner:** Siltek™ Uniliner® w/hole (cat.# 21055-214.5); **Inj.:** 1µL direct injection of 20/40/200ng/mL std. concentration in hexane; **Make-up gas:** nitrogen

Stx™-CLPesticides Columns

ID (mm)	df (µm)	temp. limits	15-Meter	30-Meter
0.25	0.25	-60 to 310/330°C	11540	11543
0.32	0.50	-60 to 310/330°C	11541	11544
0.53	0.50	-60 to 310/330°C	11542	11545

Stx™-CLPesticides2 Columns

ID (mm)	df (µm)	temp. limits	15-Meter	30-Meter
0.25	0.20	-60 to 310/330°C	11440	11443
0.32	0.25	-60 to 310/330°C	11441	11444
0.53	0.42	-60 to 310/330°C	11442	11445

Siltek™ Inlet Liners

For Siltek™-deactivation, include the suffix number to the inlet liner catalog number.

Qty.	Siltek™	Siltek™ w/Siltek™ Wool	Siltek™ w/Carbofrit™
each	-214.1, $5 addl. cost	-213.1	-216.1
5-pk.	-214.5, $20 addl. cost	-213.5	-216.5
25-pk.	-214.25, $90 addl. cost	-213.25	-216.25

Siltek™ Guard Columns

ID	5-Meter (ea.)	10-Meter (ea.)
0.25mm	10026	10036
0.32mm	10027	10037
0.53mm	10028	10038

Stx™-CLPesticides2

30m, 0.32mm ID, 0.25µm
(cat.# 11444)

Siltek™—by Restek

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

Injection Port Maintenance

Using Siltek™-treated Stx™-CLPesticides columns will improve your chlorinated pesticide analyses, but routine instrument maintenance will also help. The injection port is where a majority of analytical problems occur in the analysis of pesticides. The main problem is the cleanliness and inertness of the injection port with which the sample comes in contact. Endrin breakdown is usually indicative of a chemical reaction taking place in the injection port. The breakdown could be caused by impurities in the carrier gas, active metal surfaces, a non-deactivated inlet liner, or septa particles.

The carrier gas is usually the last troubleshooting area investigated and the hardest to eliminate. Endrin may react with a contaminant being carried into the injection port by the carrier gas. Having gas scrubbers in-line for the carrier gas will help keep this problem from occurring.

The metal surfaces of the injection port must be kept clean, including the inlet carrier gas line. Periodic rinsing of the carrier gas lines and cleaning the inside of the injection port may be necessary if endrin or 4,4'-DDT breakdown increases over short periods of time or when only analyzing standards. Rinsing of metal surfaces using solvents (e.g., methylene chloride, hexane or acetone), or in some cases silanizing the injection port, has helped. Also, Restek can coat your injection port with Sulfinert™ treatment for better inertness.

Improperly deactivated injection port liners are the primary cause of endrin breakdown. The best way to avoid this problem is to replace the liner with a Siltek™-deactivated liner when performing routine maintenance. Also, there is a standard procedure for deactivating liners that includes a process of cleaning the liners in acid and deactivating with dichlorodimethylsilane.

Septa particles are another cause of endrin breakdown. The septa particles will sit on top of a glass wool plug or at the bottom of the liner. To help eliminate septum coring, make sure your syringe needle does not have burrs. Another approach is to try different septa that features reduced coring, such as InfraRed™ septa (see pg. 14), and to change septa more often.

For more detailed information on chlorinated pesticide analysis, please request *A Guide to Preparing and Analyzing Chlorinated Pesticides* (lit. cat.# 59892).

GC Analysis of Acids, Esters, and Other Flavor Components in Distilled Liquor Products

Stabilwax®-DA GC Column—Part I

by Rebecca E. Wittrig, Ph.D., Food, Flavor, and Fragrance Innovation Team Leader; and Kevin MacNamara, Ph.D., Irish Distillers Ltd.*

- ✔ High thermal stability (250°C) and solvent rinsibility result in long column lifetime.
- ✔ Optimized configuration reduces analysis time by 4-fold.
- ✔ Low bleed for accurate quantitation.

Part I: The Separation

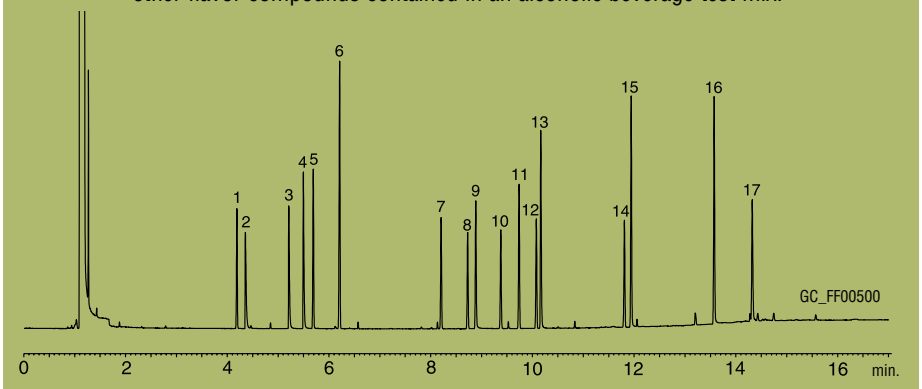
Distilled liquor products contain a wide range of volatile and non-volatile compounds in an ethanol/water matrix. The most abundant fusel alcohols and esters can be determined by gas chromatography (GC) with a simple split injection, which also minimizes the amount of matrix ethanol and water transferred to the column. However, many additional trace fatty acids and their esters—often used to indicate quality of alcoholic beverages such as whiskey and rum—cannot be determined by this approach. Because the concentrations can vary widely, splitless injection techniques with some type of preconcentration step usually are necessary to analyze fatty acids, esters, and other flavor compounds.

Large ranges of volatility and acidity for the component list make quantitating them in a single separation difficult to accomplish. The Stabilwax®-DA column, a bonded polyethylene glycol (PEG) phase, is excellent for analyzing alcoholic beverage samples (Figure 1). Using this column, flavor compounds in distilled liquor products can be quantitated in a single splitless injection. An optimized configuration of 30m, 0.18mm ID, 0.18µm allows significantly reduced analysis times. To improve the peak shape and reproducibility of acidic components in these samples, an acidic functionality has been added to the backbone of the PEG stationary phase. This results in less adsorption of free fatty acids and, thus, significantly less peak tailing. Also, because of the inertness of this stationary phase towards acidic components present in the sample, greater reproducibility and longer column lifetimes are possible.

Part II of this article will appear in the Winter '01/'02 Advantage. We will look at the Stabilwax®-DA column for alcoholic beverage analysis. Trace-level components, such as the ones displayed in Figure 1, often can be used to "fingerprint" a particular type or brand of distilled liquor. The application of this methodology to whiskey products will be discussed in Part II.

Figure 1

The Stabilwax®-DA column provides excellent peak shape of free fatty acids, esters, and other flavor compounds contained in an alcoholic beverage test mix.

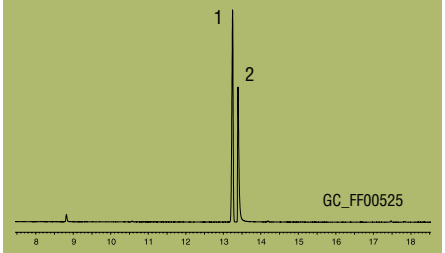


30m, 0.18mm ID, 0.18µm Stabilwax®-DA (cat.# 550752)
Oven temp.: 70 to 240°C at 12°C/min. (hold 3 min.); **GC:** ThermoQuest Trace 2000; **Inj.:** 1µL splitless at conc. shown in peak list, in ethyl acetate; **Detector:** FID; **Inj. & det. temp.:** 240°C; **Liner:** 4mm ID splitless liner w/wool (cat.# 20814-202.1); **Hold time:** 0.5 min.; **Carrier gas:** hydrogen; **Make-up gas:** nitrogen; **Linear velocity:** 28psi @ 240°C

One of the critical pairs in the GC analysis of esters and acids in distilled liquor products is caproic acid and ethyl laurate. These components can be difficult to resolve on standard Carbowax®-type columns. This is especially true if peak tailing or broadening occurs, or if one component is present at a significantly higher concentration. The Stabilwax®-DA column achieves baseline resolution of these two compounds within a reasonable analysis time of 30 minutes (Figure 2).

Figure 2

The Stabilwax®-DA column achieves complete resolution of caproic acid and ethyl laurate in 30 minutes.



1.	caproic acid	100 ppm
2.	ethyl laurate	100 ppm

30m, 0.18mm ID, 0.18µm Stabilwax®-DA (cat.# 550752)
Oven temp.: 80 to 230°C at 5°C/min.;
See Figure 1 for complete conditions.

Because alcoholic beverage samples often are injected via splitless mode, the stability of the Stabilwax®-DA column when exposed to aqueous injections is important. We verified stability by performing a splitless injection of the alcoholic beverage test mix, followed by five 1µL injections of water. This process was repeated 10 times, followed by a final injection of the test mix. The final test mix injection can be seen in Figure 3. Even after repeated splitless injections of 100% water, very little degradation occurs in the peak shapes of the test

Peak List for Figures 1 & 3

	compound	conc. (ppm)
1.	ethyl octanoate	100
2.	acetic acid	100
3.	propionic acid	100
4.	isobutyric acid	100
5.	decanol 3	50
6.	ethyl decanoate	50
7.	ethyl laurate	50
8.	*cis*-lactone	100
9.	2-phenylethanol	50
10.	*trans*-lactone	100
11.	methyl myristate	50
12.	ethyl myristate	50
13.	octanoic acid	100
14.	ethyl palmitate	50
15.	decanoic acid	100
16.	dodecanoic acid	100
17.	vanillin	100

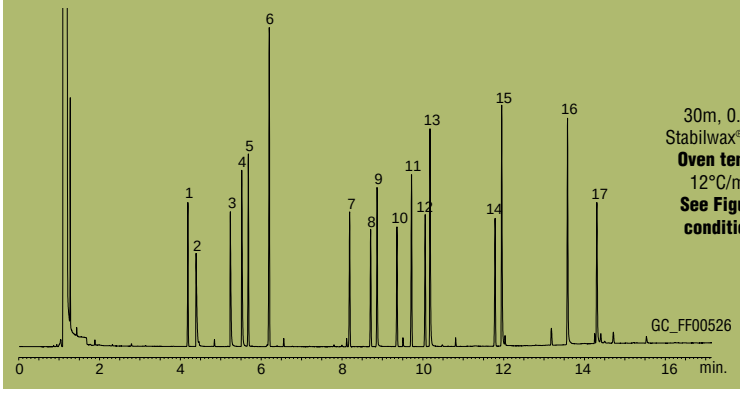
Irish Distillers Ltd., Bow Street Distillery, Smithfield, Dublin 7, Ireland

mix components. Over the course of the study, the variation in the peak retention times was 0.08-0.22% RSD. This includes the polar free fatty acids, which can be difficult to analyze under ideal conditions. The excellent stability of this stationary phase is proven by the reproducibility of the retention times over the course of the water stability study.

The Stabilwax®-DA column is an excellent choice for the analysis of acids, esters, and other flavor components in alcoholic beverage products. This highly stable column has been optimized for the analysis of acidic compounds, making it possible to analyze a wide range of compounds in a single injection. In addition, the column configuration shown in this article allows fast, efficient separation of the test compounds.

Figure 3

Excellent stability and peak shapes for free fatty acids after fifty 1µL splitless injections of water demonstrates column ruggedness and long lifetime.



30m, 0.18mm ID, 0.18µm Stabilwax®-DA (cat.# 5550752)
Oven temp.: 70 to 240°C at 12°C/min. (hold 3 min.);
See Figure 1 for complete conditions and peak list.

Stabilwax®-DA Columns

ID (mm)	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.18	0.18	40 to 250°C	—	550752	—
0.25	0.10	40 to 250°C	11005	11008	11011
	0.25	40 to 250°C	11020	11023	11026
	0.50	40 to 250°C	11035	11038	11041
0.32	0.10	40 to 250°C	11006	11009	11012
	0.25	40 to 250°C	11021	11024	11027
	0.50	40 to 250°C	11036	11039	11042
	1.00	40 to 240/250°C	11051	11054	11057
0.53	0.10	40 to 250°C	11007	11010	11013
	0.25	40 to 250°C	11022	11025	11028
	0.50	40 to 250°C	11037	11040	11043
	1.00	40 to 240/250°C	11052	11055	11058
	1.50	40 to 230/240°C	11062	11065	11068

4mm Splitless Inlet Liner for ThermoQuest Trace GCs

4.0mm ID, 5.5mm OD, 79.5mm length
Add suffix -202.1 for wool packing.

each	5-pk.	25-pk.
20814	20815	20816

for more info

request the Stabilwax®/MXT®-WAX Fast Facts flyer (lit. cat.# #59316).

Ethanol Analytical Reference Materials

For Blood Alcohol Testing

by Ken Herwehe, Analytical Reference Materials Product Marketing Manager

- ✔ Resolution control standard for calibration of whole analytical system.
- ✔ Custom mixes for 6-point calibration of your instrument.
- ✔ Confidence ensured with a Certificate of Analysis, raw materials test results, statistical QA results, analytical balance printout, and a lot sheet showing gravimetric weight of each analyte.

Ethanol Standards

All standards are available in a 5-ampul minimum purchase.

Expiration date for each solution is 36 months from the date of manufacture.

concentration	*cat.#*	*minimum purchase of 5 ampuls*
25mg/dL in water	550741	5 x 20mL ampuls
40mg/dL in water	550742	5 x 20mL ampuls
100mg/dL in water	550743	5 x 20mL ampuls
150mg/dL in water	550744	5 x 20mL ampuls
200mg/dL in water	550745	5 x 20mL ampuls
300mg/dL in water	550746	5 x 20mL ampuls

Resolution Control Standard

The ethanol concentration will be a certified value. The concentration of the other analytes will be reported as gravimetric concentration only.

Expiration date is 18 months from the date of manufacture.

Contains:

ethanol	methyl ethyl ketone
methanol	acetaldehyde
isopropanol	acetonitrile
acetone	ethyl acetate

concentration	*cat.#*
100mg/dL each compound in water	550778
minimum purchase of 10 ampuls	
10 x 1mL ampuls	

Rtx®-BAC1 Columns

Temp. limits: -20 to 240/260°C

ID	df (µm)	30-Meter
0.32mm	1.80	18003
0.53mm	3.00	18001

Rtx®-BAC2 Columns

Temp. limits: -20 to 240/260°C

ID	df (µm)	30-Meter
0.32mm	1.20	18002
0.53mm	2.00	18000

for more info

request the Application Note *Dual-Column Confirmational GC Analysis of Blood Alcohols Using the Rtx®-BAC1 and Rtx®-BAC2 Columns* (lit. cat.# #59598).

Underground Storage Tank (UST) Reference Materials

for the Latest Method Revisions

by Jingzhen Xu, R&D Chemist, and Ken Herwehe, Analytical Reference Materials Product Marketing Manager

- ✔ Complete reference materials available for Alaska, Tennessee, and Texas methods.*
- ✔ Analytical columns, sleeves, and accessories available from Restek.
- ✔ Custom chemical standards for unique requirements.
- ✔ Product listings for all states available soon.

*Products for 15 state methods listed in 2001 catalog. Call for information on other revisions and look for even more UST information in the next *Restek Advantage*.

State of Texas

Method and regulatory information is available from:
Texas Natural Resources Conservation Commission
Petroleum Storage Tank Division
Mailing address:
MC: 133
P.O. Box 13087
Austin, TX 78711-3087
(*Street address:*12100 Park 35 Circle, Austin, TX 78753)
Phone: (512) 239-2106
Fax: (512) 239-2177
UST: www.tnrcc.state.tx.us/permitting/r_e/pstta
LUST:
www.tnrcc.state.tx.us/permitting/remed/rpr/index.html

TNRCC 1005 for TPH (revision 3, 6-1-2001); Draft TNRCC 1006

Alternate Boiling Point/Carbon Number Distribution Marker Stock Standard

hexane (C6)
octane (C8)
decane (C10)
dodecane (C12)
hexadecane (C16)
heneicosane (C21
octacosane (C28)
pentatriacontane (C35)
hexatriacontane (C36)
200µg/mL each in pentane
1mL per ampul

Ea.	5-pk.	10-pk.
31639	31639-510	—
with data pack		
31639-500	31639-520	31739

Need something special?

Let Restek's Analytical Reference Materials Team prepare a custom solution for you. We offer convenience, high quality, and quick turnaround without "custom" prices. Contact us at 800-356-1688, ext. 4, or contact your local Restek representative for more information.

State of Tennessee

Method and regulatory information is available from:
Tennessee Dept. of Environment and Conservation
Division of Underground Storage Tanks
4th Floor, L & C Tower
401 Church Street
Nashville, TN 37243-1541
Phone: (615) 532-0945
Fax: (615) 532-0938
www.state.tn.us/environment/ust

TN GRO (v. 3-31-1999)

Gasoline Component Standard

Component	*Concentration, (ug/mL)*
2-methylpentane	1500
2,2,4-trimethylpentane	1500
heptane	500
benzene	500
toluene	1500
ethylbenzene	500
m-xylene	1000
p-xylene	1000
o-xylene	1000
1,2,4-trimethylbenzene	1000

10,000µg/mL total in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30486	30486-510	—
with data pack		
30486-500	30486-520	30586

New Stoddard Solvent Standards

Stoddard solvent is also known as Type I mineral spirits, Texsolve S®, or Varsol 1® mineral spirits. Restek now offers this type of mineral spirits for those who need to calibrate Stoddard solvent separately. This new standard is dissolved in methanol for analysis by either direct injection or purge and trap.

10,000µg/mL in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30487	30487-510	—
with data pack		
30487-500	30487-520	30587

State of Alaska

Method and regulatory information is available from:
Alaska Department of Environmental Conservation
410 Willoughby Avenue
Juneau, AK 99801-1795
Phone: (907)465-5203
Fax: (907)465-5218
www.state.ak.us/dec/dspar/stp_home.htm

Alaska Method AK101 and AK101AA (v. 3-1-99)

Retention Time Marker

hexane decane dodecane
1,000µg/mL each in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30483	30483-510	—
with data pack		
30483-500	30483-520	30583

Surrogate Standard

1,4-bromofluorobenzene ααα-trifluorotoluene
2,500µg/mL each in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30484	30484-510	—
with data pack		
30484-500	30484-520	30584

Certified Aromatics in Gasoline

An unleaded gasoline composite standard at 5,500µg/mL with certified concentration values for the following aromatics:

benzene
toluene
ethylbenzene
o-xylene
p-xylene
m-xylene
1,2,3-trimethylbenzene
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
isopropylbenzene
m-ethyltoluene
p-ethyltoluene
o-ethyltoluene
n-propylbenzene
methyl *tert*-butyl ether
naphthalene
1mL per ampul in P&T methanol

Ea.	5-pk.	10-pk.
30485	30485-510	—
with data pack		
30485-500	30485-520	30585

Alaska Methods AK 102, AK 103 (v. 3-1-99) and Methods AK102AA, AK 103AA (v. 6-30-98)

Retention Time Marker Standard

(Methods AK102/103/102AA/103AA)
decane C10 pentacosane C25
hexatriacontane C36
1,000µg/mL each in hexane
1mL per ampul

Ea.	5-pk.	10-pk.
31637	31637-510	—
with data pack		
31637-500	31637-520	31737

Surrogate Standard

squalane *o*-terphenyl
tetrahydronaphthol
1,000µg/mL each in methylene chloride
1mL per ampul

Ea.	5-pk.	10-pk.
31638	31638-510	—
with data pack		
31638-500	31638-520	31738

Natural and Refinery Gas Standards

by Gary Barone, GC Accessories, MPG, and Air Monitoring Products Marketing Manager

- ✔ Three different concentrations available to closely fit your method regulations.
- ✔ Mini-regulator designed specially for the standards.

Natural Gas	Natural Gas Standard #1* cat.# 34438 % of each compound	Natural Gas Standard #2* cat.# 34439 % of each compound	Natural Gas Standard #3* cat.# 34440 % of each compound
nitrogen	1.000	2.500	5.000
carbon dioxide	0.500	1.000	1.500
methane UHP	94.750	85.250	70.000
ethane UHP	2.000	5.000	9.000
propane	0.750	3.000	6.000
isobutene	0.300	1.000	3.000
n-butane	0.300	1.000	3.000
isopentane	0.150	0.500	1.000
n-pentane	0.150	0.500	1.000
hexanes plus EX2**	0.100	0.250	0.500
Concentration	mole	mole	mole
Volume	13.16L @ 200psig	13.16L @ 200psig	5.5L @ 75psig
Ideal Heating Value	1048 gross*	1142 gross*	1317 gross*

From rich to lean, each natural gas mix contains an extended list of C6+ compounds.

Refinery Gas	Refinery Gas Standard #1* cat.# 34441 % of each compound	Refinery Gas Standard #2* cat.# 34442 % of each compound	Refinery Gas Standard #3* cat.# 34443 % of each compound
hydrogen	40.650	12.500	12.500
argon	0.500	1.000	1.000
nitrogen	4.000	37.250	37.250
carbon monoxide	1.000	1.000	1.000
carbon dioxide	3.000	3.000	3.000
methane	8.500	5.000	5.000
ethane	6.000	4.000	4.000
ethylene	2.000	2.000	2.000
acetylene	-	1.000	1.000
propane	7.000	6.000	6.000
propylene	3.000	3.000	3.000
propadiene	0.850	1.000	1.000
isobutane	6.000	5.000	5.000
n-butane	4.000	4.000	4.000
isobutylene	2.000	1.000	1.000
1,3 butadiene	3.000	3.000	3.000
cis-2-butene	2.000	2.000	2.000
trans-2-butene	2.000	3.000	3.000
butene-1	2.000	2.000	2.000
2-methyl-2-butene	-	0.200	0.200
isopentane	1.000	1.000	1.000
n-pentane	1.000	1.000	1.000
cis-2-pentene	-	0.400	0.400
trans-2-pentene	-	0.150	0.150
pentene-1	-	0.400	0.400
n-hexane	0.500	0.100	0.100
Concentration	mole	mole	mole
Volume	5.2L @ 70psig	4.9L @ 60psig	4.6L @ 60psig

Each refinery gas mix contains varying degrees of C5 unsaturates or extended C6+ compounds.

**Dry BTU/SCF @ 14.696psia & 60°F.* ***Contact Restek to get a complete list of hexanes plus EX2.*

Restek now offers standards for natural gas and refinery gas applications. Restek has developed many unique columns and sampling equipment for these industries and continues to expand product offerings for analysts working in these fields. These new standards are shipped in a DOT-4B-240ET cylinder that is 3" in diameter and 9 3/8" high. These cylinders use a CGA 170/110 connection. Restek also offers a mini-regulator specifically made for these standards.

CGA 170 Mini-Regulator

- ✔ For natural gas and refinery gas standards.
- ✔ Inlet pressure range: 0-300psig; outlet pressure range: 0-15psig.
- ✔ Supplied with 0-15psig outlet pressure gauge, brass CGA 170 nut, and nipple.

cat.# 22032, (ea.) $125

Gas-Tight Syringes

- ✔ Teflon®-tipped plungers.
- ✔ Removable needles.
- ✔ Replaceable syringe barrels, plungers, and plunger tips.

Hamilton Syringes

Volume (µL)	Hamilton Model	Hamilton cat.#	Restek cat.#
10	1701	80065	21260
25	1702	80265	21261
50	1705	80965	21262
100	1710	81065	21263
250	1725	81165	21264

SGE Syringes

Volume (µL)	SGE Model	SGE cat.#	Restek cat.#
10	10R-GT-LC	002313	24866
25	25R-GT-LC	003312	24867
50	50R-GT-LC	004312	24868
100	100R-GT-LC	005312	24869
250	250R-GT-LC	006312	24870
500	500R-GT-LC	007312	24871

InfraRed™ Septa

New! High-Performance, Low Bleed

by Gary Barone, GC Accessories Product Marketing Manager

- ✔ Incredible high-quality.
- ✔ Stable to 325°C.
- ✔ Low bleed.
- ✔ Excellent puncturability.
- ✔ Do not adhere to injectors.

Restek introduces the new InfraRed™ septa. These septa are formulated with silicone and filler that maximize thermal stability to 325°C, and provide smooth puncturability and very low bleed. Experience low bleed and long lifetimes using these new InfraRed™ septa.

handy **Septum Size Chart**

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

Measure your old septum here (size in mm)

5 7 9 9.5 10 11 11.5 12.5 17

InfraRed Septa

Septum Diameter	25-pk.	50-pk.	100-pk.
9mm	21417	21418	21419
9.5mm (3/8")	21421	21422	21423
10mm	21424	21425	21426
11mm (7/16")	21427	21428	21429
11.5mm	21430	21431	21432
12.5mm (1/2")	21433	21434	21435
17mm	21436	21437	21438
Shimadzu Plug	21439	21440	21441

IceBlue™ Septa

- ✔ General purpose septa.
- ✔ Excellent puncturability.
- ✔ Preconditioned and ready to use.
- ✔ Do not adhere to hot metal surfaces.
- ✔ Usable to 250°C inlet temperatures.
- ✔ Ideal for SPME.

Septum Diameter	50-pk.	100-pk.
9mm	22381	22382
9.5mm (3/8")	22388	22389
10mm	22390	22391
11mm (7/16")	22392	22393
11.5mm	22383	22384
12.5mm (1/2")	22394	22395
17mm	22396	22397
Shimadzu plug	22398	22399

Thermolite® Septa

- ✔ Usable to 340°C inlet temperatures.
- ✔ Each batch tested on FIDs, ECDs, & MSDs to ensure low bleed.
- ✔ Excellent puncturability.
- ✔ Preconditioned and ready to use.
- ✔ Packaged in non-contaminating glass jars.

Septum Diameter	25-pk.	50-pk.	100-pk.
5mm (3/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9mm	20354	20358	20362
9.5mm (3/8")	20359	20360	20361
10mm	20378	20379	20380
11mm (7/16")	20363	20364	20365
11.5mm	22385	22386	22387
12.5mm (1/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374

Hot Tech Tip

What is Septum Bleed?

Septum bleed occurs when volatile compounds are off-gassed from the septum. During a temperature-programmed analysis, you can see septum bleed in baseline rise and/or extraneous peaks not associated with the sample or the column. Septum bleed is most noticeable during temperature-programmed analyses because the volatiles off-gassed from the septum collect on the head of the analytical column during the cool-down period and initial hold time. Then, these volatiles elute during subsequent runs. Under isothermal conditions, septum bleed is a continuous, steady interference that appears as part of the normal background noise. Either way, septum bleed interferes with quantitation and analysis accuracy. Septum bleed can be affected by inlet temperature, initial hold time, compression, injection mode, etc.; but, the single most important variable is the septum quality. To maintain analytical integrity, use high-quality septa for senstitive or high-temperature analyses.

cool tools

Tired of burned fingers, leaking injection ports, and lost scoring wafers? Try these new tools from Restek.

by Brad Rightnour and Michael Goss, Instrument Innovations Team

special OFFER!

Buy a 25-pk. of inlet liners and receive a *free inlet liner removal tool*!
Offer good through 1/31/02.

GC Inlet Liner Removal Tool

No more burnt fingers!

✔ Easily removes liners from injectors.
✔ Made from high-temperature silicone.
✔ Won't crack or chip the liner
cat.# 20181, (3-pk.)

Gently push the liner removal tool onto the liner in the injection port with a slight circular motion. This will ensure that the removal tool has a firm grip on the liner.

Slowly pull the liner out of the injection port in a straight vertical motion.

Use the liner removal tool to place a new liner into the injection port, carefully avoiding hot metal surfaces.

Injection Port Repair Tool

Remove contaminants, achieve a better seal!

✔ For Agilent split/splitless injection ports.
✔ Resurfaces critical inlet seal areas.
✔ Removes contaminants.
cat.# 21393, (ea.)

The inlet seal at the base of a split/splitless injector allows a seal to form between the injection port and the inlet liner. This inlet seal wears over time and may become scratched or pitted, which compromises the sealing ability of the injector. The new Restek injection port repair tool allows the user to easily resurface the inlet seal and remove contaminants; and it saves time and money by preventing unwanted leaks.

Try Restek's SILCOSTEEL Injection Ports

For more information, request the catalog *Genuine Restek Replacement Parts for Agilent GCs* (lit. cat.# 59627B).

Scoring Wafer with Handle

Same great scoring, better comfort!

✔ Unique, ergonomic handle is made of soft, comfortable plastic.
✔ Ceramic wafer is serrated on one side and straight-edged on the other.
✔ Cuts both fused silica and metal tubing cleanly.
cat.# 23015, (2-pk.)

Hold tubing firmly in one hand, allowing about two inches to extend freely for safe cutting. Hold the scoring wafer at a 45° angle to the tubing Exert slight pressure—just enough to put a slight arc in the tubing. Pull perpendicularly across the tubing. Move your whole arm, not just your hand. This will help ensure a square, consistent score.

If the tubing doesn't fall off on its own, it should easily break at the score with a slight tap of the wafer. If it doesn't, score again. Check the cut against the white of the scoring wafer for a clean, square cut.

RESTEK Behind the Scenes

Use your Wizard Dollars to help!

We all want to do what we can to help with the expenses incurred from the September 11th terror attack. From now until December 31st, 2001, we will donate one US$ for each Wizard$ to the fund of your choice.

If you choose to do so, you can donate the Wizard Dollars from your current order simply by specifying which organization should receive them. Also, you can donate accumulated Wizard Dollars by mailing them to Restek with a Wizard Dollar order form noting one of the listed charities.

Restek Corporation
Attn: Customer Service Wizard $
110 Benner Circle
Bellefonte, PA, 16823

One way we can help is by donating Restek Wizard Dollars to these charities:

- ★ **United Way**
- ★ **Red Cross**
- ★ **Salvation Army**

New Online Tool for GC Optimization

Our Restek webmaster has designed an interactive, multi-purpose tool that gives you the optimization data specific to your column configuration, carrier gas, and detector. Handy when installing or troubleshooting, and indispensable for obtaining the best analytical performance; with reference data, calculators, and procedural information. Find it on our homepage:

www.restekcorp.com

Restek Salutes Those in the Military

We salute all individuals involved in military and rescue efforts both at home and abroad. We especially thank the following members of the Restek family:

Roger Greene, Airforce 913th Security Force Squadron (Restek Personal Trainer)
Ken Herwehe, Army Reserves (Restek Analytical Reference Materials Product Marketing Manager)
Alvira Peak, Civilian Air Corps (Restek Environmental Health and Safety Coordinator)
Matt Reilly, Army National Guard (Restek Applied Technology Manufacturing Technician)
Pete Zucco, Naval Reserves (Restek Maintenance Technician)

New Restek Wizards

Craig Hall, Customer Service Representative
Debra Copenhaver, Customer Service Representative
Diane Thompson, Analytical Reference Materials Shipping Technician
James Weber, Maintenance Assistant
Courtney Johnson, Personal Trainer

ISO 9001 REGISTERED

Lit. Cat.# 59426

Please direct your comments on this publication to Kristin Dick, Editor, at kristind@restekcorp.com or call Restek, ext. 2313.

RESTEK

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PAID
Restek

the RESTEK Advantage
Innovators of High Resolution Chromatography Products

Intermediate Polarity Capillary Column for the GC Analysis of Basic Compounds

new

Rtx®-35 Amine GC Column

by Neil Mosesman, GC Columns Product Marketing Manager

- ✔ Achieves symmetrical peaks for basic compounds.
- ✔ Improved response over traditional columns.
- ✔ Resolves low-molecular-weight primary amines.

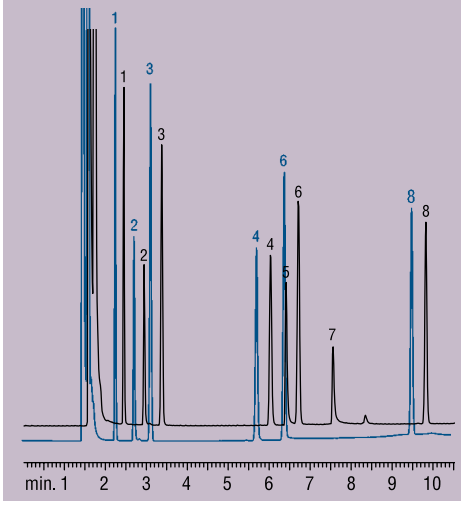
Amines and nitrogen heterocyclics are used to manufacture a wide variety of products including dyes, chelating agents, stabilizers, pesticides, and pharmaceuticals. Analyzing amines and other basic compounds by gas chromatography (GC) can be difficult as the active nature of these compounds causes adsorption and peak tailing. Several years ago Restek introduced the Rtx®-5 Amine column, which uses a unique deactivation chemistry to improve the response and peak symmetry of amines, especially alkyl amines, diamines, triamines, ethanolamines, and ethyleneamines. Now Restek is introducing the new Rtx®-35 Amine column, which employs the same deactivation technology as the Rtx®-5 Amine, but features a higher polarity that is ideal for separating more polar amines and lower molecular weight amines.

Enhanced Peak Symmetry for Amines

The proprietary deactivation of the Rtx®-35 Amine results in improved response and better peak symmetry compared to other 35% phenyl columns (Figure 1). A test mixture with a wide variety of amines at concentrations of 10 to 15ng/µL was analyzed using an Rtx®-35 Amine column and a standard 35% phenyl column. While the Rtx®-5 Amine column shows complete adsorption of diethylenetriamine and diethanolamine, the Rtx®-35 Amine offers excellent response and peak shape for these compounds.

Figure 1

The Rtx®-35 Amine column prevents adsorption and improves response for amines.



1. pyridine
2. 1,2-butanediol
3. C10
4. 2-nonanol
5. diethylenetriamine
6. C12
7. diethanolamine
8. 2,6-dimethylaniline

30m, 0.53mm ID, 1.0µm Rtx®-35Amine (cat.# 11355)
30m, 0.53mm ID, 1.0µm Rtx®-35 (cat.# 10455)
Sample: amine test mix (cat.# 35002); **Conc.:** 450-900ppm; **Solvent:** methanol/methylene chloride; **Sample size:** 1.0µL; **Inj. temp.:** 250°C; **Inj. mode:** split (10:1); **Inlet liner:** base-deactivated 4mm Single Gooseneck (cat.# 20798-210.1); **Carrier gas:** helium; **Linear velocity:** 30cm/sec.; **Detector:** FID/300°C; **Temp. program:** 110°C (hold 4.0 min.) to 200°C @ 8°C/min. (hold 5.0 min.)

GC_CH00577
GC_EX00579

in this issue

RESTEK
800-356-1688 ★ 814-353-1300
www.restekcorp.com

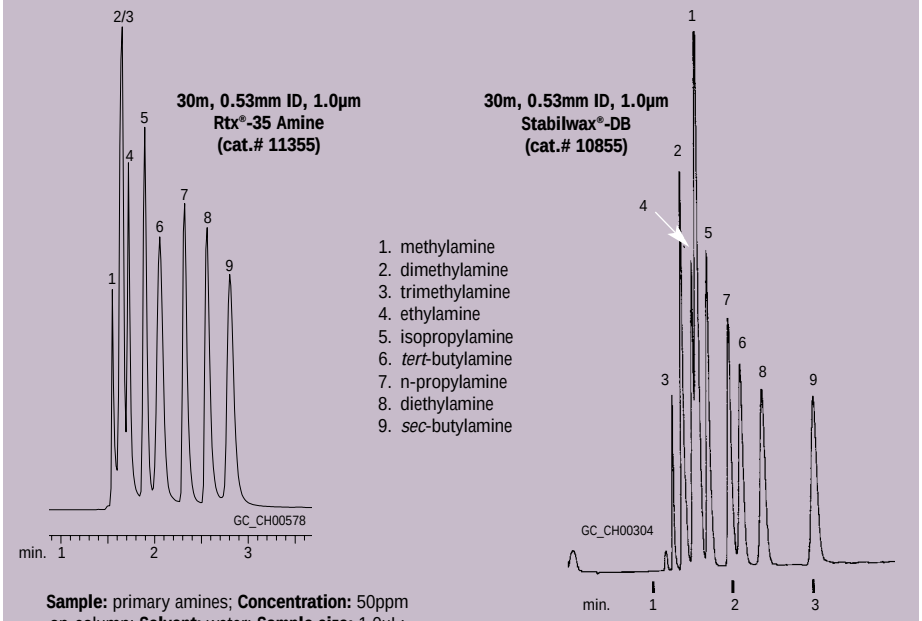
Winter '02
INTERNATIONAL

Improved Resolution of Primary Amines
Primary amines normally are analyzed on a Stabilwax®-DB column because it has a special deactivation that helps to easily resolve these compounds. However the maximum operating temperature of this column is 220°C, which limits the molecular weight range of the amines that can be analyzed.The Rtx®-5 Amine column is at the other end of the spectrum in terms of polarity and selectivity, and does not adequately resolve the primary amines even though it has excellent stability and a higher maximum operating temperature (315°C).

The Rtx®-35 Amine column was designed to provide a range of polarity and selectivity that falls between the Stabilwax®-DB and Rtx®-5 Amine columns. The benefits of the Rtx®-35 Amine column include excellent thermal stability and good resolution for primary amines as shown in Figure 2.

Because the Rtx®-35 Amine column offers excellent response and peak symmetry for more polar amines and has high thermal stability, laboratories analyzing amines and other basic compounds can benefit from better results.

Figure 2
The Rtx®-35 Amine column offers good resolution of primary amines compared to the more polar Stabilwax®-DB column.



Sample: primary amines; **Concentration:** 50ppm on-column; **Solvent:** water; **Sample size:** 1.0µL; **Inj. temp.:** 250°C; **Injection mode:** split (10:1); **Inlet liner:** base-deactivated 4mm Single Gooseneck (cat.# 20798-210.1); **Carrier gas:** helium; **Linear velocity:** 35.7cm/sec.; **Detector:** FID @ 300°C; **Oven temp.:** 35°C (hold 5.0 min.)

1.0µL direct injection of amines in water; **Oven temp.:** 45°C isothermal; **Inj. & det. temp.:** 250°C; **Carrier gas:** hydrogen; **Linear velocity:** 40cm/sec. (flow rate: 5cc/min.); **FID sensitivity:** 1 x 10^{-11} AFS

Rtx®-35 Amine (fused silica) Stable to 340°C

ID (mm)	df(µm)	temp. limits	15-Meter	30-Meter
0.25	0.50	0 to 290/310°C	11335	11338
	1.00	0 to 280/300°C	11350	11353
0.32	1.00	0 to 280/300°C	11351	11354
	1.50	0 to 270/290°C	11366	11369
0.53	1.00	0 to 260/280°C	11352	11355
	3.00	0 to 240/260°C	11382	11385

Base-Deactivated Inlet Liners for Agilent GCs

Description	ea.	5-pk.	25-pk.
4mm Split Straight w/Wool	20781-211.1	20782-211.5	20783-211.25
Cyclosplitter®	20706-210.1	20707-210.5	—
4mm Splitless Straight	20772-210.1	20773-210.5	20774-210.25
2mm Gooseneck	20795-210.1	20796-210.5	20797-210.25
4mm Gooseneck	20798-210.1	20799-210.5	20800-210.25

If you do not see the liner you need, orders can be placed on a custom basis with the appropriate suffix number added. For base deactivation, each (-210.1), 5-pack (-210-5), 25-pack (-210.25). For base-deactivated liners with base-deactivated wool: each (-211.1), 5-pack (-211.5), 25-pack (-211.25). For a complete list of inlet liners, refer to the annual *Chromatography Products Guide* (lit. cat.# 59662).

Base-Deactivated Guard Columns

For analyzing basic compounds, use base-deactivated guard columns. Order cat. #s 10000 , 10001 , 10002 (0.25, 0.32, and 0.53mm ID respectively) for 5m **base-deativated guard columns.** More information on guard columns can be found on page 7. For detailed pricing, refer to the annual *Chromatography Products Guide* (lit. cat.# 59662).

HOT techtip *from the* ***Restek Wizards***

Many GC problems can be avoided by electronically leak checking the system during the plumbing process. Thorough leak checking will prevent loss of GC gases and reduce damage to capillary columns, and will help decrease detector maintenance. Oxygen can move into the system via a leak due to the Venturi effect, and irreversible damage can occur if a column is exposed to oxygen at high temperatures. Also, some detectors are very sensitive to oxygen. Leak checking the instrument before column installation and conditioning prevents column degradation indicated by high bleed and short lifetime. Leak checking should be performed on the entire gas system and GC. Begin by checking all fittings inside the GC. Next check the external fittings along the carrier gas lines, all the way to the gas tanks. Never use liquid leak detectors that contain soap or surfactants because liquids can be drawn inside the fitting at the site of the leak and contaminate the system.

Leak Detective™ Electronic Leak Detector

- Compact, lightweight, hand-held design.
- Contamination-free leak detection.
- Detects helium or hydrogen trace leaks at ≥3 x 10^{-4}cc/sec. or ≥200ppm.
- Audible alarm and LED readout.
- Responds in less than 2 seconds to trace leaks of gases.*
- Operates on two 9-volt batteries or AC adaptor, both included.

(110 VAC): cat.# 21607, (ea.)
(220 VAC): cat.# 21609, (ea.)
European 2-prong plug (220 VAC): cat.# 21382 (ea.)
**Not designed for use in explosive atmospheres.*

New Electronic Leak Detector Coming Soon!
Designed to be more sensitive, smaller, and ergonomic. Watch the Restek Advantage newsletter and www.restekcorp.com for details.

Low-Bleed Septa with Less Coring

InfraRed™ Septa

- ✔ Stable to 325°C for high-temperature analyses.
- ✔ Low bleed prevents baseline disturbance.
- ✔ Excellent puncturability and long life means you can make hundreds of injections on one septum.
- ✔ Does not adhere to injectors so removal is easy.

For a **FREE sample of InfraRed™ septa**, call 800-356-1688 or 814-353-1300, ext. 3, or contact your local Restek representative.

InfraRed™ Septa

Septum Diameter	25-pk.	50-pk.	100-pk.
9mm	21417	21418	21419
9.5mm (3/8")	21421	21422	21423
10mm	21424	21425	21426
11mm (7/16")	21427	21428	21429
11.5mm	21430	21431	21432
12.5mm (1/2")	21433	21434	21435
17mm	21436	21437	21438
Shimadzu Plug	21439	21440	21441

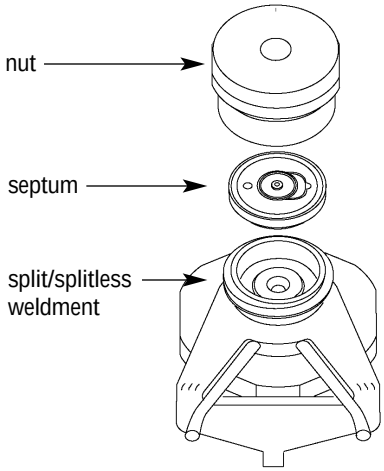
Instrument	Septum Size
Agilent (HP)	
5880A, 5890, 6890, 6850	11mm
5700, 5880	9.5/10mm
On-Column Injection	5mm
CE Instruments (TMQ)	
TRACE GC	17mm
Finnigan (TMQ)	
GC 9001	9.5mm
GCQ	9.5mm
GCQ w/TRACE	17mm
QCQ™	9.5mm
TRACE 2000	9.5mm
Fisons/Carlo Erba (TMQ)	
8000 series	17mm
Gow-Mac	
6890 series	11mm
All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

Measure your old septum here (size in mm)

Septa Alternative Provides Longer Life and Wear Resistance

Merlin Microseal™ Septa

- ✔ For Agilent 5890/6890/6850 GCs compatible with EPC.
- ✔ High-pressure capability allows operation from 2 to 100psi.
- ✔ A top wiper rib improves resistance to particulate contamination and can be taken apart for cleaning.
- ✔ Reduces shedding of septum particles into the injection port liner, eliminating a major source of septum bleed and ghost peaks.
- ✔ Reduces the risk of septum leaks occurring during extended automated runs.



Microseal™ High-Pressure Septa 400 Series	Merlin#	Similar to Agilent#	cat.#
Nut kit (1 nut, fits 300 & 400 series septa)	403	5182-3445	22809
Standard kit (nut, 2 high-pressure septa)	404	Not offered	22810
Starter kit (nut, 1 high-pressure septum)	405	5182-3442	22811
High-pressure replacement septum (1 septum)	410	5182-3444	22812

Bubble Trouble in Your HPLC Mobile Phase?

Degasys In-Line Mobile Phase Degasser

by Greg France, HPLC Product Marketing Manager

- ✔ Prevents out-gassing to improve flow rate stability and reduce baseline noise.
- ✔ Quick equilibration and changeover of mobile phase solvents.
- ✔ Less solvent waste due to low internal volume (400µL).

Dissolved gases in mobile phases can be the source of several common HPLC system problems, such as pump flow rate instability and irreproducible gradients. Out-gassing from the mobile phases can result in air bubbles in the check valves, observed as cavitations or pressure fluctuations in the pump (remember gases are compressible while liquids are not). Air bubbles also may form within the detector flow cell leading to spikes in the resulting chromatogram. Gases that remain dissolved in the mobile phase can have a quenching effect when using fluorescence detectors and can cause increased background noise in UV-visible detectors. This can result in lowered sensitivity. Therefore, in order to optimize system performance, mobile phases should be degassed prior to entering the HPLC pumps.

Some of the traditional degassing techniques include sonication with off-line vacuum degassing or helium sparging. Each has varying degrees of effectiveness, but neither approaches the level of gas removal efficiency achieved through the use of an in-line vacuum degasser.

Sonication in conjunction with off-line vacuum degassing helps reduce the presence of dissolved gases. But there is the possibility that gases can redissolve into the mobile phase over time because, once degassing is complete, the mobile phase is reexposed to the atmosphere. Helium sparging also is useful at removing dissolved gases. One benefit over sonication/vacuum is that you can prevent gases from redissolving by maintaining a helium blanket over the mobile phase after sparging. A drawback to helium sparging is dealing with bulky gas tank cylinders in the lab. You must use chromatographic-grade helium so that the mobile phase is not contaminated, which can be an expensive proposition if used routinely.

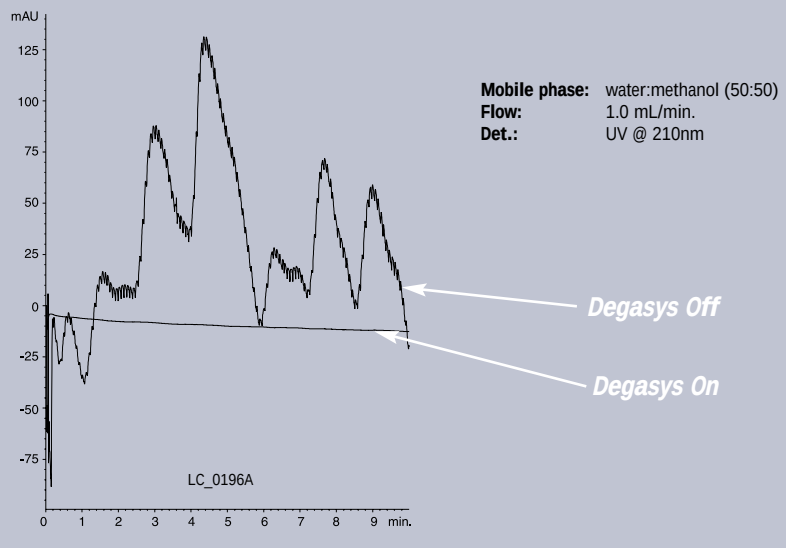
In-line vacuum degassers minimize these problems. Restek now carries an in-line degasser that offers several advantages over other manufacturers—the Degasys in-line mobile phase degasser. These units are more effective at removing dissolved gases than either vacuum/sonication or helium sparging (Figure 1), and because the mobile phase is being degassed just prior to entering the HPLC system there is significantly less opportunity for gases to redissolve in the mobile phase. In addition, the units are easy to install and take up very little bench-top space.

In-line degassers work by passing the mobile phase through a length of tubing made of a semi-permeable material, traditionally this has been PTFE. The tubing is encased in a vacuum chamber and, as the mobile phase passes through, the dissolved gases are pulled through the semi-permeable membrane while the liquid continues to flow through the tube. The Degasys system uses an amorphous fluoropolymer, rather than the traditional PTFE as the transfer tubing. This material is 200 to 300 times more gas permeable than PTFE, which allows a shorter length of tubing in the vacuum chamber and the use of lower internal volumes. Most degassers have an internal volume of 8 to 12mL. The Degasys has an internal volume of only 400µL. The smaller internal volume allows for quicker equilibration and changeover of mobile phase solvents, resulting in less solvent waste. Additionally, each of the four channels of the Degasys system is encased within its own chamber to prevent any type of cross-contamination.

Use the Degasys in-line mobile phase degasser to effectively and conveniently reduce the problems associated with dissolved gases in your mobile phases.

Figure 1

The Degasys in-line mobile phase degasser improves baseline stability.



Degasys In-Line Mobile Phase Degasser

Description	each
110V Mobile phase degasser (4 channel, 7mL/min./channel)	25189
220V Mobile phase degasser (4 channel, 7mL/min./channel)	25194

Degasys unit comes with all nuts and ferrules needed for installation and operation. Order Teflon® tubing separately.

Teflon® Tubing

Description	cat.#	3-meter length
1/8" OD x 0.063" ID	25306	
1/8" OD x 0.094" ID	25307	

for more info on tubing and HPLC Accessories, request the Fast Facts *HPLC Mobile Phase Accessories* (lit. cat.# 59728A).

Restek HPLC Columns

Three Generations of Silica Technology

by Terry Reid, HPLC Chemist

- ✔ Pinnacle II™ silica—columns packed using Restek-manufactured silica.
- ✔ Ultra silica—similar pore size, but greater retention compared to Pinnacle II™ silica.
- ✔ Allure™ silica—largest surface area and greatest retention.

Silica remains the most common support for HPLC columns despite the more recent development of alternative supports such as organic polymers and zirconia. Since the introduction of silica supports for HPLC approximately 30 years ago, the evolution of silica manufacturing technology can be described as having three generations. First generation silica (Type A) is synthesized from inorganic sols. The second generation is base deactivated silica prepared by chemical treatment of Type A silica to remove surface metal impurities. Third generation silica (Type B) is synthesized from an organic sol starting material. Type B silica contains only trace amounts (ppm levels) of metal impurities, giving it attributes similar to those of base-deactivated silica without the chemical treatments needed to remove metal impurities.

The evolution of silica from Type A to Type B was largely driven by the desire for reversed phase silica supports that could analyze basic compounds without mobile phase additives and without excessive peak tailing. Because most HPLC analysts use the reversed phase mode and a large proportion of samples analyzed by reversed phase contain basic analytes (e.g., most pharmaceuticals), base deactivation of silica is an important issue. As a result, many HPLC users prefer columns made from Type B silica. However, many HPLC analysts still want to buy columns made with Type A and/or base-deactivated silica because they are using methods that were developed with these types of columns. Also, for some applications, first or second generation silica can perform just as well as or even better than Type B products. To satisfy all these needs, Restek offers several lines of HPLC columns that cover the three generations of silica.

In 2001 Restek introduced the Pinnacle II™ product line, which is based on new Type A silica developed and manufactured in our Bellefonte facility. The Pinnacle II™ products are very similar to our original Pinnacle™ product line, but are controlled by Restek's in-house silica production. The analysis of phenolic compounds in Echinacea using a Pinnacle II™ C18 column shows excellent performance for neutral and acidic compounds (Figure 1). Research is currently in progress to develop a second-generation product from our Pinnacle II™ silica; one that will allow us to offer an alternative to the well-established Pinnacle™ Amine (base-deactivated) product line.

The Ultra and Allure™ product lines are manufactured from Type B silica, but differ in their pore sizes. Ultra silica is 100Å while Allure™ silica is 60Å. Type B silica typically is higher in surface area than Type A silica. The Ultra silica has a similar pore size to Pinnacle and Pinnacle II™ silica but has a much greater surface area, making it considerably more retentive. Because of its smaller pore size, the Allure™ silica provides even greater surface area and greater retention than the Ultra silica. The Allure™ product line was developed for LC/MS applications where it often is beneficial to have maximum retention. A separation of an antibiotic sample containing two basic compounds using an Allure™ C18 column is shown in Figure 2. Although this is UV detection, the completely volatile mobile phase is compatible with MS detection.

Figure 1

The Pinnacle II™ C18 analysis of Echinacea shows excellent performance for neutral and acidic compounds.

Pinnacle II™ C18 (cat.# 9214565)
Dimensions: 150 x 4.6mm
Particle size: 5µm; Pore Size: 110Å
Conditions:
Mobile phase: A=0.1% phosphoric acid, B=acetonitrile

Time	%A	%B
0	90	10
13	78	22
14	60	40
14.5	60	40
15	90	10
20	90	10

Flow: 1.5 mL/min.
Temp.: 35°C
Det.: UV @ 330nm

Sample: Echinacea capsule
Inj.: 10µL
Conc.: 6.7mg/mL capsule contents in sample diluent
Solvent: ethanol:water (70:30)
Sample temp.: 25°C

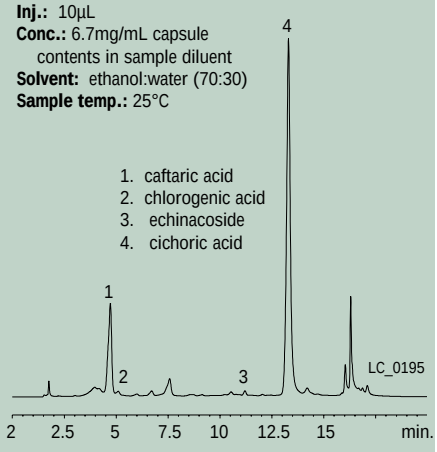


Figure 2

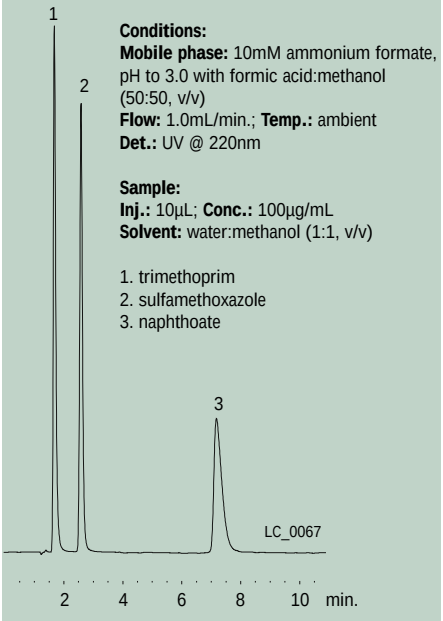
The Allure™ C18 column separates two basic compounds using a mobile phase that is compatible with MS detection.

Allure™ C18 (cat.# 9164565)
Dimensions: 150 x 4.6mm
Particle size: 5µm; Pore size: 60Å

Conditions:
Mobile phase: 10mM ammonium formate, pH to 3.0 with formic acid:methanol (50:50, v/v)
Flow: 1.0mL/min.; **Temp.:** ambient
Det.: UV @ 220nm

Sample:
Inj.: 10µL; **Conc.:** 100µg/mL
Solvent: water:methanol (1:1, v/v)

1. trimethoprim
2. sulfamethoxazole
3. naphthoate



Pinnacle II™ C18 5µm Columns

Length	4.6mm ID
50mm	9214555
100mm	9214515
150mm	9214565
250mm	9214575

Ultra C18 5µm Columns

Length	4.6mm ID
30mm	9174535
50mm	9174555
100mm	9174515
150mm	9174565
200mm	9174525
250mm	9174575

Allure™ C18 5µm Columns

Length	4.6mm ID
30mm	9164535
50mm	9164555
100mm	9164515
150mm	9164565
200mm	9164525
250mm	9164575

for more info

including a complete product listing, request lit. cat.# 59241, *HPLC Columns and Accessories Catalog.*

Rapid Analysis of Marine Oil-Based FAMEs

FAMEWAX™ Capillary GC Column

by Rebecca E. Wittrig, Ph.D., Food, Flavor, and Fragrance Innovations Team Manager

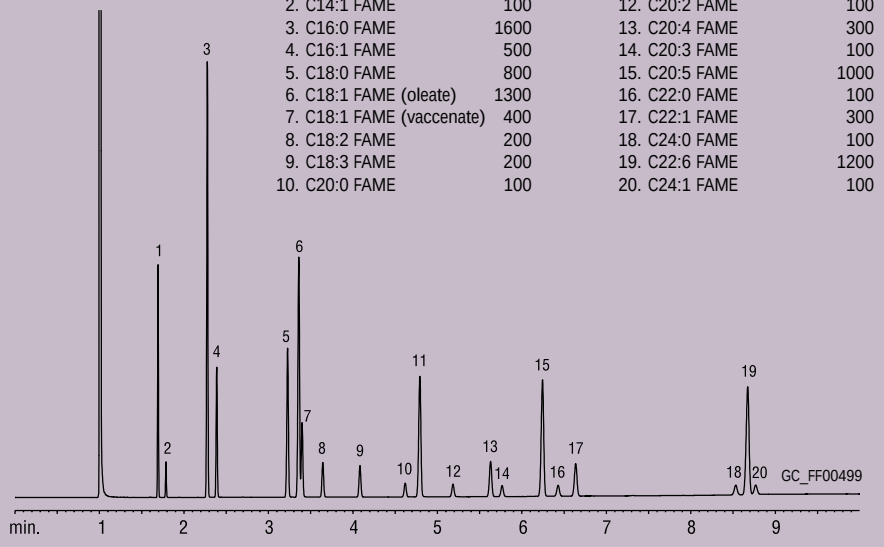
✔ Excellent resolution in less than 10 minutes!
✔ Ideal for all FAME analyses, particularly polyunsaturated FAMEs.

Marine oil products, such as fish oils, are generating a significant amount of interest based on recent medical studies indicating health benefits from consuming these products. Of these oils, the long-chain polyunsaturated fatty acids (PUFAs) such as eicosapentaenoic acid (EPA, C20:5) and docosahexaenoic acid (DHA, C22:6) have been studied extensively due to claims that they reduce the risk of a variety of diseases including hypertension, cardiovascular disease, and autoimmune disorders. These Omega-3 fatty acids are present in high quantities in fish oils. Because the health benefits are thought to be associated with these specific PUFAs, it is necessary to accurately monitor their levels in marine oil raw materials and finished products.

Figure 1

The FAMEWAX™ column shows excellent resolution of marine oil-based FAMEs **in less than 10 minutes!** Note the excellent separation of peaks 18-20.

		conc.		conc.
1.	C14:0 FAME	600µg/mL	11. C20:1 FAME	900
2.	C14:1 FAME	100	12. C20:2 FAME	100
3.	C16:0 FAME	1600	13. C20:4 FAME	300
4.	C16:1 FAME	500	14. C20:3 FAME	100
5.	C18:0 FAME	800	15. C20:5 FAME	1000
6.	C18:1 FAME (oleate)	1300	16. C22:0 FAME	100
7.	C18:1 FAME (vaccenate)	400	17. C22:1 FAME	300
8.	C18:2 FAME	200	18. C24:0 FAME	100
9.	C18:3 FAME	200	19. C22:6 FAME	1200
10.	C20:0 FAME	100	20. C24:1 FAME	100



30m, 0.32mm, 0.25µm FAMEWAX™ (cat.# 12498)
Oven temp.: 195°C to 240°C at 5°C/min. (hold 1 min.); **Inj. & det. temp.:** 250°C (inj.), 275°C (det.); **Carrier gas:** hydrogen; **Linear velocity:** 3mL/min. (constant flow); **Split ratio:** 100:1; **Injection volume:** 1µL; **Std. concentration:** 10,000µg/mL in isooctane (total FAMES), see breakdown in peak list.

✔ **FAMEWAX™** (fused silica) (Crossbond® polyethylene glycol)

Length (m)	ID (mm)	df(µm)	Temp. Limits	cat.#
30	0.25	0.25	20 to 250°C	12497
30	0.32	0.25	20 to 250°C	12498
30	0.53	0.50	20 to 250°C	12499

A typical procedure for analyzing marine oil-based FAMEs involves saponifying the oils and forming the methyl ester derivatives. The FAMEs then can be separated and quantitated by GC using a polar polyethylene glycol (PEG)-type capillary column. In specific, the Omega-3 fatty acids (EPA, C20:5 and DHA, C22:6) need to be completely resolved from all of the other FAME species present. A standard procedure requires a run time of 30-40 minutes to achieve this separation. In comparison, the Restek FAMEWAX™ column shows an excellent separation in less than 10 minutes! Using the FAMEWAX™ capillary GC column will give you much higher throughput for your marine oil-based nutraceutical products and ingredients.

Restek offers several different FAME mixtures for your food and nutraceutical testing needs. Refer to the annual *Chromatography Products Guide* (lit. cat.# 59662) or visit us online at www.restekcorp.com for the complete listing.

✔ **Marine Oil FAME Mix**

Contains:

Chain	Description	% by Weight
C14:0	methyl myristate	6.0
C14:1	methyl myristoleate	1.0
C16:0	methyl palmitate	16.0
C16:1	methyl palmitoleate	5.0
C18:0	methyl stearate	8.0
C18:1	methyl oleate	13.0
C18:1	methyl vaccenate	4.0
C18:2	methyl linoleate	2.0
C18:3	methyl linolenate	2.0
C20:0	methyl arachidate	1.0
C20:1	methyl 11-eicosenoate	9.0
C20:2	methyl 11-14-eicosadienoate	1.0
C20:4	methyl arachidonate	3.0
C20:3	methyl 11-14-17-eicosatrienoate	1.0
C20:5	methyl eicosapentaenoate	10.0
C22:0	methyl behenate	1.0
C22:1	methyl erucate	3.0
C22:6	methyl docosahexaenoate	12.0
C24:0	methyl lingnocerate	1.0
C24:1	methyl nervonate	1.0

Description	cat.#
100mg	35066

Monitor Food Packaging?

Check out our new Applications Note *Monitoring Volatile Compounds in Food Contact Packaging Using Purge and Trap GC/MS and an Rtx®-5MS Capillary Column* (lit. cat.# 59348).

Watch...

for new food, flavor, and fragrance reference materials coming soon.

GC Capillary Guard Column Options

by Christine Vargo, US Sales and Distribution Manager

- ✔ Save money by prolonging the lifetime of your analytical column.
- ✔ Ensure reproducible analyses by keeping nonvolatile residue from collecting at the front of the analytical column.
- ✔ May improve sample focusing and separation efficiency.

Selection Tips

Several types of guard columns are available. The choice of guard column should be made depending upon the compatibility of the guard column type with the compounds of interest, or if the tubing is being used as a transfer line to carry the sample from an inlet device to the column or from the column outlet to the detector. Choosing a guard column that is compatible with your compounds of interest ensures focused sample bands, and good peak shape with minimal peak tailing.

Integra-Guard™ Guard Columns

- Continuous length of tubing containing both the guard column and the analytical column—no connector required.
- Available in many phases.
- Guaranteed leak-free.

Specialized Deactivations

Siltek™ Guard Columns

Revolutionary deactivation lowers endrin breakdown to less than 1%.
Inertness retained over a wide range of sample pH.

- Minimal bleed.
- Ideal for chlorinated pesticide analysis.
- Individually tested for chlorinated pesticide breakdown.

Recommended for difficult matrix and reactive compound analysis
Ideal for use as transfer lines.

- Recommended for use with Rtx®-CLPesticides, Stx®-CLPesticides, Stx®-1HT, and Rtx®-TNT columns.

Base-Deactivated Guard Columns

- Provides excellent inertness for the analysis of basic compounds.
- Tested with basic amine test mix (chromatogram included).
- Recommended for use with Rtx®-5 Amine, Rtx®-35 Amine, and Stabilwax®-DB columns

General-Purpose Deactivations

In most cases, the standard IP tubing should be chosen. The IP surface contains methyl, as well as phenyl groups, making this surface compatible with most common solvents.

Intermediate Polarity (IP) Tubing

- The most universal guard column tubing material.
- Phenylmethyl-deactivated surface provides optimum compatibility for both polar and non-polar compounds.

Polar-Deactivated Tubing

- Provides optimum wettability for polar compounds.
- Minimizes peak splitting when using polar solvents such as methanol and water.
- Uses a polyethylene glycol deactivation layer.
- Compatible with Stabilwax®, Rtx®-225, and Rtx®-2330 capillary columns.

If methanol or water is the primary solvent, then polar surfaces should be used such as our polar-deactivated tubing. The polar-deactivated surface is not resistant to harsh water vaporization, which occurs when water in the liquid state is injected onto the tubing surface and rapidly vaporized.

Hydroguard™ Tubing

- Provides excellent inertness for water-based samples.
- Reduces effects of dirty samples on column performance.
- Reduces downtime and maintenance.

Hydroguard™ tubing is preferred for situations where there is harsh water vaporization. By using a unique deactivation chemistry, the resulting high-density surface is not readily attacked after an aggressive hydrolysis treatment. The high-density surface coverage effectively prevents water vapor from reaching the fused silica surface beneath the Hydroguard™ deactivation layer.

Why use a guard column?

Capillary gas chromatography (GC) guard columns protect analytical columns by trapping nonvolatile residues, preventing them from collecting at the front of the analytical column. These nonvolatile residues may be very high molecular weight organic compounds, inorganic salts, or particulates. If these contaminants enter the analytical column, they can cause adsorption of active compounds, retention time drift, loss of resolution, and poor peak symmetry. When this contamination begins to affect sample analysis, a small section of the analytical column must be removed to restore proper performance. Each time a section of the analytical column is removed, retention times change, and some resolution is lost. By using a guard column and removing contaminated loops from it instead of the analytical column, the separation power of the analytical column remains intact.

When should a guard column be replaced?

The guard column should be replaced as it becomes contaminated with nonvolatile residue. At this point, the performance of the entire chromatographic system will begin to deteriorate. This normally is exhibited as a drastic decrease in the response of active compounds and peak tailing.

What is the life expectancy of a guard column?

The life expectancy of a guard column depends on its length, the amount of nonvolatile residue in the samples, and the nature of samples injected on the column. When analyzing dirty samples, the guard column becomes contaminated quickly. Normally, contamination deposits in the first meter of the guard column. If a short guard column (1m) is used, it must be completely replaced when it becomes contaminated. If a longer guard column (5m) is used, the contaminated sections can be removed without having to reconnect it to the analytical column.

What length guard column do I need?

A guard column should be long enough to keep non-volatile residue from entering the column, but short enough so that the analysis time is not dramatically increased. Five-meter guard columns are more cost effective, reduce the frustrations of making constant connections between shorter guard columns and the analytical column. Ten-meter guard columns often are used when analyzing very dirty environmental samples. If a very long guard column (>10 meters) is used, the residence time of sample components increases, resulting in a slightly longer analysis time. Guard columns over 30 meters long can cause peak distortion and a loss in efficiency; they are not recommended. In any case, it is important to adjust the column flow rate to account for the length of the guard column that is used, even though the guard column does not have retention.

Fast, High-Temperature Sim Dist Analysis

MXT®-1HT Sim Dist Capillary GC Column and the GC Racer*

by Neil Mosesman, GC Columns Product Marketing Manager

- ✔ Analysis time reduced by 75%, increases throughput.
- ✔ Meets all criteria of ASTM Method D-6352.
- ✔ Excellent peak shape for high-boiling compounds.

ASTM Method D-6352 is a gas chromatography (GC) method developed for the determination of petroleum distillates with a boiling point range of 174°C to 700°C. Often referred to as "high-temperature simulated distillation," this method requires a capillary column capable of withstanding GC oven temperatures up to 430°C. This presents many challenges for analysts because most capillary columns are manufactured using polyimide-coated fused silica tubing. At temperatures above 380°C, even the best polyimide coating becomes brittle, which leads to very short column lifetimes. In addition, the methyl silicone stationary phase recommended in the method also must survive these high temperatures.

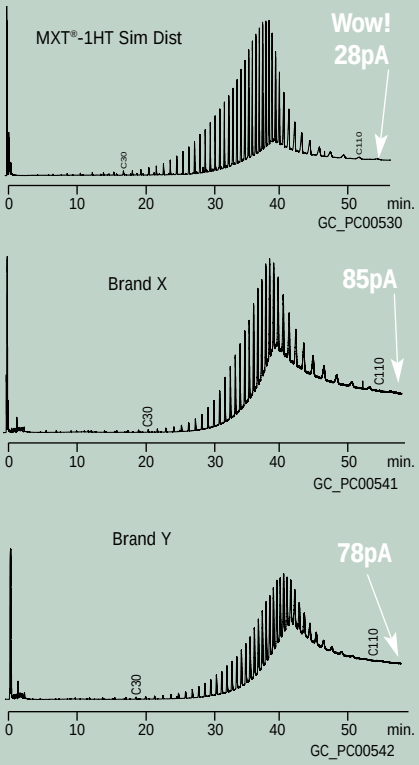
The MXT®-1 HT Sim Dist column is a major improvement in column technology for high-temperature simulated distillation. By combining a new, proprietary polymer synthesis technology, Siltek™ deactivation, and rugged tubing, we developed a capillary column that meets all the criteria of ASTM Method D-6352. This MXT®-1HT Sim Dist column is available as a 5m, 0.53mm ID, 0.1µm film to conform to the requirements of this method. It exceeds the resolution, peak shape, and bleed criteria for hydrocarbons ranging up to C-110. Because the MXT®-1HT Sim Dist column is coated with a 100% dimethyl polysiloxane polymer, it will give the correct retention time/boiling point curve. The MXT®-1HT Sim Dist column exhibits low bleed and excellent inertness, and the rugged tubing will hold up to temperatures in excess of 430°C.

To demonstrate the lower bleed and improved peak shape of this innovative column, a Polywax® 1000 reference material was analyzed using an MXT®-1HT Sim Dist column and two other columns that are commonly used for this application (Figure 1). The MXT®-1HT Sim Dist column exhibits lower bleed and improved peak symmetry compared to other columns on the market.

As part of cost reduction efforts, many laboratories try to reduce individual sample analysis times in the interest of increasing overall throughput. High-temperature simulated distillation analyses can take as long as an hour, especially when samples contain hydrocarbons up to C110. An effective technique to reduce analysis time is to use rapid temperature programming. Unfortunately, most GC systems have temperature-programming limitations of 20°C to 25°C/min. To overcome these limitations, Restek offers the GC Racer, an attachment to your Agilent

Figure 1

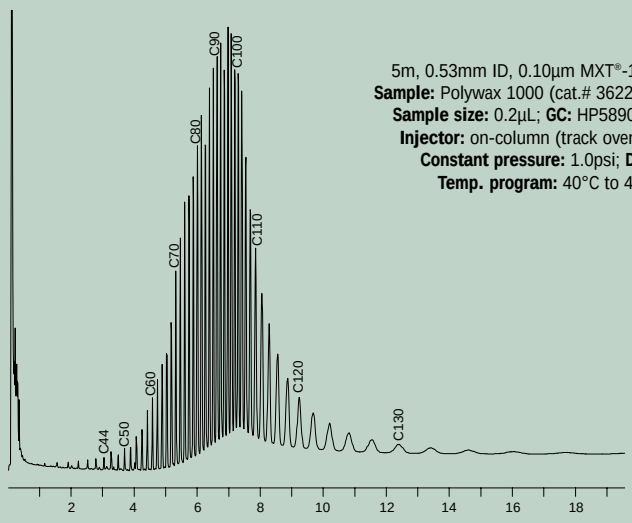
The MXT®-1HT Sim Dist column exhibits lower bleed and improved peak shape compared to other columns used for simulated distillation.



Sample: 0.2µL Polywax® 1000 standard (cat.# 36227); **Solvent:** carbon disulfide; **Injector:** on-column (track oven); **Carier gas:** helium (constant pressure); **Head pressure:** 1.0psi; **Linear velocity:** 60cm/sec.; **Flow rate:** 7.8mL/min.; **Detector:** FID @ 430°C; **Make-up gas flow:** 40cc/min.; **Temp. program:** 40°C to 430°C @ 10°C/min. (hold 30 min.)

Figure 2

Significantly reduce simulated distillation analysis time using the MXT®-1HT Sim Dist column and the GC Racer attachment.



5m, 0.53mm ID, 0.10µm MXT®-1HT Sim Dist (cat.# 70100)
Sample: Polywax 1000 (cat.# 36227); **Solvent:** carbon disulfide;
Sample size: 0.2µL; **GC:** HP5890 GC w/ GC Racer™ system;
Injector: on-column (track oven); **Carrier gas:** hydrogen;
Constant pressure: 1.0psi; **Detector:** FID @ 430°C;
Temp. program: 40°C to 430° C @ 60°C/min.

D6352-98 Polywax® Standards

These high molecular weight hydrocarbon waxes are useful for simulated distillation and other high-temperature GC work.

Description	qty.	cat.#	Ea.
Polywax 500	1 gram	36224	
Polywax 655	1 gram	36225	
Polywax 850	1 gram	36226	
Polywax 1000	1 gram	36227	

5890 GC that increases the rate of temperature programming. Using the GC Racer, the analysis of the Polywax® 1000 reference material can be reduced from over 50 minutes to less than 15 minutes by temperature programming at 60°C/min. (Figure 2)!

The Restek MXT®-1HT Sim Dist column is the ideal choice for high-temperature simulated distillation. It meets all the criteria of ASTM Method D-6352 while providing low bleed and excellent peak shape. Combining the MXT®-1HT Sim Dist column with the GC Racer attachment significantly reduces overall analysis time and greatly increases sample throughput.

MXT®-1HT Sim Dist (metal column)
Temp Limits: -60 to 430°C

Length (m)	ID (mm)	df(µm)	cat.#
5	0.53	0.10	70100

HOT techtip *from the* ***Restek Wizards***

To maintain the low bleed and high performance of the MXT®-1HT Sim Dist column, it is critical to prevent oxygen from entering the column. This can be achieved by routinely checking your entire system for leaks and using a high-quality gas purifier such as the Super-Clean™ SGT gas filter. We also recommend the use of graphite ferrules; Vespel® or Vespel®/graphite ferrules will not withstand the high temperatures required for this analysis.

Super-Clean™ SGT Gas Filters

- High-purity output (99.9999% purity).
- Features a "quick connect" for fast and simple cartridge changes.
- Full glass/metal design with easy-to-read indicators.

Ultra-High Capacity Oxygen Filter:
cat.# 22029, (ea.)

Single-Position Baseplate:
cat.# 22025, (ea.)

for more info
on SGT Super-Clean™ Gas Filters, request lit. cat.# 59280.

Fast GC Temperature Programming

GC Racer System*

new

by Gary Barone, GC Accessories Product Marketing Manager

- ✔ Save time and money by increasing throughput.
- ✔ Makes fast GC possible with any capillary GC column.
- ✔ Easy to operate and install—truly a "plug and play" accessory.

Fast temperature programs are commonly used in gas chromatographic applications to speed up elution of high boiling point compounds and late eluters. The most common gas chromatograph, the Agilent 5890, has a maximum temperature programmable rate of 70°C/min. The factory heating elements in the 5890 only allow for this maximum temperature program rate to be maintained up to a temperature of 100°C. For analysts trying to push temperature ramps as fast as possible, this inhibited program rate leads to longer analysis time and broader peaks. Now, using the GC Racer auxilliary heating unit, temperature program rates of 70°C/min. can be maintained up to 350°C (Figure 1).

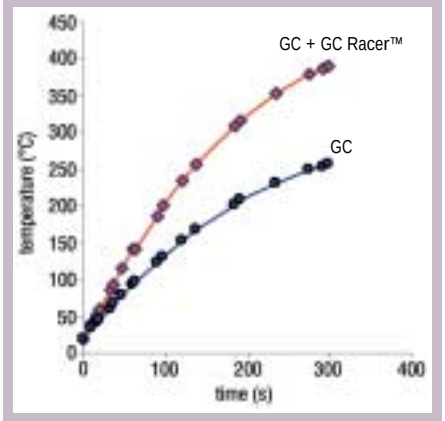
The Restek GC Racer temperature programmer consists of a resistive heating element placed on the floor of the GC oven. The heating element is connected to a controller that is plugged into the main PC board of the GC. When the GC Racer programmer detects that the factory heating elements are not keeping up with the programmed heating rate, the heater is brought into the circuit to augment the heat being supplied to the oven. The GC Racer system will maintain temperature program rates of 70°C/min. up to 350°C and 60°C/min. to temperatures as high as 450°C.

The simplicity of its components and installation makes the GC Racer system a must have add-on accessory for every 5890 GC. The auxilliary heater design is similar to that of the original GC heater. The heater plugs into the GC-Racer controller, which plugs into the main PC board on the GC. The only other connection needed is plugging the GC Racer controller into a 110V standard grounded wall outlet. At no time during the installation of the GC Racer system does the column need to be removed from the oven, or disconnected from the detector or injection port.

The GC Racer system is a new tool in the quest for high-speed GC. The speed of analysis that now can be achieved and the ease of installation will lead to direct savings of time and money by decreasing run time and increasing sample throughput.

Operate your Agilent 5890 as fast as a 6890!

Figure 1
The GC Racer allows temperature program rates of 70°C/min. to be maintained up to 350°C!



GC: Agilent 5890; **Service:** 120V/15 amp; **Start Temp:** 20 °C; Set Oven to 400°C and monitor oven temp.

GC Racer GC Temperature Programmer**

Description	each
For Agilent 5890 Series II (only) GC	23024
For Agilent 5890A (only) GC	23025

*Patent pending
**The GC Racer is currently only available for sale in the US. For availability in your area, contact your local Restek representative.

Improved GC Analysis of Semivolatile Compounds

Using a Siltek™ Drilled Uniliner® Inlet Liner & Rtx®-5Sil MS Column

by Gary Stidsen, Innovations Manager, and David Smith, Ph.D., R&D Senior Chemist

- ✔ Sensitive enough for on-column concentrations down to 4ng.
- ✔ Siltek™ deactivation ensures reproducibility.
- ✔ Fast analysis for US EPA Method 8270.
- ✔ For Agilent GCs.

A new era in inlet liner inertness was realized with the development of Siltek™ passivation. The inert surface of Siltek™ inlet liners has been shown to reduce the loss of basic and acidic compounds.[1,2] To further investigate the benefits of Siltek™ deactivation, semivolatile compounds listed in EPA Method 8270 were analyzed to test for response and linearity.

The chromatographic system used for these liner evaluations included an Agilent 6890 GC with a 5973 MS detector. Restek engineers have designed a unique drilledUniliner® liner that can be used in the split/splitless injection ports of 5890 and 6890 GCs (Figure 1). A small hole drilled into the upper part of the liner results in sample discrimination characteristics of direct injection in combination with the splitless injection technique. This equalizes the pressure between the upstream and split vent pressure sensors and eliminates pressure malfunctions. These liners also reduce injection port discrimination and prevent the injected sample from contacting metal injection port parts. This is accomplished by sealing the column into the press-tight taper in the Uniliner® liner. The compounds are then completely contained in the deactivated liner. Another important characteristic in obtaining optimum inertness is proper inlet liner deactivation. Siltek™ drilled Uniliner® inlet liners were used to analyze the complete Method 8270 list at five on-column concentration levels: 4, 10, 16, 24, and 32ng per component.

Figure 1

The drilled hole makes direct injection possible with EPC systems.

Table I

Active compounds from the Method 8270 list were evaluated for relative response and linearity (4, 10, 16, 24, 32ng on-column) using the Rtx®-5Sil MS with the Agilent 6890 GC/5973 MS.

	ISTD	QIon	4ppm RRF	10ppm RRF	16ppm RRF	24ppm RRF	32ppm RRF	ave RRF	%RSD
N-nitrosodimethylamine	1	74	0.700	0.682	0.684	0.691	0.682	0.686	1%
pyridine	1	79	0.594	0.738	0.701	0.916	0.823	0.711	16%
aniline	1	93	2.197	2.148	2.080	2.051	2.031	2.067	3%
N-nitroso-di-*n*-propylamine	1	70	0.635	0.684	0.617	0.654	0.599	0.609	5%
benzoic acid	2	122	0.291	0.185	0.196	0.221	0.215	0.209	19%
2,4-dichlorophenol	2	162	0.250	0.252	0.248	0.240	0.240	0.241	2%
2,4-dinitrophenol	3	184	0.105	0.152	0.160	0.172	0.163	0.155	17%
3-nitroanaline	3	138	0.322	0.359	0.358	0.363	0.342	0.347	5%
4-nitrophenol	3	109	0.157	0.186	0.190	0.183	0.175	0.177	7%
acenaphthene	3	152	1.200	1.160	1.130	1.030	1.010	1.110	8%
hexachlorocyclopentadiene	3	237	0.263	0.297	0.293	0.288	0.290	0.282	5%
azobenzene	3	77	1.358	1.460	1.396	1.296	1.246	1.296	6%
pentachlorophenol	4	266	0.169	0.189	0.191	0.183	0.172	0.181	5%
nitrosodiphenylamine	4	169	0.781	0.786	0.742	0.705	0.640	0.704	8%
benzidine	5	184	0.418	0.548	0.504	0.623	0.539	0.538	14%
benzo(b)fluoranthene	6	252	1.251	1.381	1.333	1.324	1.295	1.358	4%
benzo(g,h,i)perylene	6	276	1.347	1.472	1.441	1.427	1.372	1.446	4%

A subset of active compounds from the EPA Method 8270 compound list was used to determine the effectiveness of these liners. This list contains the most active compounds, which were evaluated for response factors and linearity over the five different concentrations. The compounds are listed in Table I with response factors and linearity results.

At 4ng on-column concentration per component, the Siltek™ liners show a high response with low standard deviation for these active Method 8270 compounds. Furthermore, the liners show excellent linearity over the calibration curve. Figure 2 illustrates a sample chromatogram of 24ng per component with an analysis time of less than 22 minutes. As this analytical system shows, the inertness of Siltek™ deactivation in combination with the fast analysis time using optimized run conditions has the capability of improving sample output in the laboratory. For more detailed information, request Application Note #59125.

[1]Restek Applications Note #59111 *Minimizing Breakdown of Chlorinated Pesticides Using Siltek™-Deactivated GC Accessories.*
[2]Restek Applications Note #59113 *Siltek™-Deactivation Delivers Inertness to Analyte Breakdown and Reactivity, and Durability to Physical and Chemical Challenges.*

Siltek™—by Restek

Siltek™ Press-Tight® Connectors

5-pk.	25-pk.	100-pk.
	straight	
20480	20449	20481
	angled	
20482	20483	20484
ea.		**3-pk.**
	"Y"	
20485		20486
	Angled "Y"	
20487		20469

Siltek™ Guard Columns

ID	5-Meter (ea.)	10-Meter (ea.)
0.25mm	10026	10036
0.32mm	10027	10037
0.53mm	10028	10038

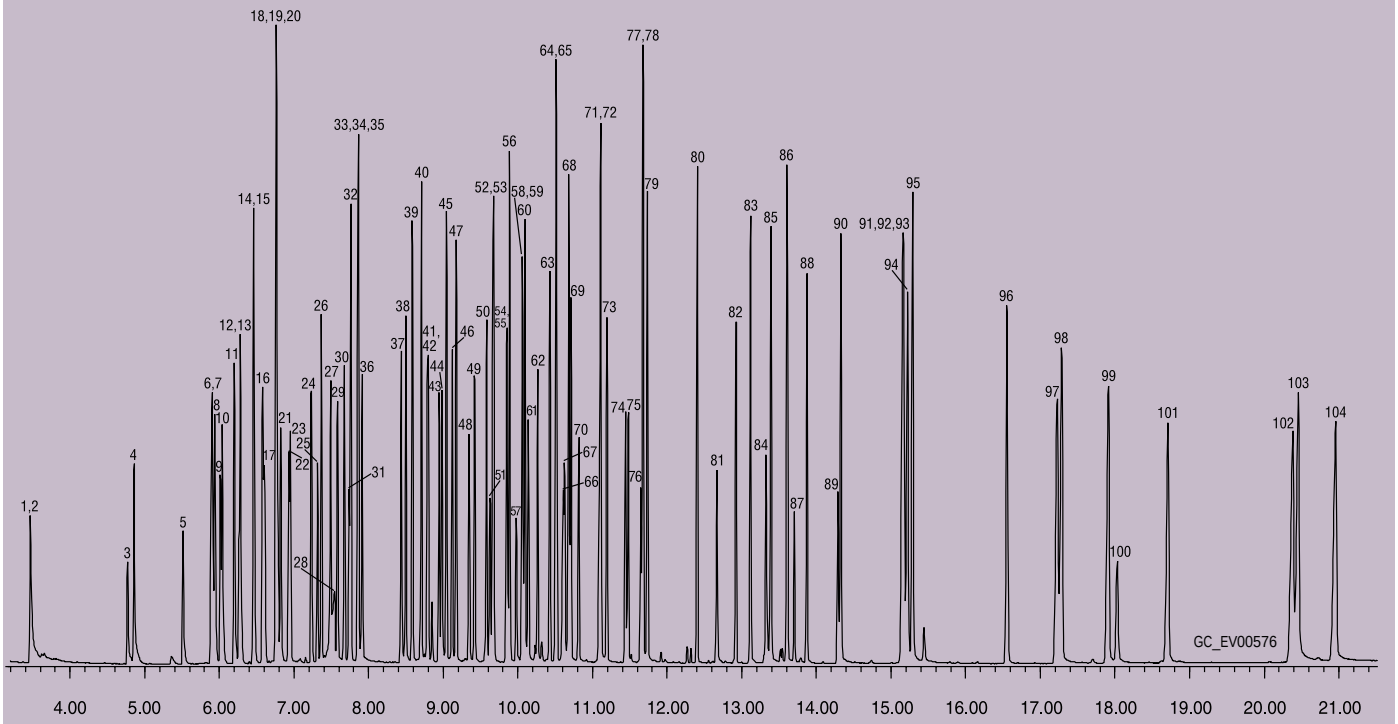
Integra-Guard™ Columns

Guard columns without press-tight connections—protecting your analytical column has never been this easy! Simply add the appropriate suffix number and price to the analytical column's catalog number and price.

ID	length	suffix #
0.25mm	5m	-124
	10m	-127
0.28mm	5m	-243
	10m	-244
0.32mm	5m	-125
	10m	-128

Figure 2

The Rtx®-5Sil MS column, combined with the Siltek™ drilled Uniliner® inlet liner exhibits excellent peak shape and response for semivolatile compounds listed in US EPA Method 8270.



1. N-nitrosodimethylamine
2. pyridine
3. methyl methanesulfonate
4. 2-fluorophenol
5. ethyl methanesulfonate
6. phenol-d6
7. phenol
8. aniline
9. bis(2-chloroethyl)ether
10. 2-chlorophenol
11. 1,3-dichlorobenzene
12. 1,4-dichlorobenzene-d4
13. 1,4-dichlorobenzene
14. 1,2-dichlorobenzene
15. benzyl alcohol
16. 2-methylphenol
17. bis(2-chloroisopropyl)ether
18. acetophenone
19. 4-methylphenol/3-methylphenol
20. N-nitroso-di-{n}-propylamine
21. hexachloroethane
22. nitrobenzene-d5
23. nitrobenzene
24. isophorone
25. 2-nitrophenol
26. 2,4-dimethylphenol
27. bis(2-chloroethoxy)methane
28. benzoic acid
29. 2,4-dichlorophenol
30. 1,2,4-trichlorobenzene
31. naphthalene-d8
32. naphthalene
33. 2,6-dichlorophenol
34. 4-chloroaniline
35. hexachloropropene
36. hexachlorobutadiene
37. 4-chloro-3-methylphenol
38. isosafrole
39. 2-methylnaphthalene
40. 1-methylnaphthalene
41. hexachlorocyclopentadiene
42. 1,2,4,5-tetrachlorobenzene
43. 2,4,6-trichlorophenol
44. 2,4,5-trichlorophenol
45. 2-fluorobiphenyl
46. safrole
47. 2-chloronaphthalene
48. 2-nitroaniline
49. 1,4-naphthoquinone
50. dimethylphthalate
51. 1,3-dinitrobenzene
52. 2,6-dinitrotoluene
53. acenaphthylene
54. acenaphthene-d10
55. 3-nitroaniline
56. acenaphthene
57. 2,4-dinitrophenol
58. pentachlorobenzene
59. 4-nitrophenol
60. dibenzofuran
61. 2,4-dinitrotoluene
62. 2,3,4,6-tetrachlorophenol
63. diethyl phthalate
64. fluorene
65. 4-chlorophenyl phenyl ether
66. 4-nitroaniline
67. 4,6-dinitro-2-methylphenol
68. diphenylamine
69. azobenzene
70. 2,4,6-tribromophenol
71. phenacetin
72. 4-bromophenyl phenyl ether
73. hexachlorobenzene
74. pentachlorophenol
75. pentachloronitrobenzene
76. phenanthrene-d10
77. dinoseb
78. phenanthrene
79. anthracene
80. di-{n}-butylphthalate
81. 4-nitroquinoline-1-oxide
82. isodrin
83. fluoranthene
84. benzidine
85. pyrene
86. {p}-terphenyl-d14
87. aramite
88. chlorbenzilate
89. kepone
90. butyl benzyl phthalate
91. benzo(a)anthracene
92. 3,3{'}-dichlorobenzidine
93. chrysene-d12
94. chrysene
95. bis(2-ethylhexyl)phthalate
96. di-{n}-octyl phthalate
97. benzo(b)fluoranthene
98. benzo(k)fluoranthene
99. benzo(a)pyrene
100. perylene-d12
101. 3-methylcholanthrene
102. indeno(1,2,3-cd)pyrene
103. dibenzo(a,h)anthracene
104. benzo(ghi)perylene

30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)
Conc.: 24µg/mL in methylene chloride
(cat.#s: 31618, 31619, 31620, 31621, 31622, 31206, 31062, 31063) Note: Internal standards at 8ppm

Inj. vol.:	1µL
Inj type:	splitless
Hold time:	0.4 min.
Inlet liner:	drilled Uniliner® liner, Siltek™ deactivation (cat# 21054-214.1)
Inj. temp.:	300°C
Carrier gas:	helium (1mL/min. constant flow)
Linear velocity:	34cm/sec.
Oven temp.:	35°C (2 min.) to 260°C @ 20°C min., to 330° @ 6°C/min. (hold 1 min.)
Det. type:	MS
Transfer line temp.:	280°C
Scan range:	35 to 550amu
Ionization:	EI
Mode:	full scan

Rtx®-5Sil MS (fused silica)

ID (mm)	df(µm)	temp. limits	15-Meter	30-Meter
0.25	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
0.28	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
0.32	0.10	-60 to 330/350°C	12706	12709
	0.25	-60 to 330/350°C	12721	12724
	0.50	-60 to 330/350°C	12736	12739
	1.00	-60 to 325/350°C	12751	12754

Siltek™ Drilled Uniliner® Inlet Liner for Agilent GCs

4.0mm ID, 6.3mm OD, 78.5mm length

each	5-pk.
21054-214.1	21055-214.5

Haloacetic Acid Mixtures

For GC/ECD Analysis of Haloacetic Acids in Water

by Ken Herwehe, Analytical Reference Materials Product Marketing Manager

- ✔ Meet requirements for US EPA Methods 552, 552.1 and 552.2.
- ✔ Prepared from purified neat reference materials for the highest quality.
- ✔ Lot-to-lot consistency ensures analytical reproducibility.
- ✔ Certificate of analysis available or data pack containing statistical QA results for concentration and homogeneity, and a lot sheet with a balance printout of each analyte.

Haloacetic acids and other disinfectant byproducts are formed during the chlorination of drinking water. The US Environmental Protection Agency (EPA) has published *Stage 1 Disinfectants and Disinfection Byproducts Rule* to regulate haloacetic acids at 60ppb on an annual average. This standard became effective December 2001 for large surface water public systems. In December 2003, it will become effective for small surface water and all ground water public water systems.

✔ Haloacetic Acid Mix, 6 Components

bromochloroacetic acid
dibromoacetic acid
dichloroacetic acid
monobromoacetic acid
monochloroacetic acid
trichloroacetic acid

2,000µg/mL each in MTBE, 1mL per ampule

Ea.	5-pk.	10-pk.
31644	31644-510	—
with data pack		
31644-500	31644-520	31744

✔ Haloacetic Acid Methyl Ester Mix, 6 Components

methyl bromochloroacetate
methyl dibromoacetate
methyl dichloroacetate
methyl monobromoacetate
methyl monochloroacetate
methyl trichloroacetate

1,000µg/mL each in MTBE, 1mL per ampule

Ea.	5-pk.	10-pk.
31645	31645-510	—
with data pack		
31645-500	31645-520	31745

✔ Haloacetic Acid Mix, 9 Components

bromochloroacetic acid	400µg/mL
dibromoacetic acid	200
dichloroacetic acid	600
monobromoacetic acid	400
monochloroacetic acid	600
trichloroacetic acid	200
bromodichloroacetic acid	400
chlorodibromoacetic acid	1000
tribromoacetic acid	2000

In MTBE, 1mL per ampule

Ea.	5-pk.	10-pk.
31646	31646-510	—
with data pack		
31646-500	31646-520	31746

✔ Haloacetic Acid Methyl Ester Mix, 9 Components

methyl bromochloroacetate	400µg/mL
methyl dibromoacetate	200
methyl dichloroacetate	600
methyl monobromoacetate	400
methyl monochloroacetate	600
methyl trichloroacetate	200
methyl bromodichloroacetate	400
methyl chlorodibromoacetate	1000
methyl tribromoacetate	2000

In MTBE, 1mL per ampule

Ea.	5-pk.	10-pk.
31647	31647-510	—
with data pack		
31647-500	31647-520	31747

✔ Internal and Surrogate Standards

Description	compound	µg/mL in MTBE	per ampul	qty.	cat.#
Internal Standard	1,2,3-trichloropropane	1000	1mL	ea.	31648
Surrogate Standard	3,5-dichlorobenzoic acid methyl ester	1000	1mL	ea.	31649
Surrogate Standard	2,3-dichloropropionic acid	1000	1mL	ea.	31650
Surrogate Standard	2,3-dichloropropionic acid methy ester	1000	1mL	ea.	31651
Surrogate Standard	3,5-dichlorobenzoic acid	1000	1mL	ea.	31652
Surrogate Standard	2-bromoproionic acid	1000	1mL	ea.	31653
Surrogate Standard	methyl 2-bromopropionate	1000	1mL	ea.	31654
Surrogate Standard	2,3-dibromoproionic acid	1000	1mL	ea.	31655
Surrogate Standard	methyl 2,3-dibromopropionate	1000	1mL	ea.	31656

50 State UST Methods

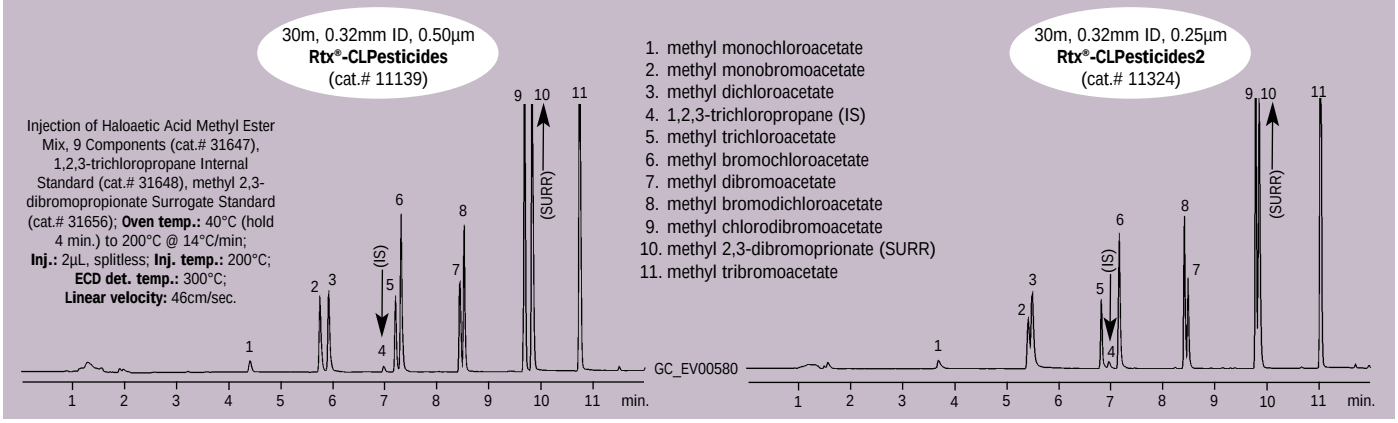
Latest Revisions for All 50 States Available Soon!

- ✔ Detailed product listing available for all 50 states in convenient *Fast Facts* format.
- ✔ Completely updated with the latest method revisions.
- ✔ Allows easy ordering and method setup.
- ✔ Convenient listing of analytical column, sample preparation, reference material, and other consumables needed for all methods.

Fast Facts available at PittCon® 2002—UST method product listings for California, Florida, Massachusetts, Texas, Washington, and Wisconsin. Call Technical Service at 800-356-1688 or 814-353-1300, ext. 4, for more information, or contact your local Restek representative.

Figure 1

The Rtx®-CLPesticides and Rtx®-CLPesticides2 columns resolve haloacetic acid methyl esters in one 12-minute, dual-column run.



Water Quality Testing Standards & Columns

ISO/DIS 9377-4 & H53 Water Quality Testing for Total Petroleum Hydrocarbons (TPH)

by Ken Herwehe, Analytical Reference Materials Product Marketing Manager

✔ Updated reference materials for GC analysis of TPH in water.
✔ Determination of hydrocarbon oil index—applicable to drinking, surface, waste, and treated water.

ISO/DIS 9377-4 describes a gas chromatography/flame ionization detection (GC/FID) method to analyze total petroleum hydrocarbons (TPHs) in drinking, surface, waste, and treated waste water. Previous methods used Freon® extraction, which was harmful to the environment. This new method uses less harmful solvents such as pentane, hexane, or cyclohexane for sample extraction.

Restek now offers mixtures for ISO/DIS 9377-4 analyses. Florisil® cleanup to remove polar compounds is accomplished using a 150-250µm (60/100 mesh) sample preparation column. The analytical column suggested is either an Rtx®-1 or an Rtx®-5 column with dimensions of 10-25m, 0.25-0.53mm ID, and 0.25-1.0µm film thickness. See highlighted columns in product listing for recommended dimensions.

✔ Standard Mixture Stock Solution

diesel #2 (additive free)
motor oil (additive free bp 325–460 or C18–C32 retention time range)
5,000 µg/mL each in cyclohexane, 1mL per ampule (prepares 8mL of 1.25µg/µL calibration curve high point)
Total hydrocarbon concentration 10,000 µg/mL

Ea.	5-pk.	10-pk.
31640	31640-510	—
with data pack		
31640-500	31640-520	31740

✔ Quality Control Standard Mixture

diesel #2 (additive free)
motor oil (additive free bp 325–460 or C18–C32 retention time range)
500µg/mL each in acetone, 1mL per ampule (enough to spike one 900mL quality control sample)
Total hydrocarbon concentration 1,000µg/mL

Ea.	5-pk.	10-pk.
31641	31641-510	—
with data pack		
31641-500	31641-520	31741

✔ Florisil® Cartridge Quality Control Standard Mixture

diesel #2 (additive free)
motor oil (additive free bp 325–460 or C18–C32 retention time range)
1,000µg/mL each in cyclohexane, 10mL per ampule (enough to check one 2g Florisil®/2g sodium sulfate cartridge)
Total hydrocarbon concentration 2,000µg/mL

Ea.	5-pk.	10-pk.
31642	31642-510	—
with data pack		
31642-500	31642-520	31742

✔ Standard Mixture of *n*-alkanes for System Performance Test

n-decane	*n*-hexacosane
n-dodecane	*n*-octacosane
n-tetradecane	*n*-triacontane
n-hexadecane	*n*-dotriacontane
n-octadecane	*n*-tetratriacontane
n-eicosane	*n*-hexatriacontane
n-docosane	*n*-octatriacontane
n-tetracosane	*n*-tetracontane

50µg/mL each in cyclohexane, 1mL per ampule

Ea.	5-pk.	10-pk.
31633	31633-510	—
with data pack		
31633-500	31633-520	31733

✔ Extraction Solvent Stock Solution #1

n-decane	20µL/L
n-tetracontane	20mg/L

in n-hexane, 5mL per ampule (makes 50mL of extraction solvent, enough for 1 sample)

Ea.	5-pk.	10-pk.
31634	31634-510	—
with data pack		
31634-500	31634-520	31734

✔ Extraction Solvent Stock Solution #2

n-decane	20µL/L
n-tetracontane	20mg/L

in n-hexane, 20mL per ampule (makes 200mL of extraction solvent, enough for 4 samples)

Ea.	5-pk.	10-pk.
31635	31635-510	—
with data pack		
31635-500	31635-520	31735

✔ Stearyl Stearate Test Solution

stearyl stearate
2,000 µg/mL in cyclohexane, 10mL per ampule, (enough to check one 2g Florisil®/2g sodium sulfate cartridge)

Ea.	5-pk.	10-pk.
31636	31636-510	—
with data pack		
31636-500	31636-520	31736

Columns

Check the annual Chromatography Products Guide for temp. limits. Highlight indicates a recommended dimension for this analysis.

✔ Rtx®-1 (fused silica)

Crossbond® 100% dimethyl polysiloxane

ID	df (µm)	temp. limits	15-Meter
0.25mm	0.25	-60 to 330/350°C	10120
0.25mm	0.50	-60 to 330/350°C	10135
0.32mm	0.25	-60 to 330/350°C	10121
0.32mm	0.50	-60 to 330/350°C	10136
0.53mm	0.25	-60 to 320/340°C	10122
0.53mm	0.50	-60 to 310/330°C	10137
0.53mm	1.00	-60 to 310/330°C	10152

✔ MXT®-1 (Silcosteel®)

Crossbond® 100% dimethyl polysiloxane

ID	df (µm)	temp. limits	15-Meter
0.25mm	0.25	-60 to 360°C	70120
0.25mm	0.50	-60 to 350°C	70135
0.28mm	0.25	-60 to 360°C	70121
0.28mm	0.50	-60 to 330°C	70136
0.53mm	0.25	-60 to 360°C	70122
0.53mm	0.50	-60 to 330°C	70137
0.53mm	1.00	-60 to 320°C	70152

✔ Rtx®-5 (fused silica)

Crossbond® 5% diphenyl/95% dimethyl polysiloxane

ID	df (µm)	temp. limits	15-Meter
0.25mm	0.25	-60 to 330/350°C	10220
0.25mm	0.50	-60 to 330/350°C	10235
0.32mm	0.25	-60 to 330/350°C	10221
0.32mm	0.50	-60 to 330/350°C	10236
0.53mm	0.25	-60 to 320/340°C	10222
0.53mm	0.50	-60 to 310/330°C	10237
0.53mm	1.00	-60 to 310/330°C	10252

✔ MXT®-5 (Silcosteel®)

Crossbond® 5% diphenyl/95% dimethyl polysiloxane

ID	df (µm)	temp. limits	15-Meter
0.25mm	0.25	-60 to 360°C	70220
0.25mm	0.50	-60 to 350°C	70235
0.28mm	0.25	-60 to 360°C	70221
0.28mm	0.50	-60 to 330°C	70236
0.53mm	0.25	-60 to 360°C	70222
0.53mm	0.50	-60 to 330°C	70237
0.53mm	1.00	-60 to 325°C	70252

Fast, Accurate Analysis of Petrochemicals in Polymers and Plastics

Using an Rtx®-5 Column & EZ Flash® GC

by Ellen Veenstra, Applications Chemist, Thermo Orion and
Christine Vargo, US Sales & Distribution Manager

- ✔ Reduce analysis time by 75%!
- ✔ Ideal for plastics or petrochemicals in packaging testing.

A wide variety of petrochemical materials are used in the synthesis and formulation of polymers and plastics. Low molecular weight monomeric compounds react with an "external" agent, such as a catalyst, UV light, or IR radiation. The reaction creates a high molecular weight polymeric compound by combining the monomers into long or branched chains. If some of the monomer is left unreacted, the small fragments cause physical and sensorial changes in the final plastic. Cracks in structural plastic can form from the stresses due to incomplete polymerization. Discoloration can occur if the reactive monomer interacts with other materials or additives in the final product. Out-gassing can cause an off-odor or "plastic" taste in packaging materials used in foods or beverages.

To ensure product consistency from batch to batch, laboratories must analyze petrochemicals in their finished product. These volatile compounds typically are analyzed by capillary gas chromatography (GC) using a 30-meter column. Analysis times often must exceed 30 minutes to achieve sufficient resolution of these volatile compounds. However, by using a high efficiency, direct thermal transfer of these compounds with an EZ-Flash® GC attachment from Thermo Orion, analysis times can be reduced greatly while still maintaining excellent separation. For example, the typical analysis of plastics in a paint sample on a 30m Rtx®-5 column takes almost 42 minutes. The same analysis using a 10m Rtx®-5 column and an EZ-Flash® system is accomplished in less than 5 minutes (Figure 1).

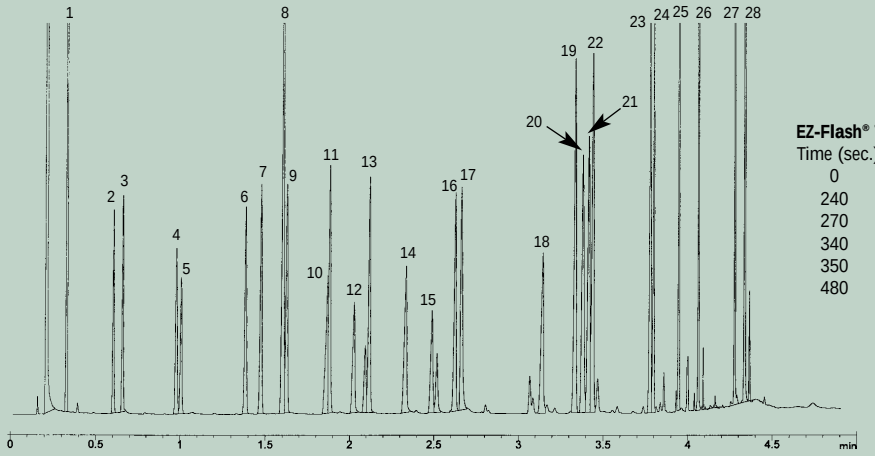
Every minute counts when improving laboratory throughput and efficiency. The EZ-Flash® system uses resistive heating techniques and fast temperature programming to achieve increased productivity. The EZ-Flash® system is compatible with 0.53mm ID, 0.32mm ID, and smaller ID (0.2 -0.10mm) columns.

For more information on the EZ Flash® system, contact Thermo Orion at 1-888-EZFLASH or **www.ezflash.com**

Figure 1

The analysis time of an industrial paint sample is reduced by 75% using the EZ-Flash® system with a 10m, 0.25mm ID, 1.0µm Rtx®-5 column.

1. vinyl acetate
2. ethyl acrylate
3. methyl methacrylate
4. *t*-butyl acrylate
5. ethyl methyl acrylate
6. isobutyl acrylate
7. allyl methacrylate
8. styrene
9. butyl acrylate
10. hydroxyethyl acrylate
11. isobutyl methacrylate
12. hydroxypropyl acrylate
13. butyl methacrylate
14. hydroxyethyl methacrylate
15. hydroxypropyl methacrylate
16. glycidyl methacrylate
17. dimethylaminoethyl methacrylate
18. Hydroxybutyl acrylate
19. cyclohexyl methacrylate
20. *t*-butylaminoethyl methacrylate
21. diethylaminoethyl methacrylate
22. 2-ethylhexyl acrylate
23. 2-ethylhexyl methacrylate
24. ethyleneglycol dimethacrylate
25. iso-bornyl acrylate
26. iso-bornyl methacrylate
27. lauryl methacrylate
28. α-methylstyrene dimer



EZ-Flash® Temperature Profile

Time (sec.)	Temp (ºC)
0	40
240	140
270	140
340	190
350	280
480	280

10m, 0.25mm ID, 1.0µm Rtx®-5 (cat.# 10250-107)
Inj. temp.: 250°C; **Inj. type:** split; **Inj. volume:** 1µL; **Split ratio:** 100:1; **Split flow:** 100mL/min.; **Carrier gas:** helium; **Mode:** ramp pressure: 4.74psi to 7.07psi @ 0.58psi/min. (hold 0.5 min.) to 8.29psi @ 1.05psi/min. to 10.82psi @ 13.38psi.min.; **Nominal initial flow:** 1mL/min.; **Det. type:** FID @290°C; **Oven program:** 40°C (hold 0.5 min.) to 150°C @20°C/min. (hold 2 min.)
Chromatogram courtesy of Thermo Orion, Beverly, MA, www.thermoorion.com.

cool tools

Try Restek's SILCOSTEEL Injection Ports

For more information, request the catalog *Genuine Restek Replacement Parts for Agilent GCs* (lit. cat.# 59627B).

Try These New Tools from Restek for Easier GC Maintenance

by Brad Rightnour and Michael Goss, Instrument Innovations Team

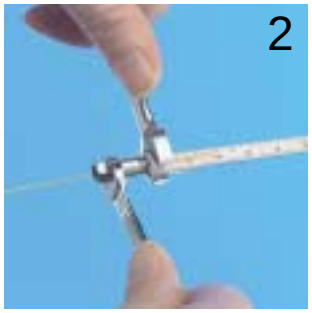
Capillary Installation Gauge

Easily pre-seat ferrules for consistent installations!

✔ Pre-seats ferrule onto column for consistent installation distances.
✔ Made from high-quality stainless steel.

For Agilent-style fittings (0-100mm from front of ferrule)**:** cat.# 21034, (ea.)
For 1/16" fittings (15-115mm from back of nut): cat.# 21399, (ea.)

Install the column nut and ferrule onto the capillary column. Cut the column end squarely to prevent ferrule particles from entering the column. Slide the column into the installation gauge to the recommended insertion distance as specified by the instrument manufacturer. Finger-tighten the column nut at the correct distance.



Using a 5/16" wrench on the installation gauge nut and a 1/4" wrench on the column nut, tighten the assembly with moderate force to ensure a properly seated ferrule.

With the same wrenches, loosen the assembly and remove the column and column nut with seated ferrule from the installation gauge. The ferrule should be properly seated in the column nut, and the column should remain in place when light force is applied. If it slides loosely in the ferrule, repeat steps 1 and 2.

Inlet Liner Packing Tool

Easy and reproducible!

✔ Position wool correctly every time.
✔ Accurate to a specific, measured depth (0-100mm).

cat.# 20339, (ea.)

Loosen the nut on the side of the tool to adjust the gauge to the manufacturer's recommended depth.

Place a one-centimeter plug of loosely bound wool at the top of your inlet liner. Be sure to wear gloves when handling glass wool.

Insert the liner packing tool completely into the liner until the tool bottoms out. Remove the tool. The wool is now positioned correctly in the liner and ready for use.

Rethreading Tool

Achieve a better seal!

✔ Save the cost of replacing expensive injectors!
✔ Repair worn or damaged threads.

For 1/16" compression fittings (thread size, 10-32): cat.# 23016, (ea.)
For 1/8" compression fittings (thread size, 5/16-20): cat.# 23017, (ea.)
For 1/4" compression fittings and Agilent-style split/splitless injection ports (thread size, 7/16-20): cat.# 23018, (ea.)
For Varian injection ports: cat.# 23019, (ea.)

Due to constant installation, removal, and exposure to extreme temperature changes, threads on GC parts easily become worn and damaged. This can cause a poor seal, and oxygen can enter the system, compromising analytical results and possibly destroying expensive analytical columns.

Screw the rethreading tool completely onto the injection port in a clockwise direction. Depending on the severity of thread damage, this may require force.

Unscrew the rethreading tool and inspect the threads. Repeat as necessary. When done, wipe clean with methanol to remove any debris.

RESTEK

Behind the Scenes

Thank You!

Thanks for donating your Wizard Dollars toward disaster relief efforts. With your contributions, Restek is donating $3,268 to the Red Cross, the Salvation Army, and the United Way. On behalf of those that will benefit from your generosity, thank you for supporting the families affected by the Sept. 11 tragedies.

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Please direct your comments on this publication to Kristin Cox, Editor, at kristind@restekcorp.com or call Restek, ext. 2313.

Coming to a location near you!

Restek-on-the-Road Seminar Tour 2002

Coming to a location near you! We offer four seminars: Environmental Analyses (ENV), Food, Flavor, and Fragrance Analyses (FFF), Comprehensive HPLC (HPLC), and Comprehensive GC (GC). Each is an in-depth, one-day course in an engaging multimedia format taught by real-world chromatographers. For more information and the most current dates and locations, visit **www.restekcorp.com**

Date	Sem.	State	City
7/15	ENV	AZ	Phoenix
5/6	ENV	CA	Walnut Creek
7/18	ENV	CA	Buena Park
5/20	ENV	FL	Orlando
5/22	ENV	GA	Atlanta
10/16	ENV	IL	Schaumburg
9/12	ENV	MA	Braintree
10/21	ENV	MD	Baltimore
10/14	ENV	MN	Minneapolis
5/23	ENV	NC	Raleigh
10/25	ENV	NY	Tarrytown
11/11	ENV	OK	Oklahoma City
10/23	ENV	PA	King of Prussia
11/13	ENV	TX	Austin
11/14	ENV	TX	Houston
5/9	ENV	WA	Seattle
6/14	FFF	CA	Buena Park
8/26	FFF	CT	Danbury
5/13	FFF	IL	Schaumburg
4/15	FFF	MD	Baltimore
8/28	FFF	MA	Cambridge
5/10	FFF	MN	Minneapolis
4/17	FFF	NJ	Paramus
8/13	FFF	OH	Cincinnati
9/19	FFF	CA	Sacramento
7/16	GC	AZ	Phoenix
5/7	GC	CA	Walnut Creek
7/19	GC	CA	Buena Park
9/12	GC	CO	Boulder
9/13	GC	CT	Hartford
11/11	GC	FL	Jacksonville
11/12	GC	FL	Orlando
11/14	GC	FL	Miami
7/15	GC	GA	Atlanta
7/8	GC	IL	Waukegan
7/10	GC	IN	Indianapolis
8/9	GC	LA	Baton Rouge
9/16	GC	MD	Baltimore
7/9	GC	MO	St. Louis
7/18	GC	NC	Raleigh
9/11	GC	NH	Portsmouth
9/19	GC	NJ	Princeton
9/20	GC	NJ	Paramus
9/9	GC	NM	Albuquerque
6/12	GC	NY	Rochester
7/12	GC	OH	Cincinnati
8/5	GC	OK	Oklahoma City
6/10	GC	PA	Pittsburgh
9/17	GC	PA	King of Prussia
7/16	GC	SC	Columbia
11/15	GC	TN	Memphis
8/6	GC	TX	San Antonio
8/8	GC	TX	Houston
9/11	GC	UT	Salt Lake City
7/19	GC	VA	Richmond
9/9	GC	VT	Burlington
5/10	GC	WA	Seattle
4/16	HPLC	IL	Schaumburg
4/17	HPLC	MO	St. Louis
5/14	HPLC	TX	Houston
5/15	HPLC	TX	San Antonio
6/11	HPLC	CO	Boulder
6/13	HPLC	CA	LaJolla
7/8	HPLC	NY	Rochester
7/10	HPLC	NY	Tarrytown
7/11	HPLC	NJ	Princeton
7/12	HPLC	PA	King of Prussia
8/5	HPLC	CA	Walnut Creek
8/8	HPLC	WA	Seattle
8/14	HPLC	OH	Cincinnati
8/27	HPLC	CT	Hartford
8/29	HPLC	MA	Marlborough
12/9	HPLC	FL	Miami
12/11	HPLC	GA	Atlanta
12/13	HPLC	NC	Raleigh

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Leak-Tight Seal for Agilent GCs

Vespel® Ring Inlet Seals

by Donna Lidgett, GC Accessories Product Marketing Manager

- ✔ Easy to use—Vespel® material seals the first time, every time, reducing variability among operators.
- ✔ Better sensitivity—lower leak rate reduces detector noise.
- ✔ Saves money—prevents oxygen from permeating the carrier gas, increasing column lifetime.
- ✔ Less maintenance—soft sealing area reduces wear on the injection port body.

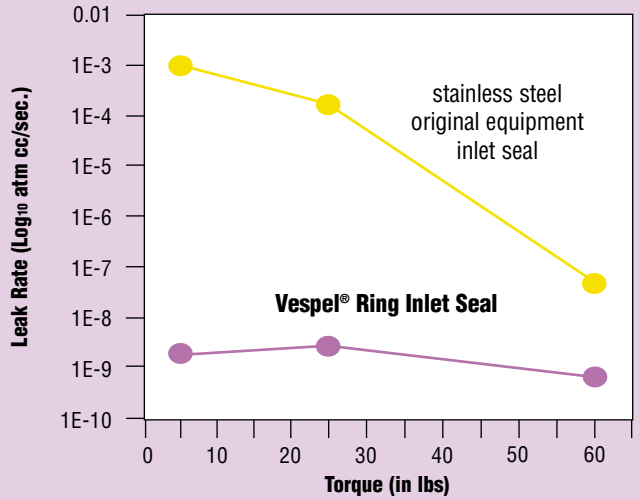
In Agilent split/splitless injection ports, the inlet seal sits at the base of the injector. Dirt, non-volatile residue, septum fragments, and other undesirable particles contaminate the inlet seal and decrease analytical linearity. The only way to maintain optimum performance is by frequently changing the inlet seal and ensuring the seal is leak-tight.

Restek designed the Vespel® Ring Inlet Seal to improve injection port performance on two levels. First, the Vespel® Ring Inlet Seal is made from high-quality stainless steel and features a Vespel® ring embedded into its face. This soft Vespel® ring will not harm the critical seal on the bottom of the injector body, and is outside the sample flow path, for worry-free chromatography.

Second, the Vespel® Ring Inlet Seal is designed to seal even after repeated temperature cycles and without retightening the reducing nut! With traditional stainless steel inlet seals, it is difficult to tighten to achieve a leak-tight seal. To determine the variances between a traditional seal and the new Vespel® Ring Inlet Seal, we compared the leak rate for each type of seal at increasing torque (Figure 1). Several inlet seals of each type were

Figure 1

The Vespel® Ring Inlet Seal achieves leak-tight seals even at low torque, reducing injection port wear and the chances of damaging the injection port.



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tested using a high sensitivity helium leak detector that can detect a helium leak rate as small as 1×10^{-10} atm cc/s. Notice how well the Vespel® Ring Inlet Seal performs at all levels, but especially at the lower torque levels. This means changing seals is now easier than ever, and the seal is reliable every time.

These new seals are available in stainless steel, gold-plated, or with a Silcosteel® coating. Use the stainless steel seal for normal analyses. To reduce breakdown and adsorption of active compounds, use the gold-plated or Silcosteel®-treated seals. The gold surface offers better inertness than standard stainless steel, and the Silcosteel® treatment provides inertness similar to that of fused silica capillary columns. Why trust a metal-to-metal seal when you can make leak-tight seals quickly and easily—and more reliably—with the Restek Vespel® Ring Inlet Seal?

Vespel® Ring Inlet Seals for Agilent 5890/6890 and 6850 GCs

0.8mm ID Vespel Ring Inlet Seal (washers included)	2-pk./price	10-pk./price
Gold-Plated	21562	21563
Silcosteel®	21564	21565
Stainless Steel	21560	21561
1.2mm ID Vespel Ring Inlet Seal (washers included)	**2-pk./price**	**10-pk./price**
Gold-Plated	21568	21569
Silcosteel®	21570	21571
Stainless Steel	21566	21567

by Brad Rightnour and Michael Goss, Instrument Innovations Team

For Easier GC Maintenance Try These New Tools from Restek

The Inlet Maintenance Kit includes these tools and many others.

Dislodge ferrules or remove silica deposits with the ***Jet Reamer/Ferrule Remover.***

The ***Capillary Installation Gauge*** makes seating the ferrule and installing the column consistent and easy.

The ***Inlet Liner Removal Tool*** safely removes an inlet liner from a hot injection port without cracking the liner—and you won't burn your fingers!

Inlet kits include:

- Viton® o-rings.
- Capillary nuts.
- Inlet seals.
- Reducing nut.
- Scoring wafer.
- 11 mm Thermolite® septa.
- 4.0mm single gooseneck liner.
- 0.4, 0.5, and 0.8mm ID graphite ferrules.
- 4.0mm split liner with wool.
- Capillary column caps.
- ¼- to 5/16-inch wrench.
- Septum puller.
- Installation gauge.
- Wire cleaning brush.
- Jet reamers/ferrule removers.
- Inlet liner removal tool.

The FID Maintenance Kit includes these tools and many others.

FID maintenance made easy with tools and replacement components specifically matched to your instrument.

The ***FID Ignitor*** meets original equipment specifications.

The ***High-Performance Silcosteel® FID Jet*** will stay clean longer—even when exposed to highly active compounds.

FID kits include:

- ¼-inch, 0.4, 0.5, and 0.8mm ID graphite ferrules.
- FID/NPD capilary adaptor.
- Capillary nuts.
- Jet reamers/ferrule removers.
- ¼-inch nut.
- Scoring wafer.
- Ignitor for either Agilent 5890 or 6890/6850 GCs.
- Capillary column caps.
- FID flow measuring adaptor.
- ¼- to 5/16-inch wrench.
- Installation gauge.
- Wire cleaning brush.
- High-performance Silcosteel® FID jet for either Agilent 5890 or 6890/6850 GCs.
- ¼-Inch nut driver for jet removal.

Description	qty.	cat.#	price
Inlet Maintenance Kit for Agilent 5890/6890/6850 GCs	kit	21069	
FID Maintenance Kit for Agilent 5890 GCs	kit	21070	
FID Maintenance Kit for Agilent 6890/6850 GCs	kit	21071	

High-Speed Analysis of Petrochemicals

Using OPN Res-Sil™ C GC Packings

by Barry Burger, Petrochemical and Packed GC Innovations Chemist

- ✔ Unique selectivity for saturated and unsaturated hydrocarbons.
- ✔ Innovative bonding chemistry for batch-to-batch reproducibility, excellent thermal stability, and long life.
- ✔ Wide range of bonded phases available.
- ✔ Equivalent to Waters Durapack® packings.

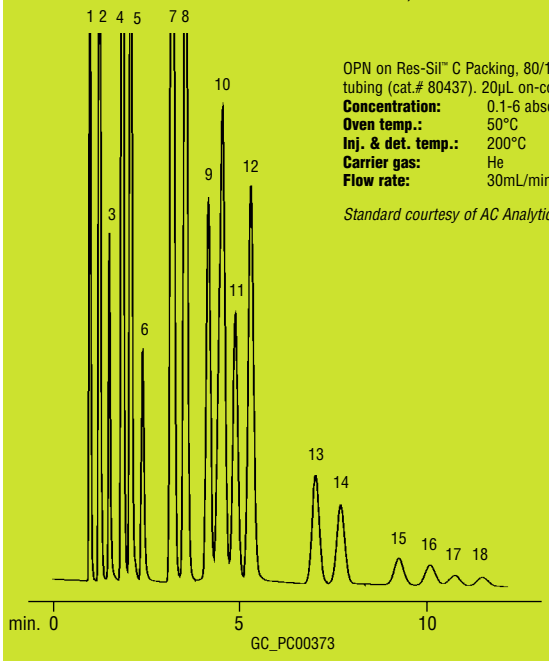
For over 25 years the process GC and petrochemical industries have used bonded silica packings such as Waters Durapak® packings for analysis of C1 to C4 hydrocarbons. These phases provide unique selectivity by modifying silica with a covalent attachment of either n-octane or cyano-propyl (OPN) functional groups. These phases have many advantages over conventional gas liquid chromatography packings because they yield faster separations, higher thermal stability, shorter conditioning times, and longer lifetimes. In the past, these packings had inconsistent reproducibility and limited availability. Restek's research team has solved these age old problems by developing Res-Sil™ C packings for consistent batch-to-batch performance and immediate delivery.

Unique Selectivity for Process GC and High-Speed Analysis of Petrochemicals

Speed of analysis is crucial for process GC, and in laboratory gas analyzers using multiple columns and valve switching for separation of complex gas mixtures. The Res-Sil™ C bonded packings are ideal for resolution of the saturated and unsaturated C4 hydrocarbons that are difficult to separate. The chromatogram in Figure 1 demonstrates the unique selectivity for separation of cis-2-butene before 1,3-butadiene using the OPN on Res-Sil™ C packing. This unique selectivity, when combined with other columns in series, provides petroleum and petrochemical method developers with a powerful tool for fast determination of C1 to C5 hydrocarbons.[1]

Figure 1

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



OPN on Res-Sil™ C Packing, 80/100 mesh, 12' x 2mm ID x ⅛" OD in SilcoSmooth™ tubing (cat.# 80437). 20µL on-column injection of refinery gas.

Concentration: 0.1-6 absolute mole %
Oven temp.: 50°C
Inj. & det. temp.: 200°C
Carrier gas: He
Flow rate: 30mL/min

Standard courtesy of AC Analytical Controls, Bensalem, PA.

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

Innovative Research and Stringent QA Provide Batch-to-Batch Consistency

Historically, one of the problems with bonded phases such as Carbowax®, n-octane, and OPN on Porasil® packing has been batch-to-batch variations in the amount of liquid stationary phase added to solid silica support. Restek's product development team pulled together chemists with experience in GC packings and HPLC phase development to innovate a new synthesis procedure. Utilizing new synthesis pathways, the amount of bonded liquid phase is precisely controlled in every batch, resulting in reproducible retention times and separations. Each production batch of Res-Sil™ C packing is quality assurance tested with a complex hydrocarbon mixture to meet demanding retention time and retention index specifications. Column bleed also is evaluated at the recommended maximum temperature of 150°C, as part of the QA test to ensure that retention shifts and high baselines are not observed.

A Full Line of Bonded GC Phases

Restek offers a wide range of bonded packings for packed GC columns, including Rtx®-1 and Stabilwax® phases, Carbowax® and n-octane phases on Res-Sil™ C packing, and OPN on Res-Sil™ C packing. Each of these packings has low bleed, conditioning time of less than 30 minutes, long lifetime, and consistent batch-to-batch reproducibility. Every batch of Restek's bonded phases is tested for bleed, efficiency, retention index and retention time reproducibility. In addition, Restek offers a full range of packed and micro-packed GC columns, available with specially-deactivated Silcosteel® tubing for improved inertness and efficiency.

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

Res-Sil™ C Packing Materials

Description	Temp. Limit (°C)	Mesh	Min. Qty.	cat.#	price/g
Res-Sil™ C	300	60/80	10g	25400	
	300	80/100	10g	25028	
Res-Sil™ B	300	60/80	10g	25401	
	300	80/100	10g	25080	
1% TCEP on Res-Sil™ B	175	80/100	10g	25081	
OPN on Res-Sil™ C	150	80/100	10g	25042	
n-Octane on Res-Sil™ C	150	80/100	10g	25030	
2% Carbowax® 1540 on Res-Sil™ C	150	80/100	10g	25044	

Restek's packed columns deliver the 1-2-3 punch!

1. Bonded stationary phases mean short conditioning times, low bleed levels, and unsurpassed column lifetimes.
2. SilcoSmooth™ tubing provides the inertness of glass and the durability of stainless steel.
3. Silcoport™ diatomaceous earth provides unsurpassed inertness for trace analyses.

HPLC Analysis of Preservatives

Using Ultra Aqueous and Pinnacle II™ Columns

by Rebecca Wittrig, Ph.D., Food, Flavors, and Fragrances Innovations Chemist

- ✔ Minimal sample preparation saves time.
- ✔ Ultra Aqueous C18 column provides superior retention and reproducibility for polar compounds.
- ✔ Pinnacle II™ Silica column resolves tocopherol isomers.

Preservatives are chemical compounds that are used in a wide range of applications to maintain overall product quality.[1] Some preservatives act as antimicrobial agents, some act as antioxidants, and some can perform both functions. Of the chemical compounds commonly used as preservatives, many can be effectively analyzed by high performance liquid chromatography (HPLC).[2] Because preservatives include a number of different compound types, there are a variety of HPLC stationary phases, mobile phases, and detectors that can be used.

Chemical preservatives kill or prevent the growth of microbes either by changing the microbes' environment or by reacting directly with them.[3] Antimicrobial compounds include organic acids, benzoate and sorbate salts, sulfur dioxide and sulfites, nitrites, propionates, and parabens. Organic acids, such as acetic acid and citric acid, can be used to control the pH of a product. For example, in food products these acidulants can lower the pH out of the optimum pH range for bacteria, yeast, and/or molds. Organic acids such as malic acid and citric acid can be found naturally in fruits, oxalic acid can be found in spinach and rhubarb, and tartaric acid can be found in grapes.

Using HPLC, concentrations of these preservatives can be monitored. However, analyzing polar organic acids can be difficult on conventional reversed phase columns, even when using low pH, highly aqueous mobile phases to suppress ionization of the acid molecules and maximize retention. The Ultra Aqueous C18 column provides enhanced retention and selectivity for challenging applications such as this. The novel bonding chemistry used for this phase allows the alkyl groups to remain extended, even in highly aqueous mobile phase, preventing the chain folding that occurs with conventional C18 phases. Therefore, stable and reproducible retention is possible even with 100% aqueous mobile phases. Notice the excellent retention for a series of organic acids using the Ultra Aqueous C18 column and UV detection (Figure 1).

Products containing fats and oils are prone to lipid oxidation, which can limit shelf life by promoting off-flavors, off-odors, and color changes. To inhibit lipid oxidation, antioxidants can be added to the product. Phenolic antioxidants include butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), propyl gallate (PG), and *tert*-butyl hydroquinone (TBHQ). These four, plus the tocopherols, are the primary antioxidants found in foods and beverages produced in the U.S. Phenolic antioxidants, such as BHT, are regulated by the US Food and Drug Administration (FDA), and can be added to many products at levels up to 200ppm, based on the fat content.

Phenolic antioxidants can be analyzed by reversed phase HPLC using a Pinnacle II™ C18 column and an acidified mobile phase. As with the analysis of organic acids, an acidic mobile phase is used to suppress ionization of the analytes. The HPLC separation of BHA, BHT, PG, and TBHQ using UV detection at 280nm shows how effectively these compounds can be separated using the Pinnacle II™ C18 column (Figure 2).

"Natural" antioxidants, such as tocopherols and tocotrienols, are used to inhibit lipid oxidation and to promote general health in the consumer. These compounds are found naturally in products such as fats and oils. When used as additives, however, they are regulated. Antioxidants such as tocopherols can be challenging to analyze, because they readily oxidize when exposed to light or oxygen. The analysis of four tocopherols by normal phase HPLC, using a Pinnacle II™ Silica column, shows how effectively these positional isomers can be separated (Figure 3). These compounds can be quantified using either fluorescence or UV detection.

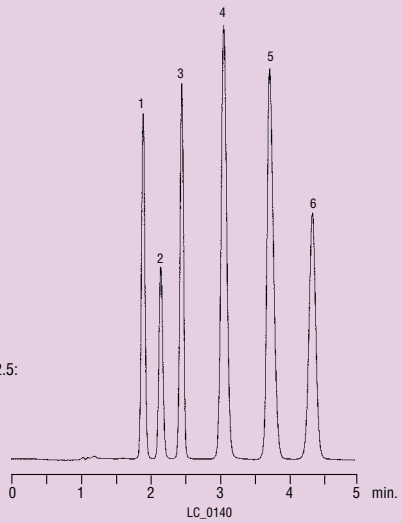
HPLC is a powerful tool for analyzing preservatives in a wide range of consumer products. One of its advantages is that many times only minimal sample preparation is required. Chromatographic techniques allow analysts to separate preservatives from other compounds in the sample matrix, improving the overall quality of the results. For analyzing organic acids, the Ultra Aqueous C18 column is the perfect choice, offering superior retention and reproducibility for polar compounds, even when using highly aqueous mobile phases. Pinnacle II™ C18 and silica HPLC columns are excellent choices for analyses of preservative compounds such as parabens, benzoate and sorbate salts, phenolic antioxidants, and tocopherols. Pinnacle II™ columns also are available with C8, phenyl, and amino stationary phases.

Figure 1

An Ultra Aqueous C18 column shows excellent retention of organic acids typically found in foods, beverages, personal care products, etc.

Peak List:	Conc. (µg/mL):
1. malonic acid	500
2. lactic acid	500
3. acetic acid	1000
4. citric acid	1000
5. succinic acid	2000
6. fumaric acid	10

Sample:	
Solvent:	HPLC-grade water
Inj.:	10µL
Column:	**Ultra Aqueous C18**
Catalog #:	9178565
Dimensions:	150 x 4.6mm
Particle size:	5µm
Pore size:	100Å
Conditions:	
Mobile phase:	50mM potassium phosphate, pH 2.5: acetonitrile (99:1)
Flow:	1.5mL/min.
Temp.:	25°C
Det.:	UV @ 210nm



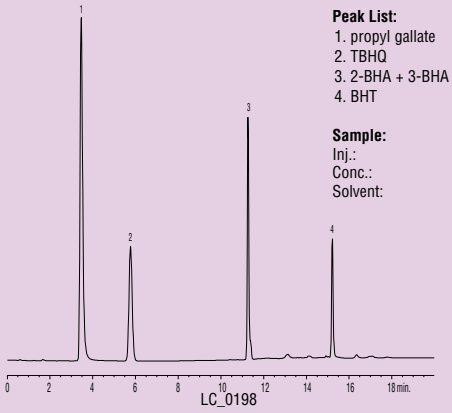
1. Fennema, Owen R. *Food Chemistry* (1996), Marcel Dekker, New York.
2. Nollet (ed.), *Food Analysis by HPLC* (2000), 2nd edition, Marcel Dekker, New York.
3. Foulke, Judith E. "A Fresh Look at Food Preservatives" in *FDA Consumer* (October 1993), US. Food & Drug Administration.

Questions?

Contact the industry's best Technical Service Team at 800-356-1688 or 814-353-1300, ext. 4, or contact your local Restek representative.

Figure 2

Phenolic antioxidants can be quantitated easily using a Pinnacle II™ C18 column and UV detection at 280nm.



LC_0198

Peak List:	conc.: (ppm)
1. propyl gallate	168
2. TBHQ	182
3. 2-BHA + 3-BHA	197
4. BHT	193

Sample:
Inj.: 10µL
Conc.: see peak list
Solvent: methanol

Column: **Pinnacle II™ C18**
Catalog #: 9214565
Dimensions: 150 x 4.6mm
Particle Size: 5µm
Pore Size: 110Å

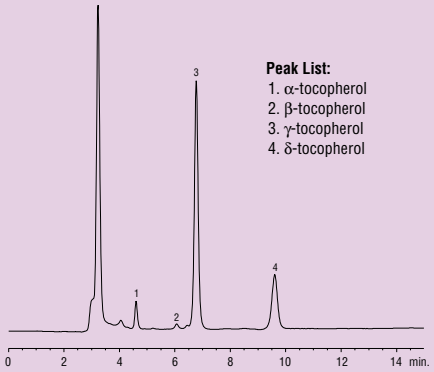
Conditions:
Mobile Phase: A = 1% acetic acid in water
B = methanol

Time (min.)	A (%)	B (%)
0	50	50
4	50	50
10	10	90
25	10	90
26	50	50

Flow: 1.0mL/min
Temp.: 30°C
Det.: UV @ 280nm

Figure 3

A Pinnacle II™ Silica column effectively separates the positional isomers of tocopherol by normal phase HPLC.



LC_0197

Peak List:
1. α-tocopherol
2. β-tocopherol
3. γ-tocopherol
4. δ-tocopherol

Sample:
Inj.: 10µL
Conc.: approx. 1.25% soy oil
Solvent: hexane

Column: **Pinnacle II™ Silica**
Catalog #: 9210565
Dimensions: 150 x 4.6mm
Particle Size: 5µm
Pore Size: 110Å

Conditions:
Mobile Phase: isopropyl alcohol:hexane (0.5:99.5)
Flow: 0.6 mL/min
Temp.: 30°C
Det.: UV @ 295nm

Ultra Aqueous C18 5µm Columns

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#	price
30mm	9178531	9178532	9178533	9178535	
50mm	9178551	9178552	9178553	9178555	
100mm	9178511	9178512	9178513	9178515	
150mm	9178561	9178562	9178563	9178565	
200mm	9178521	9178522	9178523	9178525	
250mm	9178571	9178572	9178573	9178575	

Pinnacle II™ C18 5µm Columns

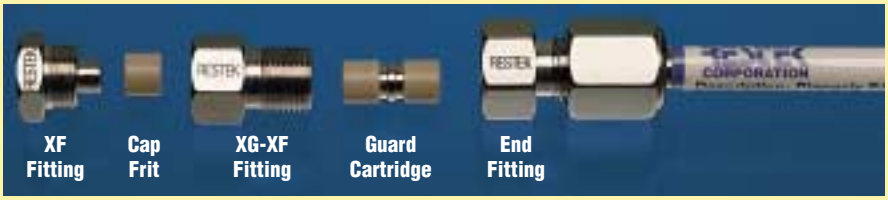
Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#	price
30mm	9214531	9214532	9214533	9214535	
50mm	9214551	9214552	9214553	9214555	
100mm	9214511	9214512	9214513	9214515	
150mm	9214561	9214562	9214563	9214565	
200mm	9214521	9214522	9214523	9214525	
250mm	9214571	9214572	9214573	9214575	

Pinnacle II™ Silica 5µm Columns

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#	price
30mm	9210531	9210532	9210533	9210535	
50mm	9210551	9210552	9210553	9210555	
100mm	9210511	9210512	9210513	9210515	
150mm	9210561	9210562	9210563	9210565	
200mm	9210521	9210522	9210523	9210525	
250mm	9210571	9210572	9210573	9210575	

Trident™ Integral HPLC Guard Column System

Maximum protection against contaminants and particulate matter.

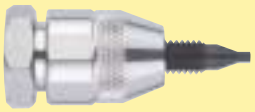


for more info

For additional information about Trident™ guard columns, request the Trident™ Fast Facts (lit. cat.# 59314 and 59896).

Trident™ Direct HPLC Guard Column System

Three levels of protection!

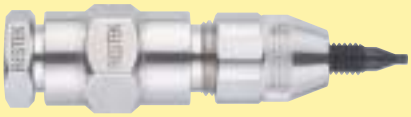


Trident™ Direct high-pressure filter

Protection against particulate matter.

Trident™ Direct 1cm guard cartridge holder with filter

Moderate protection against particulate matter and irreversibly adsorbed compounds.



Trident™ Direct 2cm guard cartridge holder with filter

Maximum protection against particulate matter and irreversibly adsorbed compounds.

MTBE & Oxygenate Analysis

Using an Rtx®-VGC GC Column

by Christopher English, Environmental Applications Chemist

- ✔ More accurate results through better resolution of target compounds.
- ✔ Determine low concentrations of oxygenates in the presence of aliphatic compounds.
- ✔ Resolve methyl-*tert*-butyl ether (MTBE) from target *tert*-butyl alcohol (TBA).

Gasoline and other fossil fuels are derived from petroleum and consist mainly of compounds containing only carbon and hydrogen atoms. Oxygenates are compounds that contain oxygen atoms in addition to carbon and hydrogen. Methyl *tert*-butyl ether (MTBE) is the most common fuel oxygenate. MTBE was first introduced into gasoline in 1979 to reduce overall emissions, replace lead and increase octane. In 1992, gasoline with up to 15% MTBE content by volume was used nationally to meet the first federally mandated wintertime reduction of carbon monoxide. With over one million underground fuel tanks in the United States alone, contamination of ground and surface water with oxygenates and gasoline components is a major environmental concern. Potentially, storage tanks worldwide will require cleanup. An equally challenging task is the identification and quantitation of these fuel-derived pollutants.

The US Environmental Protection Agency (EPA) has not sanctioned any method specifically for the analysis of oxygenates in gasoline. However, environmental laboratories have used a variety of methods to report these analytes, such as US EPA Methods 8015, 8020, and 8260. The three methods listed use a flame ionization detector (FID), photoionization detector (PID) and mass spectrometry (MS) respectively. Because gasoline range organic (GRO) samples can contain both petroleum and oxygenate components, chromatographic resolution is preferred regardless of the method used. One example involves the compounds MTBE and *tert*-butyl alcohol (TBA). Regulatory agencies recommend adding TBA to the target list for contaminated sites known to contain MTBE because it is both a breakdown product of MTBE and a gasoline additive. Both MTBE and TBA respond on the PID (Method 8020) and they share ions (MS by Method 8260), so MTBE and TBA must be resolved regardless of which detector is used.

The medium polarity Rtx®-VGC phase makes these columns ideal for the analysis of both hydrocarbons and oxygenates. The unique polarity of these columns improves the separation of oxygenates, which ensures more accurate detection when using PID. Restek does not recommend using FID alone for detecting these compounds.

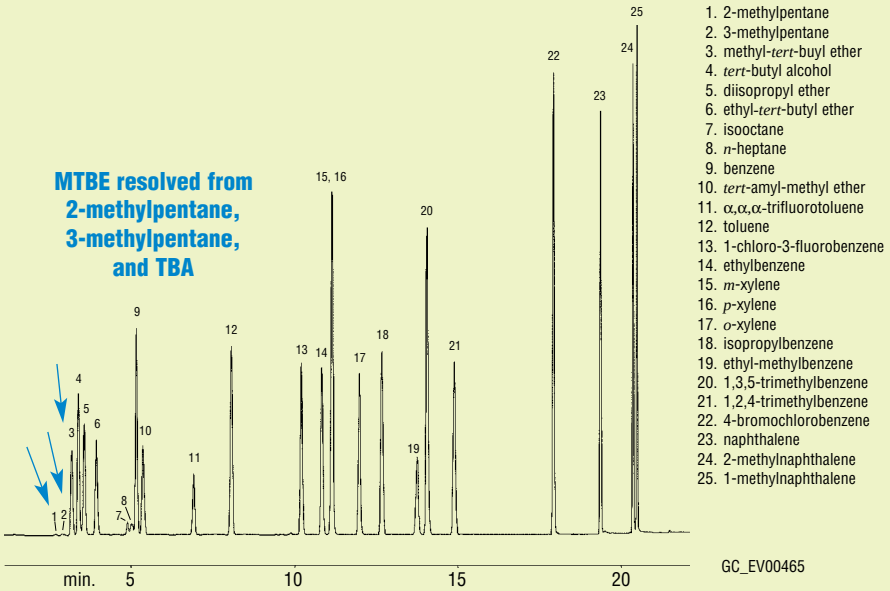
A 30m, 0.45mm ID, 2.55µm Rtx®-VGC column helps determine low concentrations of oxygenates in the presence of aliphatic compounds, resolving MTBE from 2-methylpentane, 3-methylpentane, and TBA (Figure 1). Furthermore, these optimized column dimensions allow the correct desorb flow rates from the purge and trap, faster analyses times, and better resolution of closely eluting peaks, compared to tradional 0.53mm ID columns. The oxygenates can be identified by using MS detection (Figure 2).

One commonly overlooked compound in the analysis of GRO samples is chlorobenzene. Figure 1 does not include chlorobenzene, however another analysis under identical conditions shows the retention time of chlorobenzene relative to ethylbenzene and *m/p*-xylene (Figure 3). Because the action limit for chlorobenzene is many times lower than for ethylbenzene, these compounds must be resolved. Environmental laboratories should keep in mind that even if clients do not specifically request data for chlorobenzene, these samples may require reprocessing in the future to determine if chlorobenzene is present. Without resolution of these analytes, it may not be possible to use the PID to provide such information.

The success of the GC/PID method is based on the ability of the analytical column to resolve oxygenates from the early-eluting alkanes, alkenes, and, to a lesser extent, alkynes. To minimize false positive results for MTBE or other oxygenates, it is important to separate 2-methylpentane and 3-methylpentane. Non-polar phases (e.g., Rtx®-1 and DB-MTBE columns) have been recommended for separating these compounds. However, these phases are incompatible with polar compounds, which can result in broader peaks and lower capacity for the alcohols. The Rtx®-VGC column will increase your level of confidence in your analytical data and prevent high bias. It is an ideal choice for analyzing gasoline additives in GRO samples.

Figure 1

Use purge and trap concentration and an Rtx®-VGC column to resolve oxygenates and other target gasoline compounds better than with other columns specifically designed to resolve MTBE.



Column: Rtx®-VGC 30m, 0.45mm, 2.55µm (cat.# 19408)
Inj.: Each component 100ppb in 5mL of RO water, except *tert*-butyl alcohol 5000ppb; 2/1-methynaphthalene 150ppb; ethyl methyl benzene 50ppb.
GC: Finnigan 9001
Oven temp.: 40°C (hold 2 min.) to 130°C @ 6°C/min. (hold 0 min.) to 230°C @ 30°C/min. (hold 2 min.).
Carrier gas: helium @ ~8mL/min.
Detector: Finnigan PID, make up 7mL/min., purge 7mL/min., set @ 0.35mV, base temperature 200°C.

Figure 2

Methyl-*tert*-butyl-ether, *tert*-butyl alcohol, and closely eluting hydrocarbons separated for identification by MS.

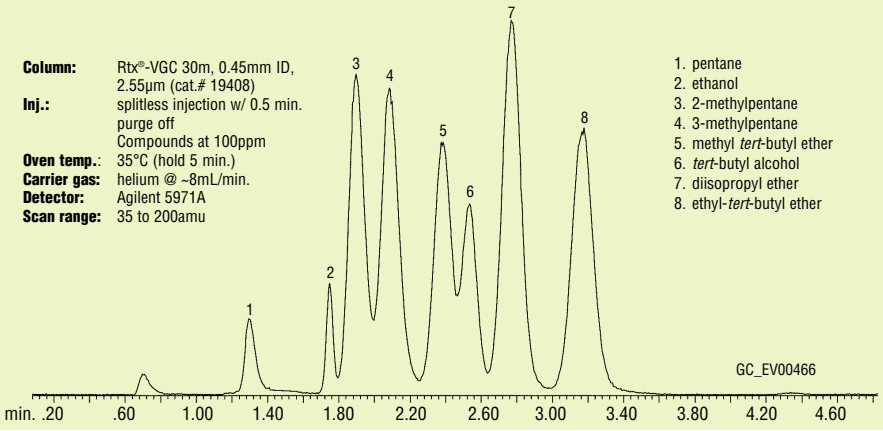
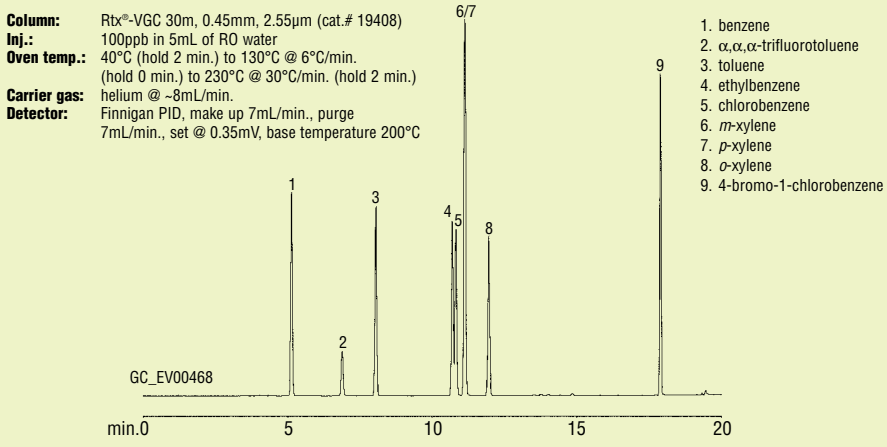


Figure 3

The Rtx®-VGC column resolves chlorobenzene from ethylbenzene, for reliable quantitation.



Rtx®-VGC (Fused Silica) *Stable to 260°C*

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	-40 to 240/260°C	19415	19416		
0.32mm	1.80	-40 to 240/260°C	19419	19420		
0.45mm	2.55	-40 to 240/260°C	19408		19409	
0.53mm	3.00	-40 to 240/260°C	19485	19488	19474	19489
ID	**df (µm)**	**temp. limits**	**20-Meter**	**40-Meter**		
0.18mm	1.00	-40 to 240/260°C	49414	49415		

Straight Silcosteel® Tubing

- Ideal for transfer lines, adsorbent traps, and thermal desorption tubes.
- Available in ⅛- and ¼-inch OD.
- Easily cut to specific lengths.

18" (457mm) Length

ID	OD	qty.	cat.#	price
0.085" (2.16mm)	⅛" (3.18mm)	ea.	20575	
0.085" (2.16mm)	⅛" (3.18mm)	5-pk.	20576	
0.210" (5.33mm)	¼" (6.35mm)	ea.	20577	
0.210" (5.33mm)	¼" (6.35mm)	5-pk.	20578	

Standards for Underground Storage Tank Monitoring (UST)

Monitoring underground storage tanks (UST) for leaks continues. Many states continue to modify existing analytical methods, with several states now using risk-based management of compounds involved. These new methods often pose challenges to the analyst, and require unique mixtures for calibration and matrix spike samples.

Restek continues to monitor the situation and respond with calibration mixtures to meet these needs. For our extensive selection of chemical standards for UST analyses, please refer to our 2002 *Chromatography Products Catalog* (lit. cat. #59662). For mixtures not listed there, please contact Technical Service at 800-356-1688 or 814-353-1300, ext. 4, or contact your local Restek representative.

California Oxygenates Mix

diisopropyl ether	2,000µg/mL
ethyl-*tert*-butyl ether	2,000
tert-amyl methyl ether	2,000
tert-butyl alcohol	10,000
methyl *tert*-butyl ether	2,000

In P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30465	30465-510	—
	with data pack	
30465-500	30465-520	30565

Universal "Y" Press-Tight® Connectors

- Split sample flow onto two different columns.
- Split a single column flow into two detectors.
- Perform confirmational analysis with a single injection.
- Fits 0.18, 0.25, 0.32, & 0.53mm ID columns.

Universal "Y" Press-Tight® Connector

qty.	cat.#	price
ea.	20405	
3-pk.	20406	

Restek is your #1 source for pesticide reference materials!

✔ Extensive selection of stock mixtures and single-component solutions.
✔ Custom mixtures made to your exact specifications.

Fax our reference materials department (814-353-1309) or contact your local Restek representative for more information.

New Analytical Reference Materials

ASTM D2887-01, Certified PAHs in Diesel #2, Single Component Explosives, US EPA 8270 Semivolatiles MegaMix™, Canadian PHC, and More!

1. Review method requirements
2. Verify compatibility and stability
3. Test raw materials
4. Certify balance and weights
5. Prepare glassware and ampuls
6. Prepare and package mixture
7. Test to assure quality
8. Validate expiration dates and shelf-life
9. Product packaging
10. Restek documentation
11. ISO 9001 registration
12. Custom reference materials program

For details on these 12 steps, refer to the annual *Chromatography Products Catalog* (lit. cat.# 59662) or contact Technical Service.

ASTM Method D2887-01 Calibration Mixes

- Meet new requirements for the 2001 revision of ASTM 2887-01.
- Pentane added.
- Equal weight/weight concentrations of all components—1% or 5%.
- Designed for both calibration and resolution tests—one sample for both test criteria.

pentane (C5)
hexane (C6)
heptane (C7)
octane (C8)
nonane (C9)
decane (C10)
undecane (C11)
dodecane (C12)
tetradecane (C14)
pentadecane (C15)
hexadecane (C16)
heptadecane (C17)
octadecane (C18)
eicosane (C20)
tetracosane (C24)
octacosane (C28)
dotriacontane (C32)
hexatriacontane (C36)
tetracontane (C40)
tetratetracontane (C44)

1% weight each in carbon disulfide, 1g solution/ampul

Each	5-pk.	10-pk.
31674	31674-510	—
	with data pack	
31674-500	31674-520	31774

5% weight each, 1g/ampul

Each	5-pk.	10-pk.
31675	31675-510	—
	with data pack	
31675-500	31675-520	31775

Certified PAHs in Diesel #2

- Confirm diesel #2 TPH and priority PAHs in a single analysis.
- Certificate of Analysis includes concentration of TPH and certified concentrations of individual PAHs.
- Complete data pack available.

Certified PAHs	Typical Certified Conc. (ppm)
acenaphthene	7
acenaphthylene	1
anthracene	13
fluorene	6
1-methylnaphthalene	110
2-methylnaphthalene	60
naphthalene	30
phenanthrene	13

50,000ppm diesel #2 in methylene chloride, PAH concentrations listed above, 1mL/ampul

Each	5-pk.	10-pk.
31673	31673-510	—
	with data pack	
31673-500	31673-520	31773

Certified Aromatics in Gasoline

- Confirm unleaded gasoline TPH, BTEX, and aromatics in a single analysis.
- Certificate of Analysis includes concentration of TPH and certified concentrations of BTEX and individual aromatics.
- Complete data pack available.

Certified for:
benzene
ethylbenzene
m-ethyltoluene
o-ethyltoluene
p-ethyltoluene
isopropylbenzene
methyl *tert*-butyl ether
naphthalene
n-propylbenzene
toluene
1,2,3-trimethylbenzene
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
m-xylene
o-xylene
p-xylene

5,500ppm gasoline in P&T methanol, certified components listed, 1mL/ampul

Each	5-pk.	10-pk.
30485	30485-510	—
	with data pack	
30485-500	30485-520	30585

Certified BTEX in Unleaded Gas Composite Standard

Certified for:
benzene
ethylbenzene
isopropyl benzene
methyl *tert*-butyl ether
naphthalene
toluene
m-xylene
o-xylene
p-xylene

5,500ppm gasoline in P&T methanol, certified components listed, 1mL/ampul

Each	5-pk.	10-pk.
30237	30237-510	—
	with data pack	
30237-500	30237-520	30337

Canadian PHC

- Meets CCME 2001 Petroleum Hydrocarbons in Soil Method–Tier1.
- Primary reference calibration standards for quantification of four fractions.

CCME PHC Calibration Mix

decane (C10)
hexadecane (C16)
tetratriacontane (C34)
5,000µg/mL each in toluene, 1mL/ampul

Each	5-pk.	10-pk.
31684	31684-510	—
	with data pack	
31684-500	31684-520	31784

C50 in Toluene

pentacontane (C50)
10µg/mL in toluene, 1mL/ampul

Each	5-pk.	10-pk.
31685	31685-510	—
	with data pack	
31685-500	31685-520	31785

New Analytical Reference Materials

ASTM D2887-01, Certified PAHs in Diesel #2, Single Component Explosives, US EPA 8270 Semivolatiles MegaMix™, Canadian PHC, and More!

Single-Component Explosives Solutions

- Support the US Department of Defense base closures and remediation.
- Mixtures and singles to support HPLC US EPA Method 8331.
- Mixtures and singles to support GC/ECD US EPA Method 8095.
- Internal standards and surrogates to support both methods.

Solvent A=acetonitrile

Compound Packaged 1mL/ampul	Solvent	µg/mL	Individual	Individual w/data pack	5-pk.	5-pk. w/data pack	10-pk. w/data pack
2-amino-4,6-dinitrotoluene mix	A	1,000	31670	31670-500	31670-510	31670-520	31770
4-amino-2,6-dinitrotoluene mx	A	1,000	31671	31671-500	31671-510	31671-520	31771
3,5-dinitroaniline mix	A	1,000	31661	31661-500	31661-510	31661-520	31761
1,3-dinitrobenzene mix	A	1,000	31662	31662-500	31662-510	31662-520	31762
2,4-dinitrotoluene mix	A	1,000	31663	31663-500	31663-510	31663-520	31763
2,6-dinitrotoluene mix	A	1,000	31664	31664-500	31664-510	31664-520	31764
HMX mix	A	1,000	31665	31665-500	31665-510	31665-520	31765
nitrobenzene mix	A	1,000	31657	31657-500	31657-510	31657-520	31757
2-nitrotoluene mix	A	1,000	31659	31659-500	31659-510	31659-520	31759
3-nitrotoluene mix	A	1,000	31660	31660-500	31660-510	31660-520	31760
4-nitrotoluene mix	A	1,000	31658	31658-500	31658-510	31658-520	31758
RDX mix	A	1,000	31666	31666-500	31666-510	31666-520	31766
tetryl mix	A	1,000	31667	31667-500	31667-510	31667-520	31767
1,3,5-trinitrobenzene mix	A	1,000	31668	31668-500	31668-510	31668-520	31768
2,4,6-trinitrotoluene mix	A	1,000	31669	31669-500	31669-510	31669-520	31769

Semivolatiles MegaMix™ US EPA Method 8270

- Fewest mixtures needed for calibration and matrix spikes.
- Mixtures formulated for maximum stability.
- Contains most routinely analyzed compounds.

Column: Rtx®-5 30m, 0.25mm ID, 0.25µm (cat.# 10223-124)
Inj. temp.: 250°C
Carrier gas: helium
Flow rate: 1mL/min.
Oven temp.: 35°C (hold 2 min.) to 330°C @ 4°C/min.
Det.: MS
Tranfer line temp.: 300°C

See peak list for compounds.

8270 MegaMix™

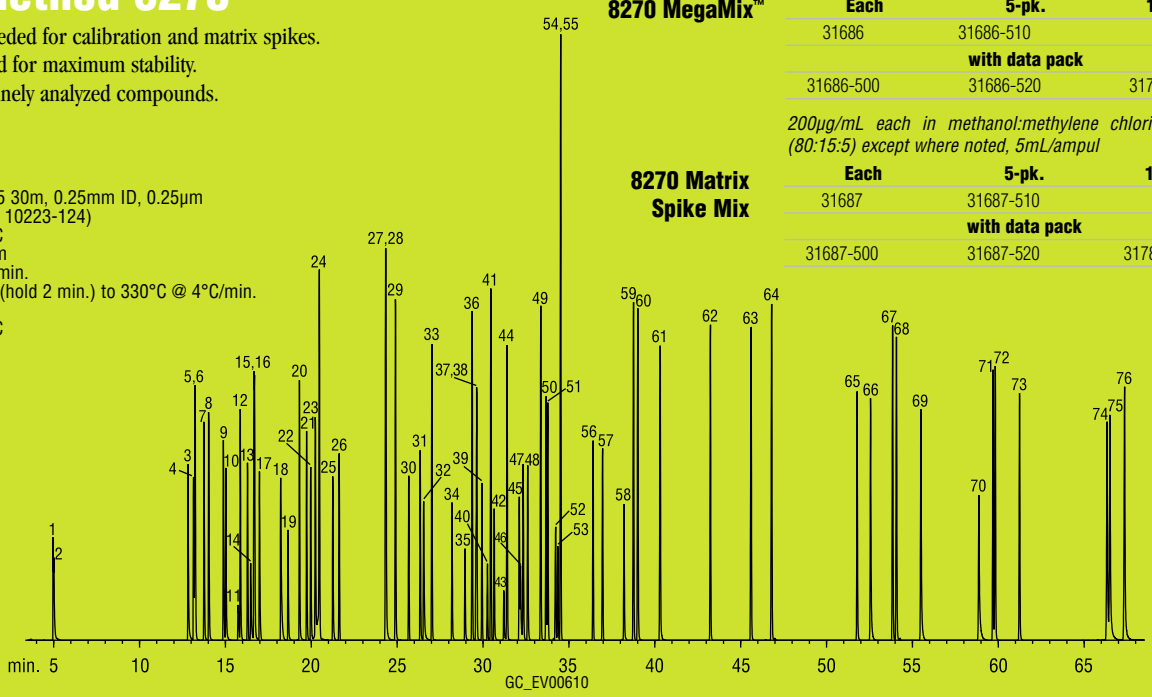
1,000µg/mL each in methylene chloride:benzene (75:25) except where noted, 1mL/ampul

Each	5-pk.	10-pk.
31686	31686-510	—
	with data pack	
31686-500	31686-520	31786

8270 Matrix Spike Mix

200µg/mL each in methanol:methylene chloride:benzene (80:15:5) except where noted, 5mL/ampul

Each	5-pk.	10-pk.
31687	31687-510	—
	with data pack	
31687-500	31687-520	31787



1. pyridine
2. N-nitrosodimethylamine
3. aniline
4. phenol
5. bis(2-chloroethyl)ether
6. 2-chlorophenol
7. 1,3-dichlorobenzene
8. 1,4-dichlorobenzene
9. 1,2-dichlorobenzene
10. benzyl alcohol
11. bis(2-chloroisopropyl)ether
12. 2-methylphenol
13. hexachloroethane
14. N-nitroso-di-*n*-propylamine
15. 4-methylphenol*
16. 3-methylphenol*
17. nitrobenzene
18. isophorone
19. 4-nitrophenol
20. 2,4-dimethylphenol
21. bis(2-chloroethoxy)methane
22. 2,4-dichlorophenol
23. 1,2,4-trichlorobenzene
24. naphthalene
25. 4-chloroaniline
26. hexachlorobutadiene
27. 2-methylnaphthalene
28. 4-chloro-3-methylphenol
29. 1-methylnaphthalene
30. hexachlorocyclopentadiene
31. 2,4,6-trichlorophenol
32. 2,4,5-trichlorophenol
33. 2-chloronaphthalene
34. 2-nitroaniline
35. 1,4-dinitrobenzene
36. acenaphthylene
37. 1,3-dinitrobenzene
38. dimethyl phthalate
39. 2,6-dinitrotoluene
40. 1,2-dinitrobenzene
41. acenaphthene
42. 3-nitroaniline
43. 2,4-dinitrophenol
44. dibenzofuran
45. 2,4-dinitrotoluene
46. 2-nitrophenol
47. 2,3,4,6-tetrachlorophenol
48. 2,3,5,6-tetrachlorophenol
49. fluorene
50. 4-chlorophenyl phenyl ether
51. diethyl phthalate
52. 4-nitroaniline
53. 4,6-dinitro-2-methylphenol
54. diphenylamine
55. azobenzene
56. 4-bromophenyl phenyl ether
57. hexachlorobenzene
58. pentachlorophenol
59. phenanthrene
60. anthracene
61. carbazole
62. di-*n*-butyl phthalate
63. fluoroanthene
64. pyrene
65. benzyl butyl phthalate
66. bis(2-ethylhexyl)adipate
67. benzo(a)anthracene
68. chrysene
69. bis(2-ethylhexyl)phthalate
70. di-*n*-octyl phthalate
71. benzo(b)fluoroanthene
72. benzo(k)fluoroanthene
73. benzo(a)pyrene
74. indeno(1,2,3-cd)pyrene
75. dibenzo(a,h)anthracene
76. benzo(ghi)perylene

**Concentration is 500µg/mL.*

Tips for Maximizing HPLC Column Lifetime

by Greg France, HPLC Product Marketing Manager, and Terry Reid, HPLC Applications Chemist

- ✔ Extend the lifetime of your analytical HPLC column.
- ✔ Achieve more reproducible analyses.
- ✔ Protect your chromatographic system.

The analytical column is the heart of your HPLC system. Taking proper care of your column ensures that you get reproducible results for a maximum number of sample injections. By following the recommendations listed below, you can extend the lifetime of your column and improve the accuracy and reproducibility of your results.

Sample Preparation
The cleaner your samples, the longer your column will last. Obviously, there will be times when you must compromise column lifetime in order to reduce sample preparation efforts. At the very least, though, you should filter samples through a 0.45µm syringe tip filter to ensure they are free of particles. Restek offers a range of syringe filters, from 0.20µm to 1.00µm, in either nylon or PTFE. Also, make sure that all samples are completely soluble in the mobile phase. If you are running a mobile phase gradient, sample solubility should be verified at the low and high extremes of organic content.

Mobile Phases
Filter mobile phases (especially buffers) through a 0.45µm membrane. A 0.2µm filter removes bacteria as well as other particles. If you are running a gradient with a buffer, make sure the buffer salt remains in solution even at the highest percent organic.

Column Storage
Store columns in an appropriate solvent that contains at least 10% organic solvent and is free of salts or buffers. Seal a stored column with end plugs and label the column with the storage solvent, to avoid inadvertently combining incompatible solvents in the column when using it later. If a column is dedicated to a particular analysis, the analyte or method name should be recorded on the column.

Column Protection
Use a guard filter and guard column. Change filters and guard columns regularly or at the first signs of impaired performance (e.g., increased pressure, loss of efficiency or peak symmetry). The Trident™ guard system allows you to choose a configuration that best suits the needs of your particular application: a Trident™ in-line guard cartridge, the Trident™ Direct system, or the Trident™ Integral guard system. (For illustrations of Trident™ systems, see page 5.) Each system can be built from a particulate filter frit, a filter frit with a 1cm guard column, or a filter frit with a 2cm guard column. Alternatively, a Trident™ in-line or direct system can be configured as a guard cartridge holder without the filter frit.

Column Cleanup
Ben Franklin said, "An ounce of prevention is worth a pound of cure." The same rings true for column maintenance: clean the column periodically. If you wait for column performance to significantly deteriorate before cleaning, you may have to repeat your analyses. The most effective cleanup technique is to backflush the column to remove the strongly adsorbed impurities that tend to accumulate at the head of the column. Do not flush into the detector because particles from the inlet frit could damage the detector. To clean the column, flush it with a strong solvent (e.g., high organic for reversed phase columns). If you are using a buffer, make sure it is completely flushed from the column and the system before switching to a mobile phase with a high percentage of organic solvent. This will prevent salts from precipitating. If you are doing repetitive isocratic analyses, periodically (i.e., every ten samples or so) use a gradient from weaker to stronger solvent to prevent the accumulation of strongly retained impurities. When cleaning columns, flush with a minimum of ten column volumes.

Avoid Extremes
Follow the column manufacturer's recommendations for usable pH and temperature ranges. Most silica-based columns will have a recommended pH range of around 2.5-7.5. Lifetimes for these columns will be maximized if the pH can be maintained between 3 and 7. Similarly, although most silica-based columns can be operated at temperatures up to 80°C, lifetime generally will be greatest if the column temperature does not exceed 40°C. Column manufacturers usually do not specify pressure limits, but higher pressures decrease column lifetime, especially as pressure exceeds 2000psi (~140 bar). Pressures above 3000psi (~200 bar) should be avoided if at all possible.

Conclusion
By following these recommendations, you will prolong the life of your analytical column and reduce the chances for unpleasant surprises during your routine analyses. If you have any questions, the Restek Technical Service Team will be happy to help you—call 814-353-1300 or 800-356-1688, ext. 4, or contact your local Restek representative.

Resprep™ SPE Syringe Filters

Filter Diameter	Porosity	qty.	Nylon	PTFE
13mm	0.20µm	100-pk.	26066	26068
13mm	0.45µm	100-pk.	26067	26069
25mm	0.20µm	50-pk.	26070	26072
25mm	0.45µm	50-pk.	26071	26073
25mm	1.00µm	50-pk.	—	— 26074

Trident™ Direct Guard Column System

Description	qty.	cat.#	price
High-pressure filter	ea.	25082	
1cm guard cartridge holder with filter	ea.	25084	
2cm guard cartridge holder with filter	ea.	25086	
Connection tip for Waters®-style end fittings	ea.	25088	
PEEK® tip standard fittings	ea.	25087	
Replacement Cap Frits: 4mm, 2.0µm	5-pk.	25022	
Replacement Cap Frits: 4mm, 0.5µm	5-pk.	25023	
Replacement Cap Frits: 2mm, 2.0µm	5-pk.	25057	

for more info — For additional information about Trident™ guard columns, request the Trident™ Fast Facts (lit. cat.# 59314 and 59896).

Inert PEEK® Tubing

Description	qty.	cat.#	price
PEEK® Tubing, 1/16" OD x 0.0025" ID Natural	3m	25320	
PEEK® Tubing, 1/16" OD x 0.005" ID Red Stripe	3m	25065	
PEEK® Tubing, 1/16" OD x 0.007" ID Yellow Stripe	3m	25066	
PEEK® Tubing, 1/16" OD x 0.010" ID Blue Stripe	3m	25067	
PEEK® Tubing, 1/16" OD x 0.020" ID Orange Stripe	3m	25068	

Fast Analysis of Aroclor® PCBs

With the GC Racer* Temperature Programming System

by Mike Goss, Instrument Innovations Engineer, Gary Stidsen, Innovations Manager, and Donna Lidgett, GC Accessories Product Marketing Manager

- ✔ Increase sample throughput without investing a large amount of capital.
- ✔ Easy to operate and install—truly a "plug and play" accessory.
- ✔ Operate your Agilent 5890 GC as fast as a 6890!

Fast temperature programs are commonly used in gas chromatographic (GC) applications to speed up elution of high boiling point compounds and late eluters. The most common GC, the Agilent 5890, has a maximum temperature program rate of 70°C/min., but heating elements in the 5890 only allow this maximum temperature program rate to be maintained up to a temperature of 100°C. For analysts trying to push temperature ramps as fast as possible, this inhibited program rate leads to longer analyses times and broader peaks. Now, using the GC Racer auxiliary heating unit, temperature program rates of up to 70°C/min. can be maintained up to 350°C (Figure 1).

Restek and Zip Scientific have teamed up to bring you the GC Racer temperature programmer, which consists of a program controller and a resistive heating element placed on the floor of the GC oven. The heating element is connected to the controller, which is plugged into the main PC board of the GC. When the GC Racer programmer detects that the factory heating elements are not keeping up with the programmed heating rate, the GC Racer heater is brought into the circuit to augment the heat being supplied to the oven. The GC Racer system will maintain a temperature program rate of 70°C/min. up to 350°C, or a rate of 60°C/min. to temperatures as high as 450°C.

The simplicity of GC Racer components and ease of installation make the GC Racer system a "must have" add-on accessory for every 5890 GC.

The auxiliary heater design is similar to that of the original GC heater. The auxiliary heater plugs into the GC Racer controller, which plugs into the main PC board on the GC. The only other connection needed is to plug the GC Racer controller into a 120V electrical service. At no time during the installation of the GC Racer system does the column need to be removed from the oven, or disconnected from the detector or injection port.

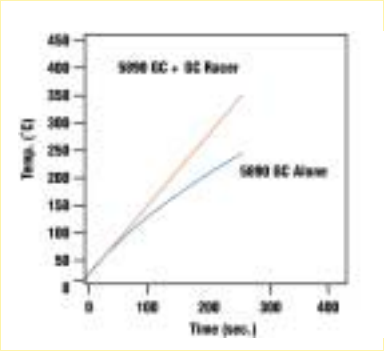
As part of cost reduction efforts, many laboratories try to reduce individual sample analysis times in the interest of increasing overall throughput. High-temperature simulated distillation analyses can take as long as an hour, especially when samples contain hydrocarbons up to C110. An effective technique to reduce analysis time is to use rapid temperature programming. By attaching the GC Racer to your Agilent 5890 GC, you can, for example, analyze Aroclor® standards in less than 6 minutes (Figure 2). This can be up to an 80% reduction in analysis time.

The GC Racer system is a highly effective, easily installed new tool in the quest for high-speed GC. The analysis speed that now can be achieved will lead to significant long-term savings of time and money by decreasing run time and increasing sample throughput.

For our large selection of Aroclor® PCB standards, refer to our annual *Chromatography Products Guide* (lit. cat.# 59662) or visit our website.

Figure 1

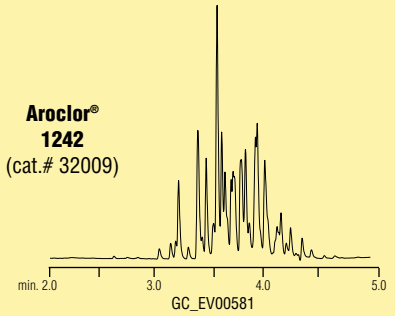
The GC Racer allows a temperature program rate of up to 70°C/min. to be maintained up to 350°C!

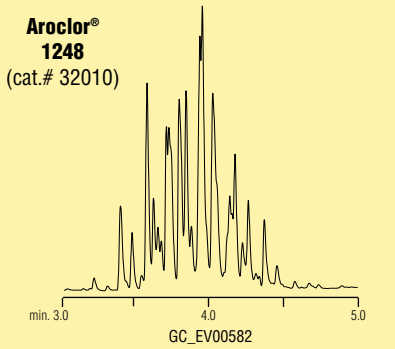


GC: Agilent 5890; **Service:** 120V/15 amp; **Start Temp:** 20°C; set oven to 400°C and monitor oven temp.

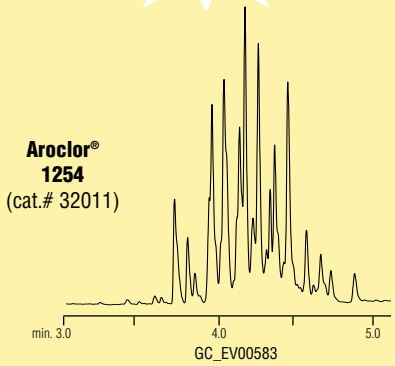
Figure 2

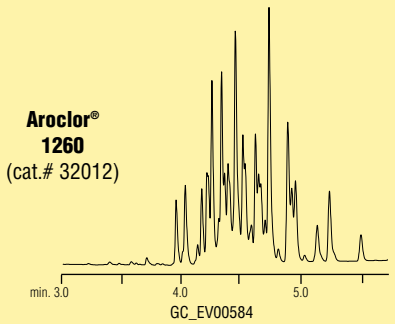
Quantitative analysis of Aroclor® standards in less than 6 minutes, using an Rtx®-5 GC column and the GC Racer system.





Reduce quantitative analysis time by up to 80%—increase sample throughput up to 5 times!





Column:	Rtx®-5 15m, 0.32mm ID, 0.50µm (cat.# 10236)
Inj.:	1µL splitless (hold 0.5 min.), Siltek™ Drilled Uniliner® Liner (cat.# 21054-214.1)
Conc.:	400 ppb
Inj. temp.:	250°C
Carrier gas:	hydrogen
Linear velocity:	head pressure at 5 psi
Oven temp.:	110°C (hold 1 min.), 60°C/min. to 300°C, (hold 5 min.)
Det. temp.:	310°C

GC Racer Temperature Programming System

Description	qty.	cat.#	price
For Agilent 5890 Series II (only) GC	ea.	23024	
For Agilent 5890A (only) GC	ea.	23025	

*Patent pending.

Inert Inlet System Improves Responses for Chlorinated Pesticides

Using the Drilled Uniliner® Inlet Liner

by Gary Stidsen, Innovations Manager

- ✔ Inert sample path eliminates injection port discrimination.
- ✔ Reduce detection limits by using a splitless injection port without an on-column injector.

For years, chemists analyzing chlorinated pesticides have tried many different injection techniques in attempts to find the best balance between inertness and ability to contend with sample contamination. Cool on-column, split, splitless, and direct injection, and variations of these injection techniques, are used today.

Now, a specially modified injection port liner, developed by Restek chemists, reduces sample contact with active metal parts in split/splitless injection ports. This Drilled Uniliner® liner, shown in Figure 1, gives the benefits of both direct injection and splitless injection. The advantage of this liner is that the column is connected to the liner by a press-fit connection, thus preventing the sample from contacting the metal at the bottom of the injection port. Also, the hole on the side of the liner allows the purge flow to escape from the liner when the injection mode is switched from splitless to split.

Figure 1

The drilled hole in a Uniliner® injection port liner makes direct injection possible with EPC systems by equalizing pressure in the injection port.

Inertness

With the Drilled Uniliner® liner, the sample is transferred directly from the injection port to the column and contacts only this glass inlet liner. The configuration allows the sample to be "funneled" into the column entrance, thereby eliminating the need for vaporization aids such as fused silica wool. The test probes endrin and 4,4'-DDT are good indicators of injection port inertness. Analyses of the performance evaluation mixture (PEM) show low breakdown of these compounds when using this liner.

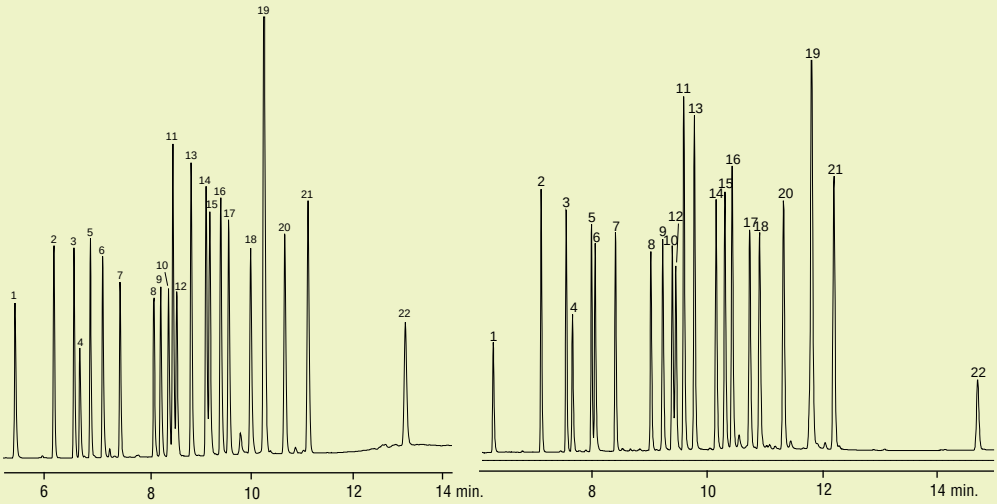
For maximum inertness, we recommend using the Drilled Uniliner® liner in combination with an Stx™-CLPesticides capillary column, as shown in Figure 2. Siltek™ surface deactivation in these columns further ensures maximum responses for the labile pesticides endrin, 4,4'-DDT, and methoxychlor. A special polymer formulation designed for organochlorine pesticides enables you to achieve excellent separation of the 22 chlorinated pesticides in Figure 2 in less than 15 minutes. Comparable analyses on Rtx®-CLPesticides columns take up to 24 minutes (Figure 2, inset).

Calibration

Linearity and continuing calibration checks for the chlorinated pesticides also are critical parameters that must be monitored. Table 1 indicates typical

Figure 2

Stx™-CLPesticides and Stx™-CLPesticides2 columns provide rapid, excellent separation of chlorinated pesticides, and a Siltek™-deactivated Drilled Uniliner® inlet liner helps ensure high responses for sensitive analytes.



30m, 0.32mm ID, 0.5µm **Stx™-CLPesticides** (cat.# 11544)

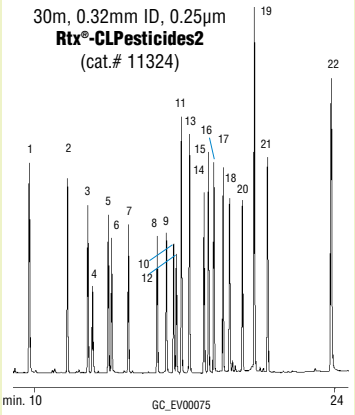
30m, 0.32mm ID, 0.25µm **Stx™-CLPesticides2** (cat.# 11444)

GC_EV00512

Oven temp.:	110°C (hold 1 min.) to 245°C @ 20°C/min. to 300°C @ 6°C/min.
Inj. & det. temp.:	210°C / 310°C
Carrier gas:	helium
Dead time:	0.8min. @ 120°C
Inlet liner:	Siltek™ Drilled Uniliner® liner (cat.# 21055-214.5)
Inj.:	1µL direct injection of 20/40/200ng/mL std. concentration in hexane
Make-up gas:	nitrogen

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

Traditional analyses on Rtx®-CLPesticides columns take up to 24 minutes.



On-column concentration:	16–160pg organochloride pesticide mix AB#2 (cat.# 32292)
Oven temp.:	120°C (hold 1 min.) to 300°C (hold 10 min.) @ 9°C/min.
Inj. port:	Direct, Uniliner® liner (cat.# 20335), at 200°C
Detector:	ECD, 300°C with Anode Purge
Dead time:	1.9 min.
Head pressure:	8.7psi (constant)
Flow rate:	1.3mL/min. @ 120°C, helium.

Table I

Small relative standard deviations for calibration factors show a Drilled Uniliner® inlet liner efficiently transfers the sample to the column.

	Relative Standard Deviation (%)	
	CLPesticides Column	**CLPesticides2 Column**
α-BHC	7.0	7.5
γ-BHC	3.3	3.8
β-BHC	10.2	9.6
δ-BHC	6.0	7.1
heptachlor	4.2	10.9
aldrin	2.3	1.2
heptachlor epoxide	10.7	8.6
γ-chlordane	6.8	6.7
α-chlordane	8.3	6.7
4,4' DDE	2.3	3.3
endosulfan I	9.2	8.3
dieldrin	7.6	6.6
endrin	4.9	5.3
4,4' DDD	2.7	4.1
endosulfan II	9.9	9.7
4,4' DDT	3.8	2.4
endrin aldehyde	12.3	13.3
methoxychlor	10.2	10.8
endosulfan sulfate	9.3	10.6
endrin ketone	7.9	5.3

Standard: cat#. 32292, 8/16/80µg/mL in hexane:toluene (1:1)
Calibration curve standards: 5/10/50ng/mL in hexane
20/40/200ng/mL in hexane
80/160/800ng/mL in hexane

Chlorinated pesticides listed in US EPA Method 8081.

linearity values (% RSD) obtained using a Drilled Uniliner® liner and Rtx®-CLPesticides columns. Equivalent results are obtained with Stx™-CLPesticides columns.

When using a Drilled Uniliner® liner, the efficient transfer of sample from the injection port to the column might allow more nonvolatile material to enter the column. For many samples this will not be an issue, especially if solid phase cleanup is performed. Using a guard column will help maintain the analytical column. When necessary, the guard column can be trimmed to remove the portion where the nonvolatile material collects (usually the first 6-12 inches).

Conclusion

The Drilled Uniliner® liner provides the advantages of both direct and splitless injection liners. The liner provides a more inert sample pathway to transfer the sample from the injector to the analytical column, and it helps eliminate injection port discrimination. A Drilled Uniliner® liner will reduce the detection limits for injections made on a splitless injection port, without the need for an on-column injector.

Stx™-CLPesticides (Fused Silica with Siltek™ deactivation)

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.25	-60 to 310/330°C	11540	11543
0.32mm	0.50	-60 to 310/330°C	11541	11544
0.53mm	0.50	-60 to 310/330°C	11542	11545

Stx™-CLPesticides2 (Fused Silica with Siltek™ deactivation)

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.20	-60 to 310/330°C	11440	11443
0.32mm	0.25	-60 to 310/330°C	11441	11444
0.53mm	0.42	-60 to 310/330°C	11442	11445

Uniliner® Inlet Liners for Agilent GCs

DI Liners for Agilent 5890/6890 GCs (For 0.25/0.32/0.53mm ID Columns)	ID*/OD & Length (mm)	cat.#/price ea.	cat.#/price 5-pk.
 **Drilled Uniliner®**	4.0 ID 6.3 OD x 78.5	21054	21055
 **Siltek™ Drilled Uniliner®**	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5
 **Siltek™ 1mm Drilled Uniliner®**	1.0 ID 6.3 OD x 78.5	21390-214.1	21391-214.5

*Nominal ID at syringe needle expulsion point.

Pesticide Surrogate Mix

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

Each	5-pk.	10-pk.
32000	32000-510	
	with data pack	
32000-500	32000-520	32100

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

Each	5-pk.	10-pk.
32292	32292-510	
	with data pack	
32292-500	32292-520	32392

RESTEK INNOVATION!

The Drilled Uniliner® inlet liner is the first inlet liner to allow direct injections in EPC systems!

Online Ordering at www.restekcorp.com

Analyzing Organophosphorus Pesticides

Using an Rtx®-OPPesticides2 Column and GC/MS

by Gary Stidsen, Innovations Manager

- ✔ Low column bleed improves resolution of OPPs.
- ✔ Fast analysis times.
- ✔ Allows GC/MS analysis of many OPPs.

Typically, organophosphorus pesticides (OPPs) are analyzed using a dual-column gas chromatograph with flame photometric detectors (FPD) or nitrogen phosphorus detectors (NPD). These detectors provide the sensitivity needed for reporting limits, but only for a finite number of compounds. However, the list of compounds continues to increase, due to the introduction of new pesticides.

As the list of compounds grows, the use of gas chromatography/mass spectrometry (GC/MS) becomes more desirable. GC/MS analysis requires only one column and detector, thereby eliminating the complexity of the dual-column GC system. Column bleed and analyte resolution are important factors in GC/MS analysis. Minimizing column bleed is important to increase the signal-to-noise ratio for the late-eluting compounds at the detection limits. Although the MS can spectrally resolve the sample components, fewer coelutions in the chromatogram make data processing easier, including confirmation by spectral identification.

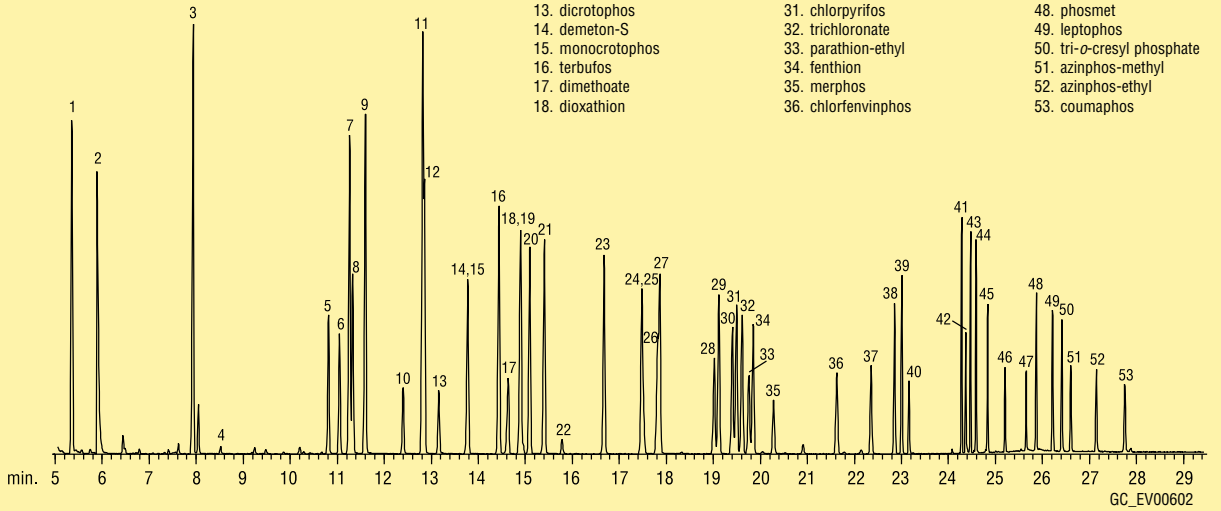
Using sophisticated computer-assisted stationary phase development (CASPD) software, Restek chemists designed the Rtx®-OPPesticides2 column to provide low bleed and improve resolution for OPP analysis by MS (Figure 1). Not only are the separations dramatically improved compared to traditional columns for this analysis, but also the analysis time can be reduced by almost 50%.

The combination of an Rtx®-OPPesticides2 GC column with MS is an excellent system for analyzing long lists of OPPs. The column exhibits very low bleed and excellent resolution for these compounds.

Figure 1

The Rtx®-OPPesticides 2 column shows excellent resolution of 53 organophosphorus pesticides.

1. dichlorvos
2. hexamethylphosphoramide
3. mevinphos
4. trichlorfon
5. TEPP
6. demeton-O
7. thionazin
8. tributyl phosphate (IS)
9. ethoprop
10. naled
11. sulfotepp
12. phorate
13. dicrotophos
14. demeton-S
15. monocrotophos
16. terbufos
17. dimethoate
18. dioxathion
19. fonophos
20. diazinon
21. disulfoton
22. phosphamidon isomer
23. dichlorofenthion
24. chlorpyrifos methyl
25. phosphamidon
26. parathion-methyl
27. ronnel
28. fenitrothion
29. aspon
30. malathion
31. chlorpyrifos
32. trichloronate
33. parathion-ethyl
34. fenthion
35. merphos
36. chlorfenvinphos
37. crotoxyphos
38. stirofos
39. tokuthion
40. merphos oxon (breakdown product)
41. ethion
42. fensulfothion
43. bolstar
44. carbophenothion
45. famphur
46. triphenyl phosphate (SS)
47. EPN
48. phosmet
49. leptophos
50. tri-*o*-cresyl phosphate
51. azinphos-methyl
52. azinphos-ethyl
53. coumaphos



Column:	Rtx®-OPPesticides2 30m, 0.25mm ID, 0.25µm (cat.# 11243)	**Carrier gas:**	helium, constant flow
Sample:	Custom Mix, plus: 8140/8141 OP Pesticides Calibration Mix A (cat.# 32277) 8141 OP Pesticides Calibration Mix B (cat.# 32278) Triphenylphosphate Standard (cat.# 32281) Tributylphosphate Standard (cat.# 32280) 1µL, 100ppm each (100ng on column)	**Flow rate:**	1.0mL/min.
		Oven temp.:	80°C (hold 0.5 min.) to 140°C @ 20°C/min. to 210°C @ 4°C/min. (hold 1 min.) to 280°C @ 30°C (hold 5 min).
		Det:	MS
Inj.:	1.0µL splitless (hold 0.4 min.), 4mm double gooseneck inlet liner (cat.# 20785)	**Transfer line temp.:**	280°C
		Scan range:	35-400amu
Inj. temp.:	250°C	**Ionization:**	EI

Rtx®-OPPesticides2 Columns (Fused Silica) *Stable to 310°C*

ID	df (µm)	temp. limits	20-Meter	30-Meter
0.18mm	0.20	-20 to 310/330°C	11244	
0.25mm	0.25	-20 to 310/330°C		11243
0.32mm	0.32	-20 to 310/330°C		11241
0.53mm	0.50	-20 to 310/330°C		11242

Restek will create the right solution for you!

"The Company Chromatographers Trust"

Restek should be your first choice for custom-made reference materials. Our inventory of over 3,000 pure, characterized, neat compounds ensures you of maximum convenience, maximum value, and minimum time spent blending mixtures in your lab.

- ✔ Quotations supplied quickly.
- ✔ Mixtures made to your EXACT specifications.
- ✔ Most reference materials shipped within 5-7 days after receipt of your order.*

For our online custom reference material request form, visit http://www.restekcorp.com/stdreq.htm

*Availability of raw materials and final product testing required may affect delivery of some mixtures. International orders require additional shipping time.

8140/8141 OP Pesticide Calibration Mix A

azinphos methyl
bolstar (sulprofos)
chlorpyrifos
coumaphos
demeton, O and S
diazinon
dichlorvos
disulfoton
ethoprop
fensulfothion
fenthion
merphos
methyl parathion
mevinphos
naled
phorate
ronnel
stirofos
tokuthion (prothiofos)
trichloronate

200µg/mL each in hexane:acetone (95:5), 1mL/ampul

Each	5-pk.	10-pk.
32277	32277-510	—
	with data pack	
32277-500	32277-520	32377

8141 OP Pesticide Calibration Mix B

dimethoate
EPN
malathion
monocrotophos
parathion
sulfotepp
TEPP

200µg/mL each in hexane:acetone (95:5), 1mL/ampul

Each	5-pk.	10-pk.
32278	32278-510	—
	with data pack	
32278-500	32278-520	32378

8140/8141 Internal Standards & Surrogates

NPD Detector:

Internal Standard: 1-bromo-2-nitrobenzene
Surrogate: 4-chloro-3-nitrobenzotrifluoride

1-bromo-2-nitrobenzene

1,000µg/mL in acetone, 1mL/ampul

Each	5-pk.	10-pk.
32279	32279-510	—
	with data pack	
32279-500	32279-520	32379

4-chloro-3-nitrobenzotrifluoride

1,000µg/mL in acetone, 1mL/ampul

Each	5-pk.	10-pk.
32282	32282-510	—
	with data pack	
32282-500	32282-520	32382

FPD Detector:

Internal Standard: none recommended
Surrogate: tributylphosphate and triphenylphosphate

tributylphosphate

1,000µg/mL in acetone, 1mL/ampul

Each	5-pk.	10-pk.
32280	32280-510	—
	with data pack	
32280-500	32280-520	32380

triphenylphosphate

1,000µg/mL in acetone, 1mL/ampul

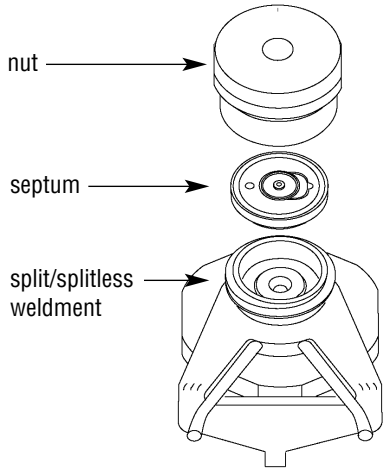
Each	5-pk.	10-pk.
32281	32281-510	—
	with data pack	
32281-500	32281-520	32381

Septum Alternative Provides Longer Life & Wear Resistance

Merlin Microseal™ Septa

by Donna Lidgett, GC Accessories Product Marketing Manager

- ✔ For Agilent 5890/6890/6850 GCs compatible with EPC.
- ✔ High-pressure capability allows operation from 2 to 100psi.
- ✔ A top wiper rib improves resistance to particulate contamination and can be taken apart for cleaning.
- ✔ Reduces shedding of septum particles into the injection port liner, eliminating a major source of septum bleed and ghost peaks.
- ✔ Reduces the risk of septum leaks during extended automated runs.



Merlin Microseal™ Septa

Microseal™ High-Pressure Septa 400 Series	Merlin#	Similar to Agilent#	cat.#	price
Nut kit (1 nut, fits 300 & 400 series septa)	403	5182-3445	22809	
Standard kit (nut, 2 high-pressure septa)	404	Not offered	22810	
Starter kit (nut, 1 high-pressure septum)	405	5182-3442	22811	
Replacement high-pressure septum (1 septum)	410	5182-3444	22812	

Microseal™ Septa, 300 Series	Merlin#	Similar to Agilent#	cat.#	price
Standard kit (nut, 2 septa)	304	5181-8833	22813	
Starter kit (nut, 1 septum)	305	5181-8816	22814	
Microseal replacement septum (1 septum)	310	5181-8815	22815	
Replacement PTFE washers (2-pk.)	311	5181-0853	22808	

RESTEK Behind the Scenes

What's New from the Analytical Reference Materials Team?

In addition to adding new product formulations to meet your changing requirements, we've been very busy behind the scenes working for you. Visit **www.restekcorp.com/certfind.htm** to view the information you need at your convenience.

- ✔ All Material Safety Data Sheets (MSDSs) have been converted to 16-part format.
- ✔ All 800+ stock product MSDSs are available on-line.
- ✔ 1000s of Certificates of Analysis are available on-line, too.

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Fax: 44 28 9081 4576
restekeurope@cs.com
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Lit. Cat.# 59430

Please direct your comments on this publication to Carrie Sprout, Graphic Designer, at carrie@restekcorp.com or call Restek, ext. 2151.

Restek air canisters are being used at Ground Zero to monitor air quality. We are proud to have our products used in the 9/11 clean-up efforts at the Twin Towers site.

Thanks for a Great Pittcon® '02!

It was our best Pittcon® conference yet! We got to meet many of you, showcase our new products, and learn about advances in our industry. Thanks for stopping by our booth and talking with the Chromatography Wizards. Be sure to check out the technical presentations and posters at **www.restekcorp.com.** The winners of our daily drawing are listed below. Congratulations!

Monday, March 18: Pinnacle II™ HPLC Column of your choice (up to $405 value)
Winner: Michael McCroan, The Minute Maid Company

Tuesday, March 19: SGT Triple Gas Filter & Single-Position Baseplate (up to $320 value)
Winner: Gregory Ostrom, Naval Warfare Center

Wednesday, March 20: 30-Meter Fused Silica Capillary GC Column of your choice (up to $525 value)
Winner: Wesley Wortham, BASF Agro

Thursday, March 21: $500 in stock Analytical Reference Materials (up to $500 value)
Winner: Neil Springarn, S & N Labs

New Literature

- ✔ Stx™-CLPesticides Columns Provide Improved System Inertness for Chlorinated Pesticides Analyses—*Applications Note* (lit. cat.# 59351B)
- ✔ HPLC Analyses of Preservatives—*Applications Note* (lit. cat.# 59398)
- ✔ EPA 8100 Analysis Using Rtx®-5Sil MS, Rtx®-CLPesticides and Rtx®-CLPesticides2 Columns—*Applications Note* (lit. cat.# 59196A)
- ✔ Low ppb-Level Sulfur Analysis Using Sulfinert™ Sample Cylinders—*Applications Note* (lit. cat.# 59164A)
- ✔ GC Accessories Products—*Flyer* (lit. cat.# 59208B)
- ✔ Gas Purification Products for GCs—*Flyer* (lit. cat.# 59216B)
- ✔ Products for the Petrochemical Market—*Flyer* (lit. cat.# 59298)
- ✔ Ultra Aqueous C18 HPLC Column—*Fast Facts* (lit. cat.# 59371)
- ✔ US EPA Method 8260B Standards—*Fast Facts* (lit. cat.# 59332A)
- ✔ UST Products for the State of Texas—*Fast Facts* (lit. cat.# 59394)
- ✔ Sulfinert™ Products—*Fast Facts* (lit. cat.# 59318A)
- ✔ GC Racer - Fast GC Temperature Programmer—*New Product Flyer* (lit. cat.# 59297)
- ✔ Vespel® Ring Inlet Seals—*New Product Flyer* (lit. ca.# 59431)
- ✔ Rtx®-200 GC Column—*New Product Flyer* (lit. cat.# 59439)
- ✔ Integra-Guard® GC Columns—*New Product Flyer* (lit. cat.# 59441)
- ✔ 2002 Seminar Tours—*New Product Flyer* (lit. cat.# 59282A)
- ✔ Air Monitoring Products—*Catalog* (lit. cat.# 59661A)
- ✔ Genuine Restek Replacement Parts for Agilent GCs—*Catalog* (lit. cat.# 59627C)

RESTEK

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the RESTEK Advantage

Innovators of High Resolution Chromatography Products

Optimized Analysis of Brominated Flame Retardants

Using an Rtx®-500 GC Capillary Column

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

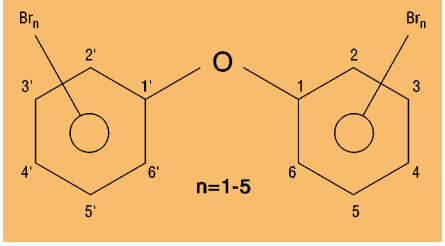
- ✔ Elutes decabromodiphenyl ether in 30 minutes.
- ✔ Low bleed for sensitive ECD and MS analyses.
- ✔ Separates other higher molecular weight compounds.

Brominated flame retardants are an emerging environmental concern that present a unique challenge to analysts. One of the most heavily used types of brominated flame retardants are the polybrominated diphenyl ethers (PBDEs). These compounds have the structure shown in Figure 1, with 1 to 10 bromines substituted on the two rings. This makes these compounds fairly heavy (up to approximately 1000amu), thus placing difficult requirements on the gas chromatographic (GC) analytical system. While the analysis may be performed using either electron capture detection (ECD) or mass spectrometric detection (MS), the compounds require a high oven temperature to elute in a reasonable amount of time. This requires an analytical GC column featuring high-temperature fused silica or metal tubing and a high-temperature stationary phase that has both low bleed and the selectivity necessary to separate the PBDE congeners.

Restek chemists, working in conjunction with Karen MacPherson and Eric Reiner at the Ontario Ministry of the Environment, have developed a new fused silica capillary GC column and analytical procedure for separating PBDE congeners in a reasonable time. The new Rtx®-500 column incorporates a carborane-stabilized polydimethylsiloxane polymer in special high-temperature fused silica tubing. The column can be heated to 380°C, and exhibits very low bleed at this extreme temperature. The column combines the stability required for separating higher molecular weight compounds with the sensitivity required for ECD or MS analysis.

PBDE congeners up to decabromodiphenyl ether (PBDE 209) are separated in less than 45 minutes using the new Rtx®-500 column in a GC-high resolution MS analysis (Figure 2). In a GC-ECD analysis of the same PBDE congeners, decabromodiphenyl ether is eluted in 30 minutes. This same system has been used for baseline separation of toxic PCB congeners and is currently under investigation for analyses of the brominated and chlorinated dioxins and furans. If you must analyze PBDEs or other high molecular weight compounds, we highly recommend the Rtx®-500 column for fast separations and reliable quantitation.

Figure 1
PBDEs can have high molecular weight, due to bromine substitution around the two rings.



(contd. on page 2)

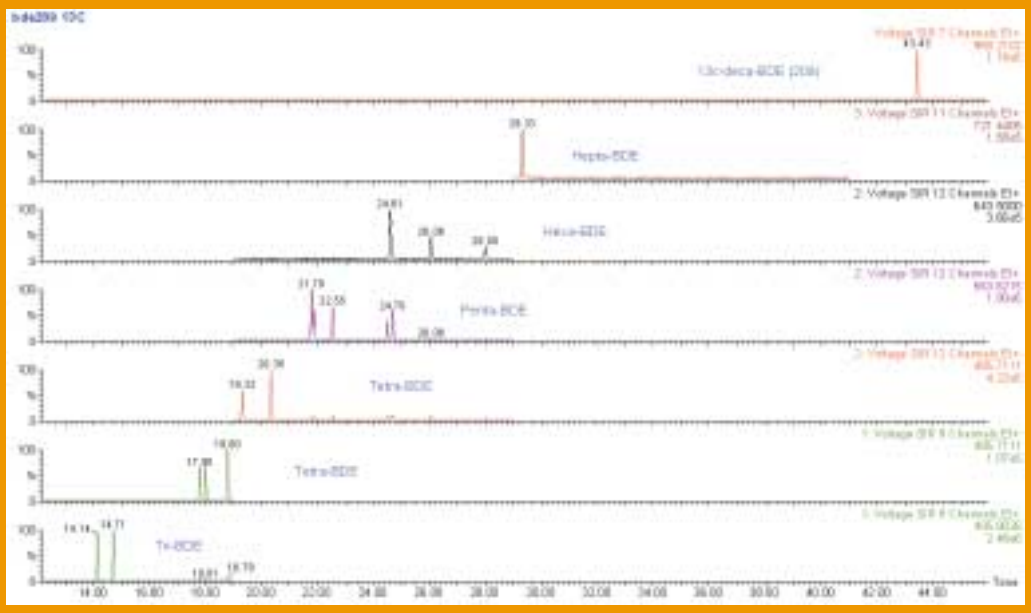
in this issue

RESTEK
800-356-1688 ★ 814-353-1300
www.restekcorp.com

2002 vol. 3

Figure 2

The Rtx®-500 column completely separates PBDE congeners in less than 45 minutes in a GC/MS analysis.



NB DecaBDE (last elutor) elutes at ~43 min.

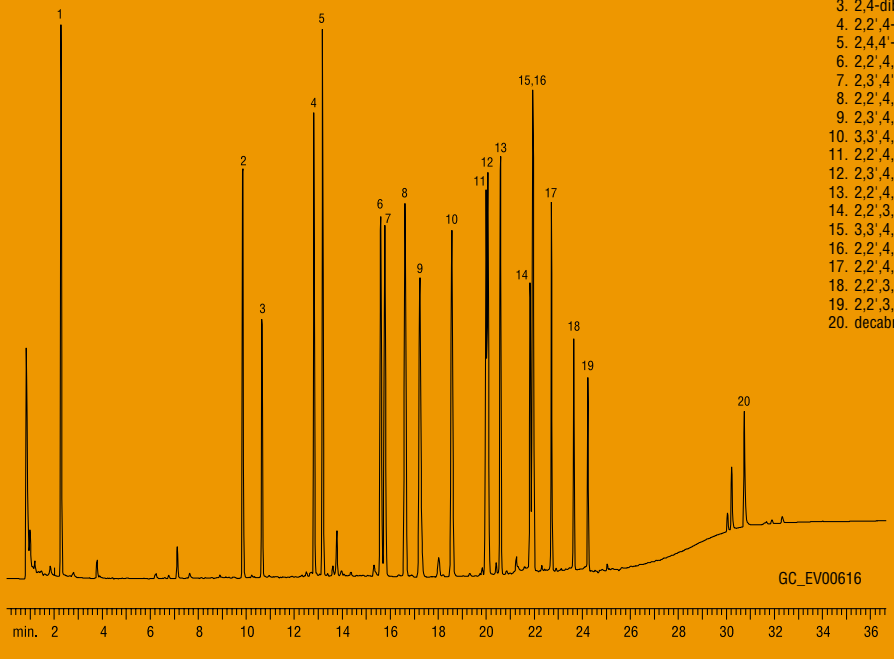
Chromatogram courtesy of Ontario Ministry of the Environment

Reference materials courtesy of Wellington Laboratories, Guelph, Ontario, Canada
www.well-labs.com
US Distributor:
TerraChem Inc. U.S.A.
8600 Shawnee Mission Pkwy., Suite 305
Shawnee Mission, KS 66202
Phone: 913-722-4919
Toll-free: 877-809-7039
Fax: 913-722-4669
Website: http://www.terrachem.com
Email: info@terrachem.com

Column: Rtx®-500 30m, 0.25mm, 0.15µm (cat.# 10750); **GC:** Agilent 6890+; **Sample:** Wellington Laboratories BDE Mix C (300pg/µL each in nonane); **Inj.:** 1µL splitless injection, drilled Uniliner® (cat.# 21054); **Oven temp.:** 100°C (hold 0.64 min.) to 110°C @ 10°C/min. (hold 0 min.) to 180°C @ 80°C/min. (hold 23 min.) to 350°C @ 5°C/min.; **Flow rate:** constant @ 1.5mL/min; **Injector temp.:** 300°C; **Instrument configuration:** Micromass Autospec-UltimaNT (High Resolution Mass Spectrometer); Source Temperature: 300°C

Figure 3

When used with less expensive ECD methodology, an Rtx®-500 column resolves PBDEs in 30 minutes.



Peak	IUPAC #
1. 4-bromodiphenyl ether	3
2. 4,4'-dibromodiphenyl ether	15
3. 2,4-dibromodiphenyl ether	7
4. 2,2',4-tribromodiphenyl ether	17
5. 2,4,4'-tribromodiphenyl ether	28
6. 2,2',4,5'-tetrabromodiphenyl ether	49
7. 2,3',4',6-tetrabromodiphenyl ether	71
8. 2,2',4,4'-tetrabromodiphenyl ether	47
9. 2,3',4,4'-tetrabromodiphenyl ether	66
10. 3,3',4,4'-tetrabromodiphenyl ether	77
11. 2,2',4,4',6-pentabromodiphenyl ether	100
12. 2,3',4,4',6-pentabromodiphenyl ether	119
13. 2,2',4,4',5-pentabromodiphenyl ether	99
14. 2,2',3,4,4'-pentabromodiphenyl ether	85
15. 3,3',4,4',5-pentabromodiphenyl ether	126
16. 2,2',4,4',5,6'-hexabromodiphenyl ether	154
17. 2,2',4,4',5,5'-hexabromodiphenyl ether	153
18. 2,2',3,4,4',5'-hexabromodiphenyl ether	138
19. 2,2',3,4,4',5',6-heptabromodiphenyl ether	183
20. decabromodiphenyl ether	209

Column: Rtx®-500 30m, 0.53mm ID, 0.15µm (cat.# 10752)
GC: Agilent 5890
Sample: Wellington Laboratories BDE-Mix C (300pg/µL each in nonane)
Inj.: 1.0µL direct injection, drilled Uniliner® (cat.# 21054)
Inj. temp.: 380°C
Carrier gas: hydrogen, constant pressure
Linear velocity: 7.69mL/min. (66.7 cm/sec.) @ 100°C
Dead time: CH_2Cl_2 headspace 0.76 min. @ 100°C
Make-up gas: 40mL/min.
Oven temp.: 100°C (hold 1 min.) to 260°C @ 15°C (hold 5 min.) to 380°C @ 15°C (hold 15 min.)

Ordering Information | Rtx®-500 Columns (Fused Silica)

(Crossbond® carborane/dimethyl polysiloxane) Stable to 380°C

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.25mm	0.15	-60°C to 380°C	10750	10751
0.53mm	0.15	-60°C to 380°C	10752	

for more info
request Applications Note 59389A.

Vespel® Ring Inlet Seal

Seals the First Time, Every Time

by Donna Lidgett, GC Accessories Product Marketing Manager

- ✔ Easy-to-use, patent-pending design makes a better seal, easily.
- ✔ Prevents oxygen from damaging your columns.
- ✔ Reduces wear on the injection port body.

In Agilent split/splitless injection ports, it can be difficult to make and maintain a good seal with a conventional metal inlet disk. The metal-to-metal seal dictates that the analyst apply considerable torque to the reducing nut, and, based on our testing, this does not ensure a leak-tight seal. Over the course of oven temperature cycling, metal seals are prone to leaks, which ultimately can degrade the capillary column, and cause other analytical difficulties.

Our Vespel® Ring Inlet Seal greatly improves injection port performance—it seals even after repeated temperature cycles and without retightening the reducing nut! This seal features a Vespel® ring embedded into its face. This soft Vespel® ring will not harm the critical seal on the injector body, and is outside the sample flow path. Tests using a high sensitivity helium leak detector indicate the Vespel® Ring Inlet Seal seals equally effectively at torques of 5lb. or 60lb. (Figure 1).

Why trust a metal-to-metal seal when you can make leak-tight seals quickly and easily—and more reliably—with the Restek Vespel® Ring Inlet Seal? Use the stainless steel seal for analysis of unreactive compounds. To reduce breakdown and adsorption of active compounds, use the gold-plated or Silcosteel®-treated seals. The gold surface offers better inertness than standard stainless steel; Silcosteel® treatment provides inertness similar to that of fused silica capillary columns.

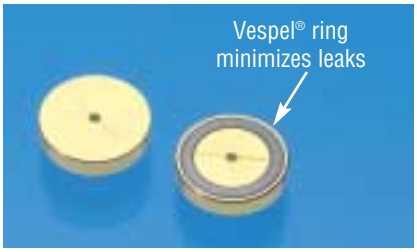
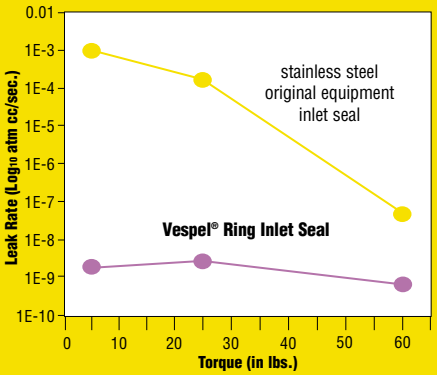


Figure 1
The Vespel® Ring Inlet Seal achieves leak-tight seals even at low torque, reducing the chance of leaks.



Ordering Information | Vespel® Ring Inlet Seals for Agilent 5890/6890 and 6850 GCs

0.8mm ID Vespel Ring Inlet Seal (washers included)	2-pk.	10-pk.
Gold-Plated	21562	21563
Silcosteel®	21564	21565
Stainless Steel	21560	21561
1.2mm ID Vespel Ring Inlet Seal (washers included)*	**2-pk.**	**10-pk.**
Gold-Plated	21568	21569
Silcosteel®	21570	21571
Stainless Steel	21566	21567

*For dual-column installations.

A Compact, Sensitive Leak Detector For Every GC Analyst

The Restek Leak Detective™ II

by Donna Lidgett, GC Accessories Product Marketing Manager

- ✔ Fast results—responds to trace leaks in less than 2 seconds.
- ✔ Sensitive—detects trace leaks at 1 x 10^{-4}cc/sec.; as low as 100ppm.
- ✔ Micro-chip design improves sensitivity and response time over previous models.
- ✔ Compact, ergonomic design is easy to hold and operate with one hand.
- ✔ Battery-operated for portability (one 9 volt) instant auto-zeroing.

Gas leaks in your GC system can increase detector noise, cause baseline instability, waste carrier gas, and damage valuable analytical columns. Leak checks should be a regular part of your GC maintenance program. The new Leak Detective™ II electronic leak detector is the affordable solution for detecting gas leaks. It will identify minute gas leaks that might go undetected by liquid leak detectors.*

The Leak Detective™ II electronic leak detector incorporates micro-chip technology and a new design, to give you better sensitivity and faster response time in a more compact unit. An auto-zero feature allows you to instantly zero the leak detector with a push of a button, and the ergonomic design brings all the controls to your fingertips for easy use. The unit responds in less than two seconds to trace leaks of gases with thermal conductivities different than air. Leaks are indicated by an audible alarm, as well as by an LED readout. For easy, sensitive, and reliable leak detection, order a new Leak Detective™ II electronic leak detector today.

Ordering Information | Leak Detective II™

Description	qty.	cat.#
Leak Detective™ II Leak Detector (9 volt, Battery-Operated)	ea.	20413

**Never use liquid leak detectors on a capillary system because liquids can be drawn into the column.*
Caution: *NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.*

Packed Column Technology Has New Life

With Restek Innovations

by Neil Mosesman, GC Columns Product Marketing Manager

- ✔ Optimized tubing and support ensure unsurpassed inertness for trace analyses.
- ✔ Bonded phases shorten conditioning times, greatly reduce bleed, and prolong column lifetimes.

For years column manufacturers have proclaimed that packed GC columns have gone the way of the dinosaurs. While packed columns do not fill every need, in some situations their use is dictated by methodology, and, for some analysts, they are the column of preference. Restek innovations have brought new life to the "expired" packed column in all three aspects of column technology: the support, the phase, and the tubing. Silcoport™ support provides unsurpassed inertness for trace analyses. Our bonded phase packings are a revolution in packed column technology. They significantly shorten conditioning times, greatly reduce column bleed, and prolong column lifetimes. SilcoSmooth™ tubing combines the inertness of glass with the durability of stainless steel.

The great sensitivity of modern detection systems and a progressing need to reduce detection limits place challenging demands on a chromatography column. Silcoport™ diatomaceous earth support is the modern solid support that we developed to meet these demands. Unlike conventional DMDCS deactivation, we use a proprietary fused silica deactivation technology and a special mixture of deactivants to ensure the greatest inertness (Figure 1) without changing the polarity of the stationary phase. Each batch of support is carefully tested to confirm a uniform particle size distribution that ensures columns with maximum efficiency.

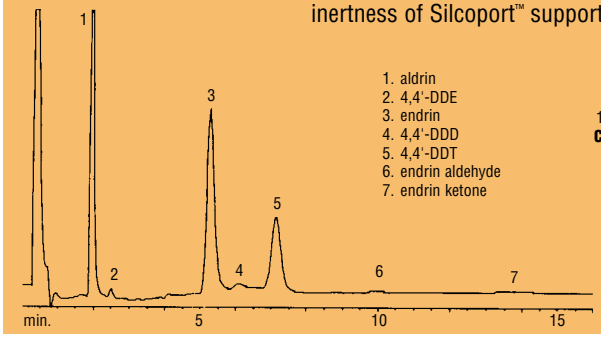
By applying our experience in stationary phase synthesis in conjunction with our unique Silcoport™ packing deactivation process we create completely bonded packing materials. To encompass a wide range of applications, we offer Rtx®-1 and Rtx®-5, bonded methyl silicone phases, and Stabilwax®, a bonded Carbowax® phase. Each phase is completely cross-linked on Silcoport™ support. In side-by-side comparisons with conventional nonbonded methyl silicone phase columns, Rtx®-1 and Rtx®-5 columns have lower bleed, improved peak shape, and longer useful lives (Figure 2). Evaluations with an Rtx®-1 column show retention times are repeatable after only 30 minutes of conditioning.

If your analysis involves reactive compounds, you probably have used fragile, inflexible glass columns, but now you can do better. Made from ultra-smooth, seamless 304 stainless steel, and treated with our innovative Silcosteel® deactivation process, SilcoSmooth™ tubing combines the inertness of glass with the strength and flexibility of stainless steel. SilcoSmooth™ tubing can replace glass tubing in virtually any application. For analyses of ppb levels of sulfur-containing compounds, use Sulfinert™ tubing packed with Rt-XLSulfur™ packing. For undemanding applications we can make columns from conventional tubing: stainless steel, Hastelloy®, nickel, copper, or Teflon®.

In combination, Silcoport™ support, our bonded phase packings, and SilcoSmooth™ tubing make packed column GC a viable alternative in many applications in which the technique had been endangered. If you use packed columns, and think that you have to live with limitations, call us. We can provide the column that will give you the separation you need, but with convenience, inertness, and column lifetimes you never expected from a packed column.

Figure 1

A trace level analysis of pesticides, including labile endrin and DDT, demonstrates the inertness of Silcoport™ support.



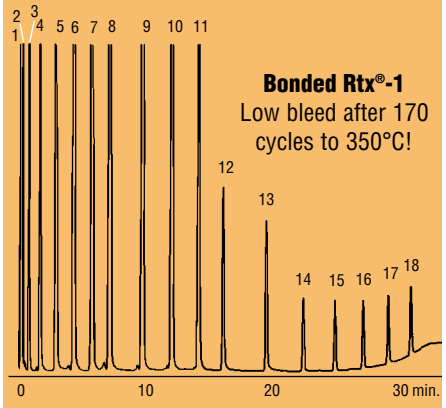
1. aldrin
2. 4,4'-DDE
3. endrin
4. 4,4'-DDD
5. 4,4'-DDT
6. endrin aldehyde
7. endrin ketone

1.5% Rt-608Pkd 2m, 2mm ID (cat.# 80221-)
Concentration: 50pg each analyte on-column
Oven temp.: 200°C isothermal
Inj. & det. temp.: 250°C
Det.: ECD
Column flow: nitrogen, 30cc/min.

GC_EV00370

Figure 2

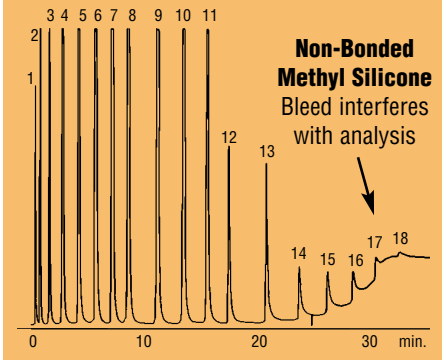
Bonded packed columns exhibit longer lifetimes than nonbonded columns.



Rtx®-1 SimDist 2887 25" x 1/8" Silcosteel® column (cat.# 80000) 1.0µL direct injection of 1-12% (w/w) each component cat.# 31647 (1% each listed analyte in CS_2) and cat.# 31675 (5% each, neat) meet requirements of ASTM D2887-01.
Oven temp.: 35°C to 350°C @ 10°C/min. (hold 5 min.)
Inj. & det. temp.: 350°C
Carrier gas: helium @ 25mL/min.
FID sensitivity: 256 x 10^{-11} AFS

1. C5
2. C6
3. C7
4. C8
5. C9
6. C10
7. C11
8. C12
9. C14
10. C16
11. C18
12. C20
13. C24
14. C28
15. C32
16. C36
17. C40
18. C44

GC_PC00369



Ordering Information | Bonded Packed Column Stationary Phases:

	Stainless Steel Tubing				SilcoSmooth™ Tubing			
Liquid Phases Bonded on 100/120 Silcoport™ P	**L (ft.)**	**OD (in.)**	**ID (mm)**	**cat.#***	**L (m)**	**OD (in.)**	**ID (mm)**	**cat.#***
3% Rtx®-1	6	1/8	2.1	80441-	2	1/8	2	80401-
10% Rtx®-1	6	1/8	2.1	80442-	2	1/8	2	80405-
20% Rtx®-1	6	1/8	2.1	80443-	2	1/8	2	80409-
3% Rtx®-5	6	1/8	2.1	80444-	2	1/8	2	80477-
10% Rtx®-5	6	1/8	2.1	80445-	2	1/8	2	80478-
20% Rtx®-5	6	1/8	2.1	80446-	2	1/8	2	80479-
5% Rtx®-Stabilwax®	6	1/8	2.1	80447-	2	1/8	2	80415-
10% Rtx®-Stabilwax®	6	1/8	2.1	80448-	2	1/8	2	80416-
20% Rtx®-Stabilwax®	6	1/8	2.1	80449-	2	1/8	2	80417-
Rtx®-1 SimDist 2887	25"	1/8	2.1	80450	25"	1/8	2	80000

*Please include configuration suffix number (refer to our catalog, lit. cat.# 59662).

Rt-Msieve™ 5A & MXT®-5A PLOT Columns

Superior Analyses of Permanent Gases

by Neil Mosesman, GC Columns Product Marketing Manager

✔ Fast, efficient separations at above ambient temperatures.
✔ 100% bonding process eliminates the need for particle traps.
✔ Stainless steel columns for durability.

Gas-liquid chromatography (GLC), the most common mode of gas chromatography, has limited application in analyses of gases. Subambient temperatures often are required to achieve a separation, and cryogenic cooling systems are costly and inconvenient. Gas-solid chromatography (GSC), in which gaseous analytes are absorbed into the packing particles, rather than into a surface coating, is far more effective for separating gases. Difficult-to-separate small molecules, such as argon and oxygen, butene isomers, and many others, can be separated by GSC at above ambient temperatures.

Just as capillary columns offer important advantages over packed GLC columns, porous layer open tubular columns—PLOT columns—offer significant advantages over packed GSC columns. Their open design gives PLOT columns greater permeability, and their narrow diameter ensures sharper peaks. The open construction also affords a smaller pressure drop per unit length, so longer columns can be used. This means much higher column efficiency and, therefore, superior resolution. In brief, PLOT columns provide faster and more sensitive analyses than packed GSC columns.

Restek PLOT columns are especially effective for separating mixtures of gaseous analytes. Rt-Msieve™ 5A and MXT®-Msieve 5A PLOT columns contain molecular sieve 5A particles that are bonded to the inner surface of the tubing, using a proprietary process that prevents particle dislocation that could damage valves and detection systems. They are designed for fast, efficient separation of argon and oxygen, hydrogen and helium, and other permanent gases, including permanent gases admixed in refinery or natural gas. Special coating and deactivation procedures ensure chromatographic efficiency and the integrity of the porous layer bonding. Finely controlled pore size allows selective adsorption of specific target compounds, ensuring difficult separations can be made without subambient temperatures. Figure 1 shows an Rt-Msieve™ 5A column can separate oxygen from argon to baseline, at above ambient temperature, in approximately 2 min. Figure 2 shows the permanent gases resolved from methane in 4 minutes.* Stainless steel MXT®-Msieve 5A PLOT columns offer the same powerful separating capabilities as fused silica Rt-Msieve™ 5A PLOT columns, plus high resistance to physical damage and ability to be coiled to diameters as small as 3.5" (<9cm), making MXT® columns ideal for portable GCs, process control applications, and other demanding situations.

In addition to Rt-Msieve™ 5A and MXT®-Msieve 5A columns, we manufacture PLOT columns for a wide range of other applications. Rt-Alumina™ PLOT columns (Al_2O_3 solid phase) offer fast, reproducible performance for determining hydrocarbon purity or monitoring hydrocarbon streams. Porous polymer Rt-QPLOT™ and MXT®-QPLOT columns (nonpolar), Rt-SPLOT™ and MXT®-SPLOT columns (intermediate polarity), and Rt-UPLOT™ and MXT®-UPLOT columns (highly polar) are particularly useful for situations in which water is likely to be encountered. Applications for these columns include permanent gases at subambient temperatures, carbon dioxide and other inorganic gases, hydrocarbon mixtures, and many nonpolar, intermediate polarity, and polar solvents. For more information and example analyses on Restek PLOT columns, refer to our current chromatography products catalog or our website, or request our new PLOT column flyer (lit. cat. #59456).

If your analyses call for difficult separations of gaseous analytes, and neither conventional packed GC columns nor WCOT capillary columns are providing the separations you want, or your analyses depend on costly or time-consuming conditions, a Restek PLOT column can make your work simpler.

Restek PLOT columns are superior for 5 reasons:

1. Most efficient and consistent analyses.
2. No need for particle traps.
3. Reproducible quality at affordable prices.
4. Most effective phase for your separation: alumina, molecular sieve 5A, or porous polymer.
5. Fused silica columns for most applications, metal columns for exceptional durability.

Ordering Information | Rt-Msieve™ 5A (Fused Silica PLOT) Temp. limit to 300°C

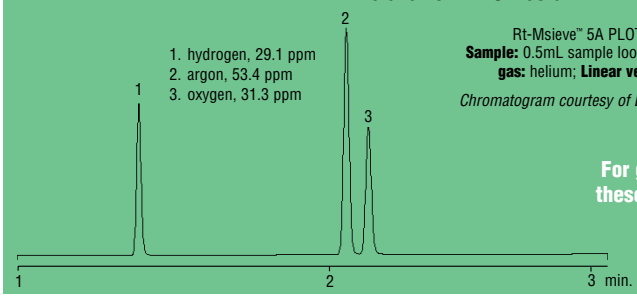
ID	df (µm)	15-Meter	30-Meter
0.32mm	30	19720	19722
0.53mm	50	19721	19723

Ordering Information | MXT®-Msieve 5A (Metal PLOT) Temp. limit to 300°C

ID	df (µm)	15-Meter	30-Meter
0.53mm	50	79721	79723

Figure 1

Oxygen and argon resolved to baseline at above ambient temperature, using an Rt-Msieve™ 5A PLOT column.



Rt-Msieve™ 5A PLOT 30m, 0.53mm ID (cat. # 19723)
Sample: 0.5mL sample loop injection; **Column temp.:** 27°C; **Carrier gas:** helium; **Linear velocity:** 34 cm/sec.; **Det.:** Valco HID

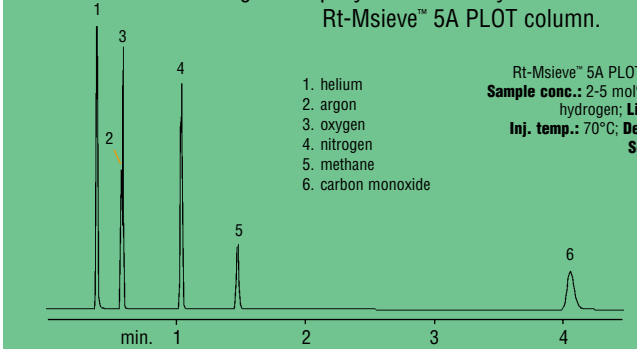
Chromatogram courtesy of Larry McElmurry, Mobile Analytical Labs.

For gas reference standards for these applications, see page 10.

GC_PC00170

Figure 2

Permanent gases rapidly and effectively resolved from methane by an Rt-Msieve™ 5A PLOT column.



Rt-Msieve™ 5A PLOT 15m, 0.32mm ID (cat. # 19720)
Sample conc.: 2-5 mol%; **Column temp.:** 70°C; **Carrier gas:** hydrogen; **Linear velocity:** 64cm/sec.; **Inj. temp.:** 70°C; **Det.:** HP µTCD; **Det. temp.:** 200°C ; **Sensitivity:** high

GC_PC00165

*Carbon dioxide is difficult to elute from a molecular sieve column, but is isolated easily on an Rt-QPLOT™ porous polymer column at 30°C. Request lit. cat.# 59540 for details.

HPLC Analysis of Narcotic/ Acetaminophen Admixtures

What to Do If a Compendium Method Doesn't Work

by Vernon Bartlett, HPLC Innovations Manager

✔ Make changes or modifications stepwise, with defined purpose in mind.
✔ When possible, create and validate a single method for a range of similar analytes.

Sometimes methods described in the United States Pharmacopoeia (USP), the European Pharmacopoeia (EP), the British Pharmacopoeia (BP), or other compendia do not provide the desired robustness in separation or reproducibility, or results barely pass system suitability requirements. Modifications can be made to improve the methodology, and the results compared statistically to the original. To improve analysis efficiency and reduce costs associated with revalidating and testing, it may be desirable to create and validate a single analytical method for a range of similar drug products.

Many narcotics are very similar in structure, often varying by only a single substitution. Morphine, codeine, hydrocodone, and oxycodone are quite similar, for example (Figure 1). Some of these closely related compounds—all but morphine, in fact—might be blended with other analgesics, such as acetaminophen (APAP). USP 25 describes more than 7 different methods to test these raw materials and admixtures; some of the older methods do not use HPLC as a primary test for purity.

Figure 1
Chemical structures of narcotics and acetaminophen.

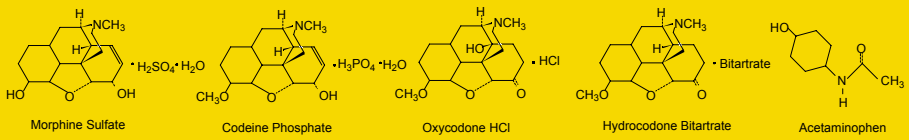


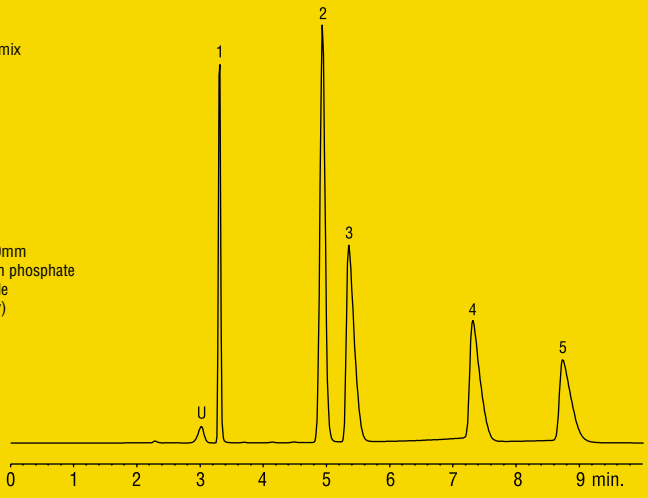
Figure 2
A single, modified USP procedure for separating structurally similar narcotic analgesics and acetaminophen on an Ultra C18 column.

Peak	Conc. (µg/mL)	Ret. Time (min.)	Tailing	Resolution
U. unknown	unknown	3.0	NA	NA
1. morphine sulfate	204	3.3	0.97	2.3
2. acetaminophen	92	5.0	1.1	14.9
3. codeine phosphate	216	5.3	1.8	2.1
4. oxycodone HCl	206	7.3	1.9	6.9
5. hydrocodone bitartrate	218	8.8	1.9	4.1

Sample:
Inj.: 10µL
Sample: raw material mix
Solvent: mobile phase

Column: **Ultra C18**
Catalog #: 9174575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile Phase: A: pH 2.8 10mm potassium phosphate
B: acetonitrile
(85A:15B, v/v)
Flow: 1.0 mL/min.
Temp.: 27°C
Det.: UV @ 235nm



LC_0219

One of the chromatographic applications in USP 25 is for the analysis of oxycodone raw material. After reading the mobile phase section, we saw some potential problems with the method, including:

1) The use of methanol in this analysis could lead to high background absorption and loss of linear range, because the analytical wavelength is 206nm, and the UV cutoff for methanol is 235nm. In extreme cases this also can reduce sensitivity—the more energy the background absorbs, the less is available to the analyte.

2) An ion-pairing agent (hexane sulfonic acid) is introduced into the mobile phase without a buffer to maintain pH. This could lead to widened peaks, tailing peaks, and retention time drift.

3) Triethylamine (TEA) modifier is included in the method. When basic compounds are analyzed on older-type HPLC columns, TEA often is added as competing base, to reduce the tailing caused by acidic silanol activity. If the analytical species are neutral, or have been "neutralized" by an ion-pairing agent, TEA should have no beneficial effect. Adding TEA, a base, to a mobile phase containing sulfonic acids will cause acid/base neutralization, producing a salt and water and reducing the effective concentration of the acidic ion-pairing agent. This could lead to the formation of undesirable side products in the mobile phase that also will absorb in the low UV range, creating noisy baselines. Furthermore, TEA is volatile, and its composition might change over time if the mobile phase is sparged.

Thus, some aspects of the method appear redundant and some might actually compromise the separation. In addition, some of the reagents, such as TEA, might not be necessary for modern columns. After performing the USP 25 method as written, we made some tests to determine actual needs to achieve the system suitability requirements as specified.

With peak shape, separation, and proper analytical technique in mind, we attempted to eliminate some of the perceived problems. We realized that by using 284nm as the detection wavelength, rather than 206nm as used in USP 25, we might not see some impurities, but in real life the material should be tested against some known source for potency. (Note that with the additional reagents removed, both Ultra C8 and Pinnacle II™ C8 columns provided good results at the 206nm wavelength.)

Next we removed the ion pairing agent and the TEA. We elected to keep a 20 mM phosphate buffer system to maintain a pH of 2.5. Then we reduced the temperature from 35°C to 27°C, to determine whether the greater mass transfer and analyte solubility in the mobile phase at 35°C had been masking other potential problems.

These changes led to a slight increase in tailing for all compounds on both Ultra C8 and Pinnacle II™ C8 columns, but this was acceptable, especially because the run time for the analysis was reduced by a factor of 3 and resolution was improved by 59% to 79%. The system passed the system suitability requirements in the USP monograph.

In the next experiment, we re-introduced the ion pair reagent hexane sulfonic acid into the system under the control of the pH 2.5 phosphate buffer system. The run time doubled, relative to the original procedure, demonstrating that TEA did affect the concentration of the ion-pairing agent. Reducing the concentration of ion pairing agent, or using a shorter chain length ion-pairing agent, might have been a better alternative to adding TEA. The system still passed the system suitability requirements listed by the USP, but the chromatogram was much noisier—and equilibration problems seen in the USP 25 analysis returned.

Figure 3

A small amount of methanol finely tunes the separation of narcotic analgesics and acetaminophen on an Ultra C18 column, as indicated by less peak tailing.

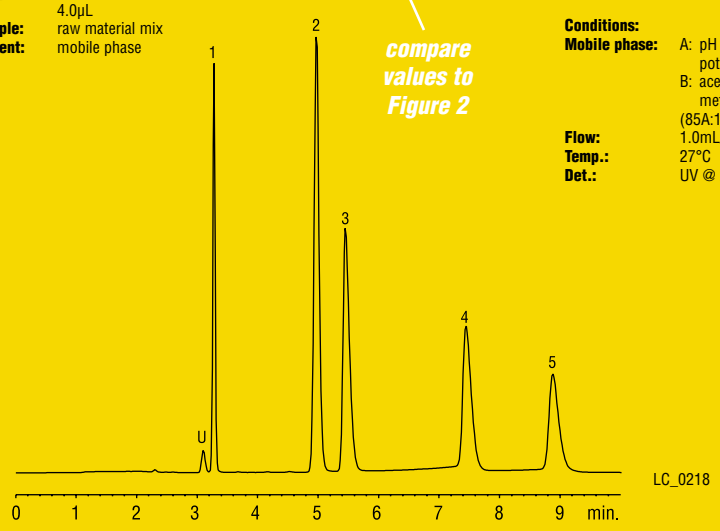
Peak List:	Conc. (µg/mL)	Ret. Time (min.)	Tailing	Resolution
U. unknown	unknown	3.1	NA	NA
1. morphine sulfate	204	3.3	1.0	1.8
2. acetaminophen	92	5.0	1.1	14.1
3. codeine phosphate	216	5.5	1.4	2.6
4. oxycodone HCl	206	7.5	1.4	8.5
5. hydrocodone bitartrate	218	8.9	1.4	5.0

Sample:
Inj.: 4.0µL
Sample: raw material mix
Solvent: mobile phase

compare values to Figure 2

Column: **Ultra C18**
Catalog #: 9174575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: A: pH 2.8 10mm potassium phosphate
B: acetonitrile: methanol (90:10 v/v)
(85A:15B, v/v)
Flow: 1.0mL/min.
Temp.: 27°C
Det.: UV @ 235nm



Ordering Information | Ultra C18 5µm Columns

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.0mm ID cat.#	4.6mm ID cat.#
30mm	9174531	9174532	9174533	—	9174535
50mm	9174551	9174552	9174553	—	9174555
100mm	9174511	9174512	9174513	9174514	9174515
150mm	9174561	9174562	9174563	9174564	9174565
200mm	9174521	9174522	9174523	—	9174525
250mm	9174571	9174572	9174573	—	9174575

After reviewing the monographs for admixtures containing structurally related narcotics and acetaminophen, we created a single separation for morphine sulfate, acetaminophen, codeine phosphate, oxycodone HCl, and hydrocodone bitartrate. The goal was to create an adequate separation while keeping the method as simple as possible. We chose an Ultra C18 column and set detection to 235nm. All components, including a small unknown peak, were separated to baseline (Figure 2).

Next, we increased the amount of buffer to 90% (a 5% increase). This simple increase doubled the analysis time. Resolution doubled between most components, with the greatest change between acetaminophen and codeine. The unknown peak disappeared and probably co-eluted with morphine.

We adjusted the mobile phase ratio to 85:15, buffer:organic solvent, using a 90:10 mixture of acetonitrile and methanol as the organic solvent. Resolution improved, relative to the original mobile phase composition, analysis again was under 10 minutes, and the unknown peak returned (Figure 3). For this analysis, these conditions provided the most desirable results.

In summary, the goal of any method should be to achieve the most stable and robust separation. Sometimes methods are more complicated than they need to be, and this can make analysis unnecessarily difficult. Even troubleshooting such methods adds to production costs. When preparing to follow a method always attempt to determine the reason a reagent would be included in a mobile phase. Any change or modification should have an established scientific purpose. By creating more universal methods for analyses of structurally related compounds, it should be possible to reduce costs for supplies, increase laboratory analysis efficiency, and reduce personnel training time.

For chromatograms illustrating the changes in separation that occur with each change in the mobile phase, please request Applications Note #59453. If you encounter problems when analyzing your samples according to an established method, our experienced Technical Service chemists will be glad to help. Contact them at 800-356-1688, ext. 4 or 814-353-1300, ext. 4, or contact your Restek representative.

More Reliable Results From Semivolatiles Analysis

Using Restek Columns and Standards

by Christopher English, Environmental Innovations Chemist

✔ Rtx®-5Sil MS columns resolve critical pairs and minimize bleed.
✔ Integral guard column available.
✔ 8270MegaMix™ minimizes mixtures needed, has maximum stability.
✔ Monitor all relevant semivolatiles at one detector sensitivity—8270 MegaMix™ includes 3 and 4 methylphenol at 0.5x concentrations of other components.

Complex mixtures of semivolatile organic compounds are extracted from water, soil, or solid waste samples, concentrated, and analyzed by gas chromatography. The current compound list for US EPA Method 8270D, for example, includes basic, neutral, and acidic compounds with boiling points from 150°C to 500°C. Other semivolatiles methods are similarly complex. Because these analyses encompass a broad range of compound classes and require low detection limits, and because sample extracts can include non-target contaminants, significant demand is placed on the efficiency, inertness, thermal stability, and sample capacity of the analytical column. These parameters must be optimized to provide good resolution, fast analysis times, and high sample throughput. The column must have adequate sample capacity to handle the high concentrations of contaminants sometimes found in these extracts, while exhibiting the high inertness needed for accurate quantification of target analytes down to low ng/µL levels.

Restek has designed Rtx®-5Sil MS capillary columns to address the demands of semivolatile by GC/MS. Silarylene polymer technology stiffens the siloxane chain, preventing its thermal breakdown (column bleed). The content of this aryl functionality has been adjusted to give excellent efficiency and lower bleed, compared to conventional 5% diphenyl/95%dimethyl phases; Rtx®-5Sil MS columns exhibit excellent inertness and low bleed, even at 330°C. The optimized stationary phase, proprietary deactivations, and inherently low bleed of the Rtx®-5Sil MS phase, combined with the integral guard column, overcome the problems presented by the compounds and conditions inherent to semivolatiles analysis. High column efficiency ensures the resolution needed to quantify critical pairs and structural isomers, as shown by the separation of benzo(b)- and benzo(k)fluoranthene (peaks 83/84) in Figure 1.

Rtx®-5Sil MS columns are available with an integral, deactivated 5- or 10-meter Integra-Guard™ guard column that prevents non-volatile residues from collecting in the analytical column, where they could interfere with the analytes. Made from a continuous length of tubing, innovative Integra-Guard™ columns offer the column-protecting advantages of a guard column without the potential for leaks at the interface.*

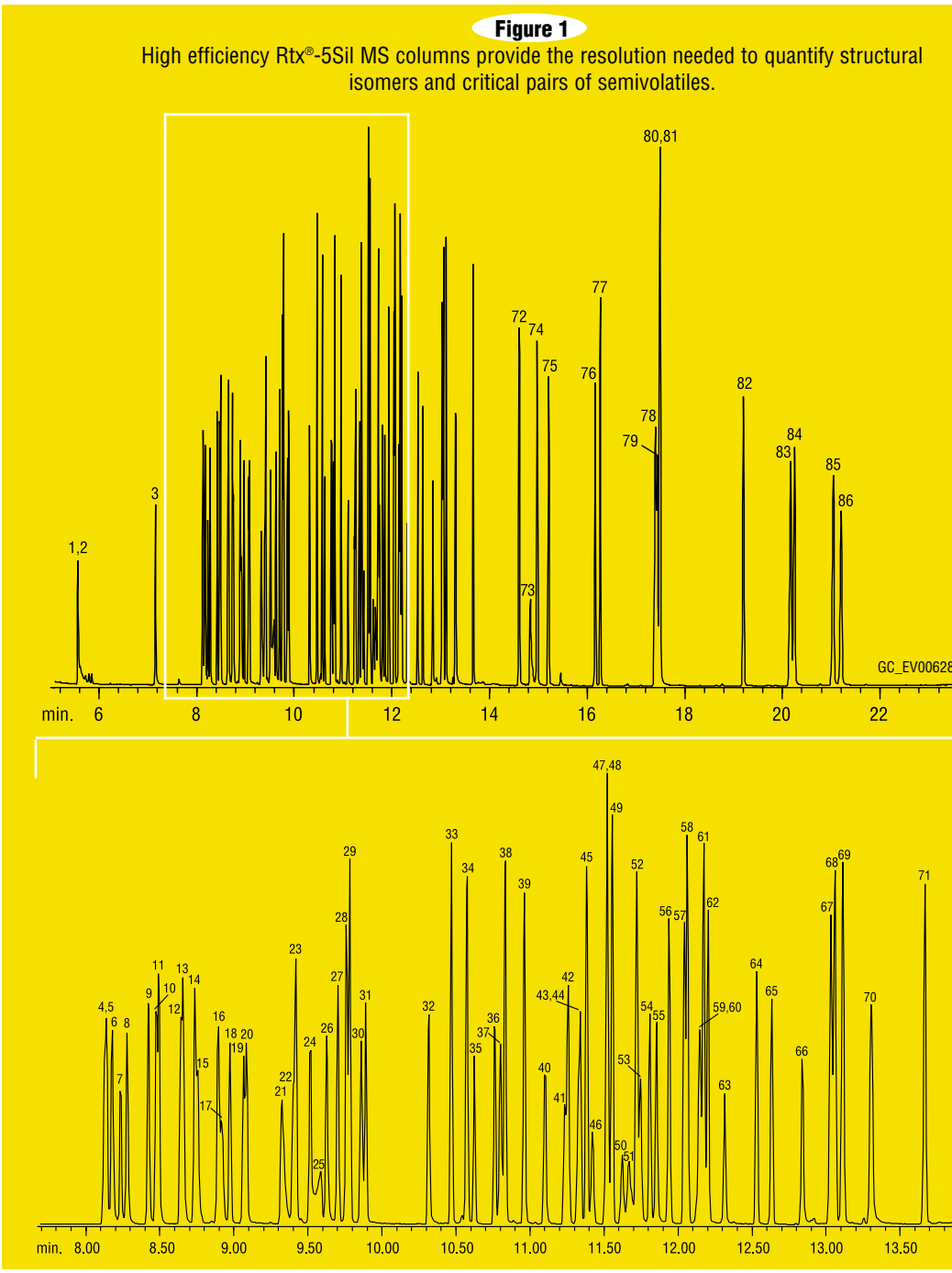
*For more information about Integra-Guard™ columns, request lit. cat.# 59441.

Our 8270 MegaMix™ eliminates mixing and minimizes preparation time for calibration and laboratory control samples—it combines all current target analytes in EPA Method 8270D. 8270 MegaMix™ components are indicated in bold in the list of analytes in Figure 1. A unique feature of this mix is the inclusion of 3-methyl- and 4-methylphenol at 0.5x the concentration of the other components, so you won't have to adjust reporting limits when analyzing for these compounds. A long shelf life for unopened ampuls of 8270 MegaMix™ minimizes ordering and inventory problems.

If you are monitoring semivolatile analytes according to US EPA Method 8270D, or similar methods, trust Restek Rtx®-5Sil MS columns and 8270 MegaMix™ to help you obtain reliable, consistent results.

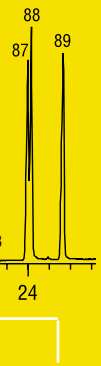
Figure 1

High efficiency Rtx®-5Sil MS columns provide the resolution needed to quantify structural isomers and critical pairs of semivolatiles.



1. **N-nitrosodimethylamine**
2. **pyridine**
3. 2-fluorophenol
4. phenol-d6
5. **phenol**
6. **aniline**
7. **bis(2-chloroethyl)ether**
8. **2-chlorophenol**
9. **1,3-dichlorobenzene**
10. 1,4-dichlorobenzene-d4
11. **1,4-dichlorobenzene**
12. **benzyl alcohol**
13. **1,2-dichlorobenzene**
14. **2-methylphenol**
15. **bis(2-chloroisopropyl)ether**
16. **4-methylphenol/ 3-methylphenol***
17. **N-nitroso-di-n-propylamine**
18. **hexachloroethane**
19. nitrobenzene-d5
20. **nitrobenzene**
21. **isophorone**
22. **2-nitrophenol**
23. **2,4-dimethylphenol**
24. **bis(2-chloroethoxy)methane**
25. benzoic acid
26. **2,4-dichlorophenol**
27. **1,2,4-trichlorobenzene**
28. naphthalene-d8
29. **naphthalene**
30. **4-chloroanaline**
31. **hexachlorobutadiene**
32. **4-chloro-3-methylphenol**
33. **2-methylnaphthalene**
34. **1-methylnaphthalene**
35. **hexachlorocyclopenta-diene**
36. **2,4,6-trichlorophenol**
37. **2,4,5-trichlorophenol**
38. 2-fluorobiphenyl
39. **2-chloronaphthalene**
40. **2-nitroaniline**
41. **1,4-dinitrobenzene**
42. **dimethylphthalate**
43. **1,3-dinitrobenzene**
44. **2,6-dinitrotoluene**
45. **acenaphthylene**
46. **1,2-dinitrobenzene**
47. **3-nitroaniline**
48. acenaphthene-d10
49. **acenaphthene**
50. **2,4-dinitrophenol**
51. **4-nitrophenol**
52. **dibenzofuran**
53. **2,4-dinitrotoluene**
54. **2,3,4,6-tetrachlorophenol**
55. **2,3,5,6-tetrachlorophenol**
56. **diethyl phthalate**
57. **4-chlorophenyl phenyl ether**
58. **fluorene**
59. **4-nitroaniline**
60. **4,6-dinitro-2-methylphenol**
61. **diphenylamine****
62. **azobenzene*****
63. 2,4,6-tribromophenol
64. **4-bromophenyl phenyl ether**
65. **hexachlorobenzene**
66. **pentachlorophenol**
67. phenanthrene-d10
68. **phenanthrene**
69. **anthracene**
70. **carbazole**
71. **di-*n*-butylphthalate**
72. **fluoranthene**
73. benzidine
74. **pyrene**
75. *p*-terphenyl-d14
76. **butyl benzyl phthalate**
77. **bis(2-ethylhexyl)adipate**
78. **benzo(a)anthracene**
79. chrysene-d12
80. **chrysene**
81. **bis(2-ethylhexyl)phthalate**
82. **di-*n*-octyl phthalate**
83. **benzo(b)fluoranthene**
84. **benzo(k)fluoranthene**
85. **benzo(a)pyrene**
86. perylene-d12
87. **indeno(1,2,3-cd)pyrene**
88. **dibenzo(a,h)anthracene**
89. **benzo(ghi)perylene**

Bold indicates a component of the 8270D MegaMix™



Column:	Rtx®-5Sil MS w/ 5-meter Integra-Guard™ 30m, 0.25mm ID, 0.25µm (cat.# 12723-124)
Instrument:	Agilent 5973 GC/MS
Sample:	US EPA Method 8270D Mix 1µL, 16 ppm each component (16ng on column) 8270 MegaMix™ (cat.# 31686) Benzoic Acid Standard (cat.# 31415) Benzidine Standard (cat.# 31441) Acid Surrogate Mix (4/89 SOW)(cat.# 31063) B/N Surrogate Standard Mix (4/89 SOW) (cat.# 31062) SV Internal Standard Mix (cat.# 31006)
Solvent:	dichloromethane
Inj.:	1.0µL splitless (hold 0.3 min.), 4mm Drilled Uniliner® (cat.# 21055)
Inj. temp.:	300°C
Carrier gas:	helium, constant flow
Flow rate:	1.1mL/min.
Dead Time:	1.8 minutes @ 35°C
Oven temp.:	35°C (hold 4 min.) to 245°C @ 25°C/min. (no hold) to 330°C @ 6°C/min.(hold 3 min.)
Det:	GC/MS
Transfer line temp.:	280°C
Scan range:	35–550 amu
Solvent Delay:	5 min.
Tune:	DFTPP
Ionization:	EI

**Each at 0.5x concentration of other components.*

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

****1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).*

Ordering Information | Rtx®-5Sil MS Columns (Fused Silica)

(Equivalent selectivity of Crossbond® 5% diphenyl/95% dimethyl polysiloxane) Stable to 360°C

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

8270 Matrix Spike Mix (76 components)

See **bold** compounds in Figure 1 peak list.

200µg/mL each (except noted) in methanol:methylene chloride:benzene (80:15:5), 5mL/ampul

Each	5-pk.	10-pk.
31687	31687-510	—
	with data pack	
31687-500	31687-520	31787

8270 MegaMix™ (76 components)

See **bold** compounds in Figure 1 peak list.

1,000µg/mL each (except noted) in methylene chloride:benzene (75:25), 1mL/ampul

Each	5-pk.	10-pk.
31686	31686-510	—
	with data pack	
31686-500	31686-520	31786

New Analytical Reference Materials

FAMEs, Acetates, BTEX, Glycols

Food Industry FAME Mix

(37 components)

- Includes *trans* FAMEs.

Chain	% by Weight
C4:0	4.0
C6:0	4.0
C8:0	4.0
C10:0	4.0
C11:0	2.0
C12:0	4.0
C13:	2.0
C14:0	4.0
C14:1(*cis*-9)	2.0
C15:0	2.0
C15:1(*cis*-10)	2.0
C16:0	6.0
C16;1(*cis*-9)	2.0
C17:0	2.0
C17:1(*cis*-10)	2.0
C18:0	4.0
C18:1(*trans*-9)	2.0
C18:1(*cis*-9)	4.0
C18:2(all-*trans*-9,12)	2.0
C18:2(all-*cis*-9,12)	2.0
C18:3(all-*cis* 6,9,12)	2.0
C18:3(all-*cis* 9,12,15)	2.0
C20:0	4.0
C20:1(*cis*-11)	2.0
C20:2(all-*cis* 11,14)	2.0
C20:3 (all-*cis* 8,11,14)	2.0
C20:3(all-*cis* 11,14,17)	2.0
C20:4(all-*cis* 5,8,11,14)	2.0
C20:5(all-*cis* 5,8,11,14,17)	2.0
C21:0	2.0
C22:0	4.0
C22:1(*cis* 13)	2.0
C22:2(all-*cis* 13,16)	2.0
22:6(all-*cis* 4,7,10,13,16,19)	2.0
C23:0	2.0
C24:0	4.0
C24:1(*cis*-15)	2.0

30mg/mL in methylene chloride, 1mL/ampul

ea.
35077

8260B Acetate Mix (7 components)

- Includes methyl acetate and n-amyl acetate.

n-amyl acetate
butyl acetate
ethyl acetate
isopropyl acetate
methyl acetate
propyl acetate
vinyl acetate

2,000µg/mL each in P&T methanol, 1mL/ampul

	Each	5-pk.	10-pk.
	30489	30489-510	
w/data pack	30489-500	30489-520	30589

BTEX Standard (6 components)

- m- and p-xylene at ½ concentration.
- Contact Restek for future formulations.

benzene
ethylbenzene
toluene
m-xylene*
o-xylene
p-xylene*

2,000µg/mL each in P&T methanol, 1mL/ampul

	Each	5-pk.	10-pk.
	30488	30488-510	
w/data pack	30488-500	30488-520	30588

*1,000µg/mL

Glycols Standard

- Assay for de-icing compounds.

ethylene glycol
propylene glycol

50,000µg/mL each in DI water, 1mL/ampul

	Each	5-pk.	10-pk.
	30471	30471-510	
w/data pack	30471-500	30471-520	30571

Scotty® Transportables

For Laboratory or Field Application

by Donna Lidgett, Air Monitoring Product Marketing Manager

new

- ✔ Portability makes your job easier.
- ✔ 4-Liter, 14-liter, and 48-liter sizes.
- ✔ Long shelf life.

Restek now offers a broad selection of Scotty® Transportables, ranging from pure gases to multi-component mixes. These standards have found many uses in the analytical lab, including purging, calibrating, and peak identification.

We offer three sizes to choose from. The 4-liter container has a delivery tube for purge gas or connection to sample loop or bag. The 14-liter container has a CGA 160 fitting for connection to an analytical system. The 48-liter cylinder has a CGA 165 connection, and can deliver large volumes of sample.

Regulators for Scott Transportable Gases

Specifications
Maximum Inlet Pressure: 300psig
Outlet Pressure Range: 2–10psig
Operating Temperature Range: 35°F to 150°F (2°C to 65°C)
Outlet Connection: ¼" Female NPT

Materials of Construction
Body: Brass
Diaphragm: Viton®
Seat: Acetal
Seal: Viton®

Description	qty.	cat.#	price
Regulator with CGA 160 inlet connection for 14L Scott container	ea.	22690	$110
Regulator with CGA 165 inlet connection for 48L Scott container	ea.	22691	$110

Description	Shelf Life	Scotty 4 (4 Liter) cat.#	Scotty 14 (14 Liter) cat.#	Scotty 48 (48 Liter) cat.#
Pure Gases				
Air, zero (THC <1ppm)	2 yrs.	34447	34448	34449
Argon, 99.995%	2 yrs.	34456	34457	—
Carbon dioxide, 99.80%	2 yrs.	34450	34451	34452
Hydrogen, 99.99%	2 yrs.	—	34453	—
Methane, 99.00%	2 yrs.	—	34454	—
Oxygen, 99.60%	2 yrs.	—	34455	—
Two-Component Mixtures				
Benzene in air (1ppm)	1 yr.	—	—	34458
Benzene in air (100ppm)	1 yr.	—	—	34459
1,3-Butadiene in nitrogen (10ppm)	2 yrs.	—	34460	34461
Carbon dioxide in helium (100ppm)	2 yrs.	—	34462	—
Carbon dioxide in nitrogen (100ppm)	2 yrs.	—	34463	34464
Carbon dioxide in nitrogen (1000ppm)	2 yrs.	—	34465	34466
Ethylene in air (8-10ppm)	2 yrs.	—	34467	34468
Ethylene in helium (100ppm)	2 yrs.	—	34489	—
Hydrogen in helium (100ppm)	2 yrs.	—	34469	—
Hydrogen in nitrogen (1%)	2 yrs.	34470	34471	34472
Hydrogen in nitrogen (100ppm)	2 yrs.	—	34473	34474
Methane in helium (100ppm)	2 yrs.	34475	34476	34477
Methane in nitrogen (100ppm)	2 yrs.	—	34478	—
Methane in nitrogen (1%)	2 yrs.	34481	34482	34483
Nitrogen in helium (100ppm)	2 yrs.	—	34479	—
Nitrous oxide in nitrogen (1ppm)	2 yrs.	—	34484	34485
Oxygen in helium (100ppm)	2 yrs.	—	34480	—
Oxygen in nitrogen (2%)	2 yrs.	34486	34487	34488
Oxygen in nitrogen (6%)	2 yrs.	34490	34491	34492
1,1,1-Trichloroethane in nitrogen (10ppm)	2 yrs.	—	—	34493
Trichloroethylene in nitrogen (10ppm)	2 yrs.	—	34494	34495
Vinyl chloride in nitrogen (1ppm)	2 yrs.	—	34496	34497
Vinyl chloride in nitrogen (10ppm)	2 yrs.	—	34498	34499
Vinyl chloride in nitrogen (50ppm)	2 yrs.	—	34500	—
Vinyl chloride in nitrogen (100ppm)	2 yrs.	—	34501	—
Vinyl chloride in nitrogen (1000ppm)	2 yrs.	—	34502	—
Multi-Component Mixtures				
Carbon monoxide, carbon dioxide, hydrogen and oxygen in nitrogen (0.5% each)	2 yrs.	34503	34504	34505
Carbon monoxide, carbon dioxide, hydrogen and oxygen in nitrogen (1% each)	2 yrs.	34506	34507	34508
Carbon monoxide, carbon dioxide, methane, ethane, ethylene and acetylene in nitrogen (1% each)	1 yr.	34509	34510	34511
Carbon monoxide, carbon dioxide, nitrogen, and oxygen, (5% each) and methane and hydrogen (4% each) in helium	2 yrs.	—	34512	—
Carbon monoxide (7%), carbon dioxide (15%) and oxygen (5%) in nitrogen	2 yrs.	34513	34514	—
Carbon monoxide (7%), oxygen (7%), carbon dioxide (15%) and methane (4.5%) in nitrogen	2 yrs.	—	34515	34516
C1-C6 *n*-Paraffins: methane, ethane, propane, butane, pentane, hexane in nitrogen (15ppm each)	2 yrs.	34517	34518	34519
C1-C6 *n*-Paraffins: methane, ethane, propane, butane, pentane, hexane in helium (100ppm each)	2 yrs.	34520	34521	34522
C1-C6 *n*-Paraffins: methane, ethane, propane, butane, pentane, hexane in helium (1000ppm each)	2 yrs.	34523	34524	34525
C1-C6 *n*-Paraffins: methane, ethane, propane, butane, pentane, hexane in nitrogen (100ppm each)	2 yrs.	34526	34527	34528
C2-C4 Alkynes: acetylene, propylene, 1-butylene, 2-butylene in nitrogen (15ppm each)	2 yrs.	34535	—	—
C2-C6 Olefins: ethylene, propylene, 1-butene, 1-pentene, 1-hexene in helium (100ppm each)	2 yrs.	—	34529	34530
C2-C6 Olefins: ethylene, propylene, 1-butene, 1-pentene, 1-hexene in nitrogen (100ppm each)	2 yrs.	—	34531	34532
Branched Paraffins: 2,2-dimethylbutane, 2,2-dimethylpropane, iso-butane, 2-methylbutane, 2-methylpentane, 3-methylpentane in nitrogen (15ppm each)	2 yrs.	34533	34534	—
Methane, ethane, ethylene, acetylene, propane, propylene, *n*-butane in nitrogen (15ppm each)	1 yr.	34536	—	34537
n-butane, iso-butane, *cis*-2-butene, *trans*-2-butene, 1-butene, iso-butylene, 1,3-butadiene, ethyl acetylene in nitrogen (15ppm each)	1 yr.	34538	—	34539

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by Ken Herwehe, Analytical Reference Materials Marketing Manager

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Ordering Information | ChemService Pesticides (popular examples)

Pesticide	CAS No.	qty.	cat.#
Acetochlor	34256-82-1	100mg	PS-2040
Aldrin®	309-00-2	100mg	PS-69
Aramite (5ml at 0.1mg/ml in hexane)	140-57-8	5ml	PS-850
Aspon®	3244-90-4	1g	PS-663
Atrazine	1912-24-9	1g	PS-380
BHC mixed isomers	608-73-1	1g	PS-70
Bladex	21725-46-2	1g	PS-387
Buprofezin	69327-76-0	100mg	PS-2067
Carbaryl	63-25-2	1g	PS-84
Chlormephos	24934-91-6	250mg	PS-2209
Chloroxynil	1891-95-8	500mg	PS-2090
Chlorpyrifos	2921-88-2	1g	PS-674
Crotoxyphos	7700-17-6	50mg	PS-603
o,p'-DDT	789-02-6	50mg	PS-698
Demeton S	126-75-0	100mg	PS-662
Diazinon	333-41-5	1g	PS-90
Dicamba	1918-00-9	1g	PS-346
Dichlorprop	120-36-5	1g	PS-44
Dieldrin	60-57-1	250mg	PS-76
Dikegulac acid	18467-77-1	250mg	PS-2190
Dimehypo	52207-48-4	250mg	PS-2184
Dioxathion	78-34-2	100mg	PS-658
Fenchlorphos	299-84-3	100mg	PS-657
Flutriafol	76674-21-0	100mg	PS-2177
tau-Fluvalinate	102851-06-9	100mg	PS-1071
Gibberellic acid	77-06-5	100mg	PS-49
Glyphosate	1071-83-6	1g	PS-1051
Heptachlor	76-44-8	100mg	PS-78
trans-Heptachlor epoxide	28044-83-9	50mg	PS-700-1
Imazamethabenz-methyl	81405-85-8	100mg	PS-2195
Isopropyl-4,4'-dichloro-benzilate	5836-10-2	1g	PS-857
2-Isovaleryl-1,3-indanedione	83-28-3	250mg	PS-911
Lindane	58-89-9	1g	PS-71
Malathion	121-75-5	1g	PS-86
Metasystox® (i)	919-86-8	50mg	PS-1096
Methamidophos	10265-92-6	100mg	PS-676
Metribuzin	21087-64-9	1g	PS-398
Metsulfuron methyl	74223-64-6	100mg	PS-1078
cis-Mevinphos	26718-65-0	100mg	PS-87-1
Monolinuron	1746-81-2	250mg	PS-2210
Parathion®	56-38-2	1g	PS-95
Phenmedipham	13684-63-4	250mg	PS-1014
Phosalone	2310-17-0	1g	PS-682
Pirimicarb	23103-98-2	1g	PS-757
Pirimiphos-methyl	29232-93-7	1g	PS-644
Sulfosulfuron	141776-32-1	500mg	PS-2224
Tebufenozide	112410-23-8	100mg	PS-2188
Terbutol	1918-11-2	100mg	PS-550
2,3,5-Trimethylphenyl methyl carbamate	2655-15-4	1g	PS-541
Vinclozolin	50471-44-8	1g	PS-1049

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Enhanced Retention of Polar Analytes by HPLC

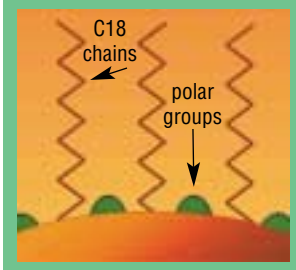
Using the New Ultra Aqueous C18 Column

by Terrence Reid, HPLC Applications Team Chemist

✔ Excellent peak shape for basic analytes.
✔ Compatible with 100% aqueous to 100% organic mobile phases.
✔ Compatible with MS detection.

The newest addition to our selection of HPLC columns, the Ultra Aqueous C18 column, is designed to enhance the retention of polar compounds by reversed phase HPLC. The Ultra Aqueous C18 stationary phase is a true C18 chain (USP L1), but it is immobilized on the silica surface through a unique chemistry that creates polar groups on the silica surface, between the C18 chains (Figure 1). This secondary polar character has several benefits. First, polar analytes that are insufficiently retained on a conventional C18 column interact with the polar groups in an Ultra Aqueous C18 column, producing enhanced retention. Second, the polar groups aid the retention of polar compounds by keeping the stationary phase completely wetted, even in 100% aqueous mobile phases. In theory, eliminating organic solvent from the mobile phase should maximize retention in reversed phase HPLC, and this is true for Ultra Aqueous C18 columns. In contrast, many conventional C18 columns lose ability to retain analytes in highly aqueous mobile phases because the C18 chains self-associate or fold down on the silica (Figure 1), a phenomenon sometimes referred to as chain folding. Third, the polar groups on the Ultra Aqueous C18 stationary phase shield analytes from active silanol sites on the silica surface, ensuring excellent peak shape for basic analytes (Figure 2).

Figure 1
An Ultra Aqueous C18 column is compatible with mobile phases from 100% organic content to 100% aqueous.



Ultra Aqueous C18 phase is stable in highly organic or highly aqueous mobile phase

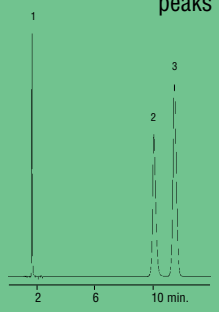
Conventional C18 chains collapse and lose capacity for retention in highly aqueous mobile phases

Although they were designed to be used with highly aqueous mobile phases, Ultra Aqueous C18 columns also are completely compatible with highly organic mobile phases. The ability to cover the full range of mobile phase composition, from 100% aqueous to 100% organic, is useful for developing gradient methods for analyzing samples containing both highly polar and nonpolar analytes. For example, Figure 3 shows water-soluble vitamins are eluted from an Ultra Aqueous C18 column with excellent resolution and as sharp, symmetric peaks.

Ultra Aqueous C18 columns also are compatible with MS detection-the minimal noise generated by an Ultra Aqueous C18 column is comparable to background in a blank analysis with no column in line (Figure 4). LC/MS is the analytical approach for a steadily increasing variety of analytes, and the versatile Ultra Aqueous C18 column is an obvious column choice.

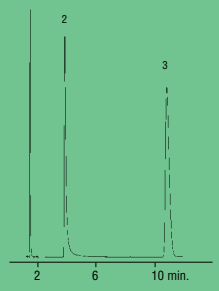
If you are analyzing samples containing polar analytes, or mixtures of polar and nonpolar analytes, and are contending with unsatisfactory resolution, insufficient retention of polar compounds, poorly shaped peaks, and/or complicated mobile phases, an Ultra Aqueous C18 column can be the solution to your problems.

Figure 2
Silanol-shielding polar groups on the Ultra Aqueous C18 stationary phase ensure symmetric peaks for basic analytes, compared to other C18 phases.



1. uracil 5.0µg/mL
2. pyridine 0.1µL/mL
3. phenol 1.86mg/mL

Sample: 5µL
Mobile Phase: 20mM potassium phosphate pH 7.0: acetonitrile (80:20)
Flow: 1.0mL/min.
Temp: 25°C
Det.: UV @ 254nm

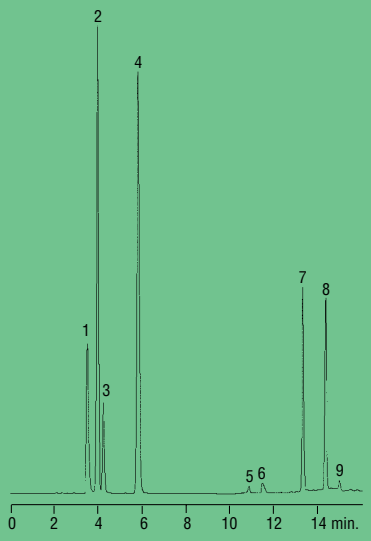


Column: Ultra Aqueous C18 (cat.# 9178565)
Dimensions: 150 x 4.6mm; **Particle Size:** 5µm
Pore Size: 100Å

LC_0143

Column: Base-deactivated C18
Dimensions: 150 x 4.6mm; **Particle Size:** 5µm
Pore Size: 100Å

Figure 3
Excellent resolution and peak shapes for water-soluble vitamins eluted from an Ultra Aqueous C18 column.



Peak	Conc. (mg/mL)
1. thiamin (B1)	250
2. ascorbic acid (C)	1000
3. unknown	n/a
4. nicotinic acid (B3)	1000
5. unknown	n/a
6. pantothenic acid (B5)	1000
7. folic acid (B9)	500
8. riboflavin (B2)	250
9. methyl paraben	0.2

Sample: Analytes in water; initial dilutions of B1 and B2 basified with ammonium hydroxide
Column: Ultra Aqueous C18 (cat.# 9178575)
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Mobile Phase: A: 25mM potassium phosphate, pH 2.00: methanol (95:5, v/v)
B: methanol:25mM potassium phosphate, pH 3.5 (60:40, v/v)

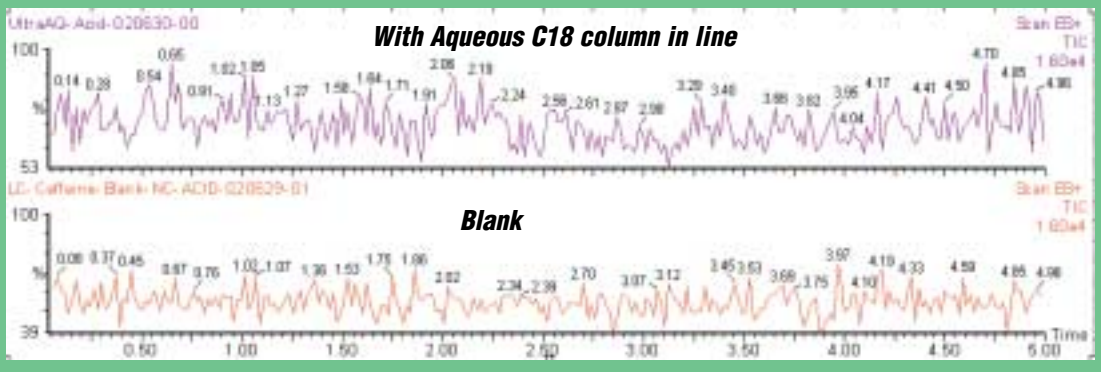
Time (min.)	%B
0-6	Hold 0
6.01	Step to 25
6.01-11	25-100
11-16	Hold 100

Flow: 1.0mL/min.
Temp.: 27°C
Det.: UV @ 254nm

LC_0141

Figure 4

Minimal background makes Ultra Aqueous C18 columns ideal for LC/MS.



Ordering Information | Ultra Aqueous C18 5µm Columns

	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
Length	**cat.#**	**cat.#**	**cat.#**	**cat.#**
30mm	9178531	9178532	9178533	9178535
50mm	9178551	9178552	9178553	9178555
100mm	9178511	9178512	9178513	9178515
150mm	9178561	9178562	9178563	9178565
200mm	9178521	9178522	9178523	9178525
250mm	9178571	9178572	9178573	9178575

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by Greg France, HPLC Product Marketing Manager

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HPLC Piston Seal Insertion Tool

- Simplify your pump maintenance.
- One end removes old piston seal, other easily and securely installs new seal.

Description	qty.	cat.#
HPLC Piston Seal Insertion Tool	ea.	21356

Description	Waters Instrument Model #	Waters™ part #	Equivalent Restek Part qty.	cat.#
Inlet Check Valve Assembly	M6KA, 501, 510, 515, 590, 600E	33679, 25214	ea.	25360
Inlet Check Valve Housing	M6KA, 501, 510, 515, 590, 600E	25203	ea.	25361
Inlet Check Valve Rebuild Kit	M6KA, 501, 510, 515, 590, 600E	60495	2-pk.	25362
Outlet Check Valve Assembly (Actuator Style)	M6KA, 501, 510, 515, 590, 600E	25030	ea.	25363
Outlet Check Valve Housing (Actuator Style)	M6KA, 501, 510, 515, 590, 600E	25212	ea.	25364
Outlet Check Valve Rebuild Kit (Actuator Style)	M6KA, 501, 510, 515, 590, 600E	26016	2-pk.	25365
Outlet Chack Valve Assembly (Ball & Seat Style)	M6KA, 501, 510, 515, 590, 600E	25216	ea.	25366
Outlet Check Valve Housing (Ball & Seat Style)	M6KA, 501, 510, 515, 590, 600E	25207	ea.	25367
Outlet Check Valve Rebuild Kit (Ball & Seat Style)	M6KA, 501, 510, 515, 590, 600E	26014	2-pk.	25368
Inlet Check Valve Assembly, 225µL (Extended Flow)	M6KA, 501, 510, 515, 590, 600E	60307	ea.	25369
PerformancePLUS™ Check Valve Housing	M6KA, 501, 510, 515, 590, 600E	700000254	2-pk.	25370
Check Valve Rebuild Kit (Extended Flow)	M6KA, 501, 510, 515, 590, 600E	88223	2-pk.	25371
PerformancePLUS™ Check Valve Housing	M6KA, 501, 510, 515, 590, 600E	—	ea.	25372
Round Pump Head w/ Actuator Outlets	M6KA, 510, 590, 600	60058	ea.	25413
Round Pump Head, Ball & Seat Check Valves	M6KA, M45, 501	—	ea.	25414
Check Valve Cartridges	Alliance™	WAT270941	2-pk.	25373
Super Seal™ for Analytical Heads	M6KA, 501, 510, 515, 590, 600E	22946, 22934	ea.	25374
Plunger Seal, Gold (Analytical Heads)	M6KA, 501, 510, 515, 590, 600E	22934	ea.	25375
Plunger Seal, Tan	M6KA, 501, 510, 515, 590, 600E	25384	ea.	25376
Plunger Seal, Black	M6KA, 501, 510, 515, 590, 600E	26613	ea.	25378
Plunger Seal, Gold (EF Heads)	510, 590, 600E	26644	ea.	25380
Seal Wash Plunger Seal	Alliance™	WAT271018	2-pk.	25386
Head Plunger Seal Kit	Alliance™	WAT270938	2-pk.	25387
Insert Seal Parts Kit	M6KA, 501, 510, 515, 590, 600E	60012	kit	25389
Sapphire Plunger	M6KA, 510, 590, 600	25656	ea.	25381
Sapphire Plunger	M45, M501	26524	ea.	25383
Sapphire Plunger	M515	WAT207069	ea.	25384
Sapphire Plunger	616, 625, 626	31788	ea.	25420
Sapphire Plunger	Alliance™	WAT270959	ea.	25385
Single Solvent Inlet Manifold	600E	60034, 60042	ea.	25390
Gradient Proportioning Valve, 12Volt	600E	62037	ea.	25419
Wash Face Seal	Alliance™ 2690	WAT271017	ea.	25428
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Xenon Lamp	474	—	ea.	25405
Deuterium Lamp (UV/Vis)	484	80357	ea.	25406
Deuterium Lamp	996, 2996	WAT052586	ea.	25408
Deuterium Lamp	2487	WAS081142	ea.	25409

Super-Clean™ Gas-Trapping System for LC/MS

New Quick-Change System Removes Hydrocarbon Impurities from Nitrogen

by Donna Lidgett, GC Accessories Product Marketing Manager

✔ Fast, easy cartridge changes—no tools, no purge time, no potential contamination.
✔ Durable, patented full glass/metal design.

The Super-Clean™ Gas-Trapping System is the latest technology in cartridge systems for gas purification, and is ideal for purifying nitrogen for LC/MS systems. Changing cartridges is quick and easy. A two-position base plate, installed in the gas line, allows you to exchange cartridges without introducing oxygen into the system: spring-loaded check valves seal when a cartridge is removed and open only when a new cartridge has been locked in place. There is no need for loosening and tightening fittings every time you change cartridges, and your system cannot become contaminated during the changing process.

To meet the high flow needs of an LC/MS system, the two cartridges are positioned and connected in parallel. The incoming gas stream is split equally between the two cartridges, and the two streams are rejoined after purification but before the gas exits the base plate. This approach allows longer contact between the nitrogen and the charcoal adsorbent, ensuring higher gas purity. The system can deliver up to 20L of 99.9999% pure nitrogen per minute, at a maximum pressure of 11 bar (160psi). Estimated cartridge lifetime is 3 to 6 months.

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(2) 2-Position baseplates & (4) charcoal filters
cat.# 22063 Offer ends 12/31/02.

Ordering Information | Super-Clean™ Gas-Trapping for LC/MS

Description	qty.	cat.#
LC/MS 2-Position Base Plate with ¼" Fittings	ea.	22060
Charcoal Replacement Filters	2-pk.	22061
Super-Clean™ Gas-Trapping System (includes 2-position base plate and 2 charcoal replacement filters)	ea.	22062

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by Jack Crissman, Seminar and Educational Products Manager

For review of these books, visit our website.

The Merck Index. 13th Edition: *S. Budvari, Merck, 2001, 2560pp., ISBN 0-91-191013-1 cat.# 21383 (ea.),*

A Century of Separation Science: *Haleem J. Issaq, Marcel Dekker, Inc., 2002, 755pp., ISBN 0-8247-0576-9* cat.# 20473 (ea.),

Chromatography Theory: *Jack Cazes and Raymond P.W. Scott, Marcel Dekker, Inc., 2002, 475pp., ISBN 0-8247-0778-8 cat.# 21573 (ea.),*

HPLC of Biological Macromolecules: Second Edition, Revised and Expanded: *Karen M. Gooding and Fred E. Regnier, Marcel Dekker, Inc., 2002, 777pp., ISBN 0-8247-0665-X cat.# 21574 (ea.),*

Advances in Chromatography, Volume 41: *Phyllis R. Brown and Eli Grushka, Marcel Dekker, Inc., 2001, 425pp., ISBN 0-8247-0509-2* cat.# 21575 (ea.),

Milestones in the Evolution of Chromatography: *Leslie S. Ettre, ChromSource, Inc., 2002, 220pp., ISBN 0-9717144-0-1 cat.# 20472 (ea.),*

Chromatography in Food Science and Technology: *Tibor Cserháti and Esther Forgács, CRC Press, LLC, 1999, 552pp., ISBN 1-56676-749-0* cat.# 21492 (ea.),

Gas Chromatographic Techniques and Applications: *Alan J. Handley and Edward R. Ardlard, CRC Press, LLC, 2001, 320pp., ISBN 0-8493-0521-7* cat.# 21491 (ea.),

Handbook of Chemistry and Physics, 83rd Edition: *D. R. Lide, CRC Press, LLC, 2002, 2,672pp., ISBN 0-8493-0483-0* cat.# 21442 (ea.),

Multidimensional Chromatography: *L. Mondello, A. C. Lewis and K. D. Bartle, John Wiley, 2002, 436pp., ISBN 0-471-98869-3* cat.# 21443 (ea.),

Modern Derivatization Methods for Separation Sciences: *T. Toyo'oka, John Wiley, 1999, 298pp., ISBN 0-471-98364-0* cat.# 21444 (ea.),

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by Brad Rightnour and Michael Goss, Instrument Innovations Team

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Place a 1cm plug of loosely bound wool in the liner. Adjust its position with the puller/inserter tool.

Use the hooked end of the puller/inserter to retrieve the plug when it's time to replace it.

Use with conventional 2mm ID or 4mm ID liners and most other liner configurations, but not with double gooseneck liners.

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Easily pre-seat ferrules for consistent installations in Agilent 5973 MS!

✔ Prevents damage to the column end
✔ Ensures leak-free connection
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Install the nut and ferrule onto the column, then insert the column through the installation tool, exposing several centimeters at the exit end.

Tighten the nut.

Score and remove the exposed end of the column making sure of a clean, square cut, then loosen the nut.

The ferrule will be properly seated and should remain in place when light force is applied. Install the column into the GC/MS interface.

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by Donna Lidgett, GC Accessories Product Marketing Manager

new

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✔ Teflon®-faced 0.125" silicone septa.
✔ Each case lot numbered.
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Description	qty.	cat.#
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40ml CLEAR Pre-Cleaned VOA Vials	72-pk.	21796
40ml AMBER Pre-Cleaned VOA Vials	72-pk.	21797

RESTEK Behind the Scenes

Happy Birthday, USA

Restek continues to be a major sponsor of one of the world's largest fireworks displays-Centre County, PA's 4th Fest. More than 100,000 people participated in part or all of the day-long festivities, which culminated in an aerial display of 12,000 shells. Restek also supplied the birthday cake—all 6 feet by 12½ feet of it. Approximately 1100 people had a slice, in celebration of our country's 226th birthday. Restek's founder and head coach, Paul Silvis, is a long-time supporter and co-chair of the event.

Restek Wins Healthy Workplace Award

Restek Corporation has been awarded the Healthy Workplace Award for Small Sized Companies by the Pennsylvania Psychological Association. This award is given annually to companies that demonstrate a commitment to family support, employee development, employee involvement, community involvement, and health and safety in the workplace. Last year, Restek was awarded honorable mention. The award was presented on June 21 at an official award ceremony in Lancaster.

Restek offers numerous employee-friendly benefits, including contributions toward child care costs, reimbursement for continuing education and development, on-site fitness and recreational facilities with subsidized personal trainers, 401k and employee stock ownership programs, open-book management, and a casual dress code. For the last 2 years, the company has won awards for being among the 100 Best Companies to Work for in Pennsylvania.

Restek Employees Prove They're Healthy and Involved

Proving our Healthy Workplace Award was no accident, two Restek employees, Becky Wittrig (Innovations Team chemist) and Matt Reilly (ATM technician) took first place in their divisions in a 5K run benefiting a local library, in June. Becky's son, James, Matt's daughter, Leena, and Alex Reid, son of HPLC Applications Team chemist Terry Reid, ran in the children's divisions. Our Employee Action Group made a $100 sponsorship donation.

Also in June, Restek's Relay for Life team won the Bronze Award at the local Relay event by raising more than $1500 for the American Cancer Society.

New Literature

- ✔ Narcotics / Acetaminophen by HPLC
  Applications Note (lit. cat.# 59453)
- ✔ Organophosphorus Pesticides by Capillary GC
  Applications Note (lit. cat.# 59359)
- ✔ Calibration Standards for ASTM Method D2887-01
  Fast Facts (lit. cat.# 59383A)
- ✔ Certified PAHs in Diesel Fuel #2
  Fast Facts (lit. cat.# 59384A)
- ✔ Environmental Gas Standards
  Fast Facts (lit. cat.# 59276)
- ✔ Pesticide Reference Materials
  Fast Facts (lit. cat.# 59446)
- ✔ Pinnacle II™ Amino HPLC Columns
  Fast Facts (lit. cat.# 59385A)
- ✔ Rtx®-5Sil MS Capillary Columns
  Fast Facts (lit. cat.# 59323)
- ✔ UST Products for Massachusetts
  Fast Facts (lit. cat.# 59391)
- ✔ UST Products for the Northwest Region
  Fast Facts (lit. cat.# 59396)
- ✔ UST Products for Wisconsin
  Fast Facts (lit. cat.# 59392)
- ✔ Rtx®-VMS Capillary Columns
  New Product Flyer (lit. cat.# 59209A)
- ✔ Bonded PLOT Columns—Flyer (lit. cat.# 59456)
- ✔ Integra-Guard™ Capillary Columns
  Flyer (lit. cat.# 59441)
- ✔ Review of Restek Literature for Pharmaceuticals Analyses—Flyer (lit. cat.# 59450)
- ✔ Genuine Restek Replacement Parts for Agilent GCs—Catalog (lit. cat.# 59627C)

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Please direct your comments on this publication to Carrie Sprout, Graphic Designer, at carrie@restekcorp.com or call 814-353-1300, ext. 2151.

the RESTEK Advantage

Innovators of High Resolution Chromatography Products

Optimized Analysis of Volatile Organics in Hazardous Waste

new

Using an Rtx®-VMS GC/MS Column and New Restek Reference Materials

by Christopher English, Environmental Innovations Chemist

- ✔ Rtx®-VMS polymer formulated specifically for volatiles analysis by GC/MS.
- ✔ Greater throughput—excellent separations and faster analyses for 100+ volatiles and associated compounds.
- ✔ Excellent sensitivity—high thermal stability minimizes bleed.
- ✔ Convenience—8260B Calibration Mix #1A is a stable mixture of 76 + 1 volatiles.

Accurate and rapid analyses of over 100 volatile organic compounds (VOCs), in a wide range of matrices, are a major challenge to environmental laboratories. US EPA Method 8260B, described in US EPA SW-846, is widely used as a guideline for analyses of VOCs in hazardous waste, sludges, or other discarded material, prior to depositing these materials in hazardous waste facilities. The method also is followed for monitoring ground water at these facilities.

Restek chemists designed the Rtx®-VMS column to address the large—and increasing—number of analytes listed in US EPA Methods 8260 and 524.2, plus unlisted but commonly encountered compounds, such as acetates and oxygenates (ethers). With the aid of computer modeling, we tuned the stationary phase in the Rtx®-VMS column specifically toward resolving compounds that share common quantification ions. This has improved selectivity, reduced bleed, and shortened analysis time, relative to traditional "624/1301" phases. In designing this stationary phase we considered performance for compounds that are not listed in the usual test methods (e.g., acetates and oxygenates). We added these compounds to the design criteria for the Rtx®-VMS phase because many of them have been discovered in ground water, and analysts in environmental laboratories often add them to calibration mixes.

A 40m x 0.18mm ID x 1.0µm Rtx®-VMS column, and the chromatographic conditions used to obtain Figure 1 (page 2), are optimal for a 14-minute analysis, which is less than the cycle time for a standard purge and trap unit. The conservative initial temperature enhances the resolution between *tert*-butyl alcohol and methyl *tert*-butyl ether (peaks 21 & 22)—the two analytes are baseline resolved under these conditions. An initial temperature of 50°C or lower enhances the resolution between chloromethane and vinyl chloride (peaks 4 and 5). Alternatively, an initial temperature of up to 60°C can be used, to promote faster oven cycling and to prevent large amounts of methanol from condensing at the head of the column.

The Rtx®-VMS column is available in a wide variety of internal diameters. Base your choice of ID and column length on the number of target analytes in your samples, and on your instrumentation.

We now offer a comprehensive acetates mix (cat.# 30489) and a comparable oxygenates mix (cat.# 30465), which complement our 8260B Calibration Mix #1 of VOCs (cat.# 30475). These three reference solutions account for more than 80% of the most common target compounds shown in Figure 1.

In volatiles analysis, oven cycle time and/or purge and trap cycle time, not analysis time, are the factors that limit productivity. Because Rtx®-VMS columns are designed for higher starting temperatures, they shorten oven cycle times - and thus increase sample throughput - without sacrificing resolution of gaseous analytes. To see example chromatograms, request a copy of our Rtx®-VMS capillary columns flyer (lit. cat.# 59209A).

in this issue

RESTEK
800-356-1688 ★ 814-353-1300
www.restekcorp.com

2002 vol. 4

Column:	Rtx®-VMS 40m, 0.18mm ID, 1.0µm (cat.# 49915)
Sample:	5ppb each compound in 25mL of RO water
Concentrator:	Tekmar LSC-3100 Purge and Trap
Trap:	Vocarb™ 3000
Purge:	11 min. @ 40mL/min.
Dry purge:	1 min. @ 40mL/min. (MCS by-passed with Silcosteel® tubing, cat.# 21035)
Desorb preheat:	245°C
Desorb:	2 min. @ 250°C
Desorb flow rate:	45mL/min. helium.
Bake:	8 min. @ 260°C
Interface:	plumbed through injection port
Transfer line:	Silcosteel® transfer line (cat.# 20591)
Mount Temp:	40°C
Split ratio:	1:40 split
Inlet liner:	1mm split (cat.# 20972)
Inj. temp.:	250°C
Carrier gas:	1.1mL/min. helium, constant flow
Linear velocity:	32cm/sec. @ 40°C
Oven temp.:	35°C (hold 2 min.) to 60°C @ 4°C/min. (hold 0 min.) to 225°C @ 40°C/min. (hold 5 min.)
Detector:	5973 GC/MS w/ turbomolecular pump
Source Temp.:	280°C
Scan range:	35–260amu
Ionization:	EI

Standards:

8260B Calibration Mix #1	cat.# 30475
502.2 Calibration Mix #1 (gases)	cat.# 30042
VOA Calibration Mix #1 (ketones)	cat.# 30006
8260 Internal Standard Mix	cat.# 30074
8260 Surrogate Mix	cat.# 30073
8260 Acetate Mix	cat.# 30489
California Oxygenates Mix	cat.# 30465
Acrolein Mix	cat.# 30478
Ethanol Mix	cat.# 30466
Freon® 114	cat.# 30476

Ordering Information | Rtx®-VMS Columns (Fused Silica)

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter
0.25mm	1.40	-40 to 240/260°C	19915	19916	
0.32mm	1.80	-40 to 240/260°C	19919	19920	
0.45mm	2.55	-40 to 240/260°C	19908	19909	
0.53mm	3.00	-40 to 240/260°C	19985	19988	19974
ID	**df (µm)**	**temp. limits**	**20-Meter**	**40-Meter**	
0.18mm	1.00	-40 to 240/260°C	49914	49915	

8260B Calibration Mix #1

(76 +1 components)

Ampul 1: 8260B Calibration Mix #1A
*Components in **bold** in peak list below.*
Ampul 2: 2-chloroethyl vinyl ether
2,000µg/mL each in P&T methanol, 1mL/ampul

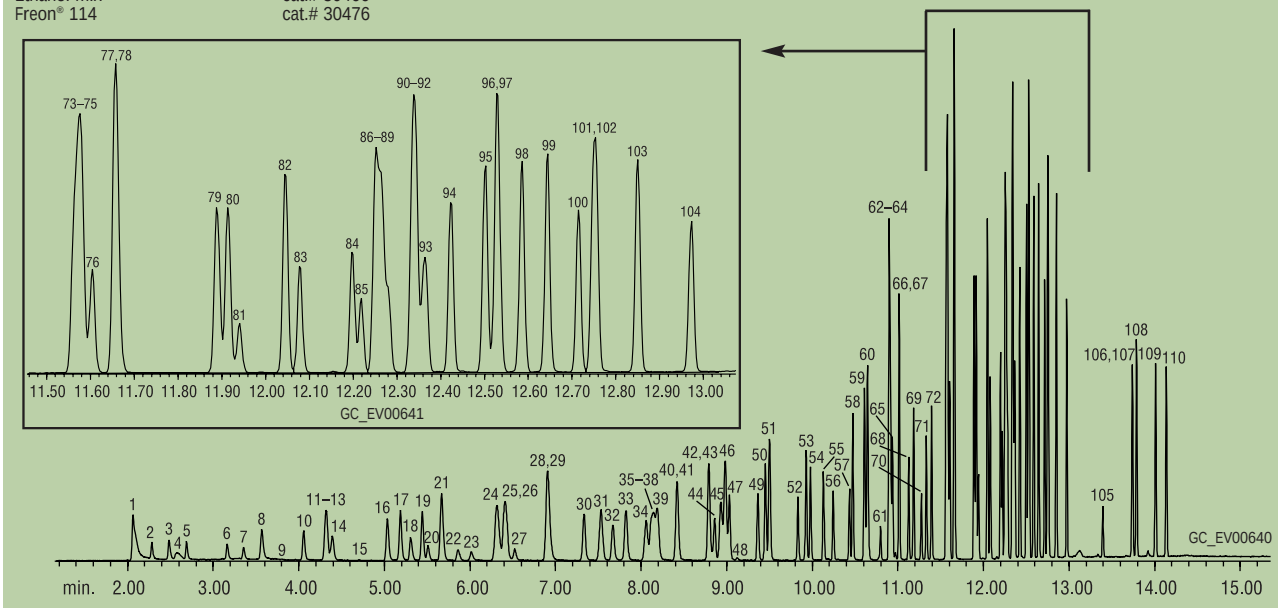
Each	5-pk.	10-pk.
30475	30475-510	—
	with data pack	
30475-500	30475-520	30575

for more info

For more information about Rtx®-VMS columns, request lit. cat.# 59209A.

Figure 1

Rtx®-VMS columns improve selectivity, minimize bleed, and shorten cycle time for VOCs.



1. carbon dioxide
2. dichlorodifluoromethane
3. Freon® 114
4. chloromethane
5. vinyl chloride
6. bromomethane
7. chloroethane
8. trichlorofluoromethane
9. ethanol
10. **diethyl ether**
11. **1,1-dichloroethene**
12. **iodomethane**
13. **carbon disulfide**
14. **Freon® 113**
15. acrolein
16. **allyl chloride**
17. **methylene chloride**
18. acetone
19. **trans-1,2-dichloroethene**
20. methyl acetate
21. methyl *tert*-butyl ether
22. *tert*-butyl alcohol
23. **acetonitrile**
24. diisopropyl ether
25. **chloroprene**
26. **1,1-dichloroethane**
27. **acrylonitrile**
28. vinyl acetate
29. ethyl-*tert*-butyl ether
30. ***cis*-1,2-dichloroethene**
31. **2,2-dichloropropane**
32. **bromochloromethane**
33. **chloroform**
34. **carbon tetrachloride**
35. ethyl acetate
36. **methyl acrylate**
37. dibromofluoromethane
38. **tetrahydrofuran**
39. **1,1,1-trichloroethane**
40. 2-butanone
41. **1,1-dichloropropene**
42. **benzene**
43. **propionitrile**
44. **methacrylonitrile**
45. pentafluorobenzene
46. *tert*-amyl-methyl ether
47. **1,2-dichloroethane**
48. **isobutyl alcohol**
49. isopropyl acetate
50. **trichloroethene**
51. 1,4-difluorobenzene
52. **dibromomethane**
53. **1,2-dichloropropane**
54. **bromodichloromethane**
55. **methyl methacrylate**
56. *n*-propyl acetate
57. 2-chloroethyl vinyl ether*
58. ***cis*-1,3-dichloropropene**
59. toluene-d8
60. **toluene**
61. **2-nitropropane**
62. **tetrachloroethene**
63. 4-methyl-2-pentanone
64. ***trans*-1,3-dichloropropene**
65. 2-bromo-1-chloropropane
66. **1,1,2-trichloroethane**
67. **ethyl methacrylate**
68. **dibromochloromethane**
69. **1,3-dichloropropane**
70. **1,2-dibromoethane**
71. *n*-butyl acetate
72. 2-hexanone
73. chlorobenzene-D5
74. **chlorobenzene**
75. **ethylbenzene**
76. **1,1,1,2-tetrachloroethane**
77. ***m*-xylene**
78. ***p*-xylene**
79. ***o*-xylene**
80. **stryrene**
81. **bromoform**
82. **isopropylbenzene**
83. *n*-amyl acetate
84. 4-bromo-1-fluorobenzene (ss)
85. ***cis*-1,4-dichloro-2-butene**
86. ***n*-propylbenzene**
87. **bromobenzene**
88. 1,4-dichlorobutane
89. **1,1,2,2-tetrachloroethane**
90. **2-chlorotoluene**
91. **1,3,5-trimethylbenzene**
92. **1,2,3-trichloropropane**
93. ***trans*-1,4-dichloro-2-butene**
94. **4-chlorotoluene**
95. ***tert*-butylbenzene**
96. **pentachloroethane**
97. **1,2,4-trimethylbenzene**
98. ***sec*-butylbenzene**
99. ***p*-isopropyltoluene**
100. **1,3-dichlorobenzene**
101. 1,4-dichlorobenzene-d4
102. **1,4-dichlorobenzene**
103. ***n*-butylbenzene**
104. **1,2-dichlorobenzene**
105. **1,2-dibromo-3-chloropropane**
106. **nitrobenzene**
107. **hexachlorobutadiene**
108. **1,2,3-trichlorobenzene**
109. **naphthalene**
110. **1,2,4-trichlorobenzene**

Analytes in bold are components of 8260B Calibration Mix #1A (cat.# 30475).

Note: 2-chloroethanol is in 8260B mix, but requires lower scan range for identification; 1,4-dioxane is in 8260B mix, but requires fortification or identification using single point high calibration standard.
**Component of 8260B Calibration Mix #1, packaged in a separate ampul.*

Vespel® Ring Inlet Seal

Seals the First Time, Every Time

by Donna Lidgett, GC Accessories Product Marketing Manager

- ✔ Easy-to-use, patent-pending design makes a better seal, easily.
- ✔ Prevents oxygen from damaging your columns.
- ✔ Reduces wear on the injection port body.

In Agilent split/splitless injection ports, it can be difficult to make and maintain a good seal with a conventional metal inlet disk. The metal-to-metal seal dictates that the analyst apply considerable torque to the reducing nut, and, based on our testing, this does not ensure a leak-tight seal. Over the course of oven temperature cycling, metal seals are prone to leaks, which ultimately can degrade the capillary column, and cause other analytical difficulties.

Our Vespel® Ring Inlet Seal greatly improves injection port performance—it stays sealed even after repeated temperature cycles, without retightening the reducing nut! This seal features a Vespel® ring embedded into its face. This soft Vespel® ring will not harm the critical seal on the injector body, and is outside the sample flow path. Tests using a high sensitivity helium leak detector indicate the Vespel® Ring Inlet Seal seals equally effectively at torques from 5 in. lb. to 60 in. lb. (Figure 1).

Why trust a metal-to-metal seal when you can make leak-tight seals quickly and easily—and more reliably—with the Restek Vespel® Ring Inlet Seal? Use the stainless steel seal for analysis of unreactive compounds. To reduce breakdown and adsorption of active compounds, use the gold-plated or Silcosteel®-treated seals. The gold surface offers better inertness than standard stainless steel; Silcosteel® treatment provides inertness similar to that of fused silica capillary columns.

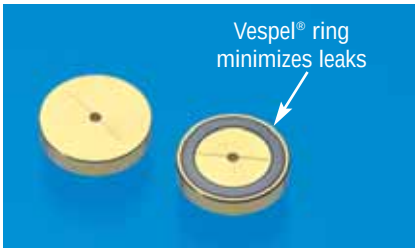
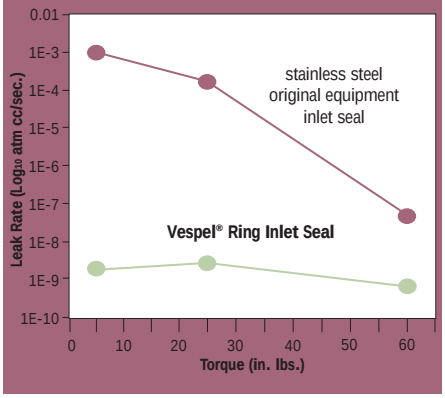


Figure 1
The Vespel® Ring Inlet Seal achieves leak-tight seals even at low torque, reducing the chance of leak-related problems.



Ordering Information | Vespel® Ring Inlet Seals for Agilent 5890/6890 and 6850 GCs

0.8mm ID Vespel® Ring Inlet Seal (washers included)	**2-pk.**	**10-pk.**
Gold-Plated	21562	21563
Silcosteel®	21564	21565
Stainless Steel	21560	21561
1.2mm ID Vespel® Ring Inlet Seal* (washers included)	**2-pk.**	**10-pk.**
Gold-Plated	21568	21569
Silcosteel®	21570	21571
Stainless Steel	21566	21567

*For dual-column installations.

A Compact, Sensitive Leak Detector For Every GC Analyst

The Restek Leak Detective™ II

by Donna Lidgett, GC Accessories Product Marketing Manager

- ✔ Fast results—responds to trace leaks in less than 2 seconds.
- ✔ Sensitive—detects trace leaks at 1 x 10^{-4}cc/sec.; as low as 100ppm.
- ✔ Microchip design improves sensitivity and response time over previous models.
- ✔ Compact, ergonomic design is easy to hold and operate with one hand.
- ✔ Battery-operated for portability (one 9 volt); instant auto-zeroing.

Gas leaks in your GC system can increase detector noise, cause baseline instability, waste carrier gas, and damage valuable analytical columns. Leak checks should be a regular part of your GC maintenance program. The new Leak Detective™ II electronic leak detector is the sensitive, affordable solution for detecting gas leaks.*

Microchip technology and a new design give you better sensitivity and faster response time in a more compact unit. You can instantly zero the leak detector with a push of a button, and the ergonomic design brings all the controls to your fingertips for easy use. The unit responds in less than two seconds to trace leaks of gases with thermal conductivities different than air: detect helium, hydrogen, or nitrogen at 1x10^{-4}cc/sec or at an absolute concentration as low as 100ppm. Leaks are indicated by an audible alarm, as well as by an LED readout. For easy, sensitive, and reliable leak detection, order a new Leak Detective™ II electronic leak detector today.

Ordering Information | Leak Detective II™

Description	**qty.**	**cat.#**	**price**
Leak Detective™ II Leak Detector	ea.	20413	

**Never use liquid leak detectors on a capillary system because liquids can be drawn into the column.*
Caution: *NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.*

Specialty Reversed Phase HPLC Columns for Polar Analytes

Ultra Aqueous C18 and Ultra IBD Columns Solve Retention Dilemmas

by Terrence S. Reid, HPLC Innovations Chemist

✔ Both columns provide sharp peaks for basic analytes.
✔ Both columns compatible with 100% aqueous mobile phases.
✔ Complementary selectivity for acidic and basic analytes.

Over the past several years, HPLC column manufacturers have been creating new stationary phases in attempts to address some of the separation problems encountered by analytical chemists.

Stationary phases in traditional reversed phase columns are strictly non-polar alkyls, like C18. In contrast, many newer specialty reversed phase columns have stationary phases that are primarily alkyls, but with some secondary polar functionality. The polar functionality offers several advantages, including: unique selectivity, enhanced retention of polar compounds, and compatibility with completely aqueous mobile phases.

These specialty reversed phase columns can differ either in the type of polar group they incorporate or in how the polar group is incorporated into the stationary phase. Restek offers two specialty reversed phase columns that represent two different approaches to introducing secondary polar groups into a straight chain alkyl ligand (Figure 1). The stationary phase in the Ultra Aqueous C18 column has small polar groups attached to the silica surface, between the C18 chains. In contrast, the Ultra IBD stationary phase is a "polar embedded" type stationary phase, because its polar groups are embedded within a straight alkyl chain.

One drawback to these specialty columns is that their potential for mixed mode interactions makes it more difficult to predict which column will perform best for a particular application. With this in mind, we used a series of simple tests to directly compare the performance characteristics of Ultra Aqueous C18 and Ultra IBD columns. From the results of these tests, we can offer some useful guidelines for selecting a specialty reversed phase column.

The first test measured the hydrophobic retention of each column, using a sample mixture of completely nonpolar analytes and a mobile phase containing a high proportion of organic solvent. For pure alkyl stationary phases, hydrophobic retention usually is directly proportional to the percent carbon (%C) in the bonded phase silica, if the phases are bonded on silica particles of comparable surface area. Figure 2 shows that the hydrophobic retention of Ultra Aqueous C18 columns is approximately twice that of Ultra IBD columns, based on capacity factors for pyridine, despite the two bonded phases' similar surface area and %C (Ultra Aqueous C18: 100Å pores, 14%C; Ultra IBD: 100Å pores, 12%C). The hydrophobic retention of Ultra Aqueous C18 columns is equivalent to that of conventional C18 columns with the same surface area and %C. The considerably reduced hydrophobic retention of Ultra IBD columns can be attributed to the embedded polar group in the stationary phase shielding the lower portion of the alkyl chain from the nonpolar analytes.

We compared the columns' base deactivation by measuring the peak shape for a basic analyte, pyridine (Figure 3). Both Ultra Aqueous C18 and Ultra IBD columns show excellent base deactivation, with pyridine peak symmetry values better than those for highly base-deactivated C18 phases made through conventional chemistry. Although they are similarly base-deactivated, Ultra Aqueous C18 columns exhibit much greater retention of pyridine than do Ultra IBD columns.

Next, we compared Ultra Aqueous C18 and Ultra IBD columns' ability to separate small carboxylic acids. It is difficult for conventional reversed phase columns to retain these molecules. A very weak, highly aqueous mobile phase is required. Many C18 phases are not compatible with highly aqueous mobile phases, and show a gradual or sudden loss of retention that is attributed to "chain folding" or "phase collapse." Both Ultra Aqueous C18 and Ultra IBD columns are completely compatible with 100% aqueous mobile phases, as shown in Figure 3A. Neither column showed any loss of retention, even after mobile phase flow was temporarily stopped. (Absence of pressure maximizes the potential for phase collapse, thus exposure to 100% aqueous mobile phase under no flow is the most extreme test of phase integrity.) This comparison did reveal

Figure 1

The stationary phase in an Ultra Aqueous C18 column has small polar groups attached to the silica surface; in the Ultra IBD stationary phase polar groups are embedded in the alkyl chain.

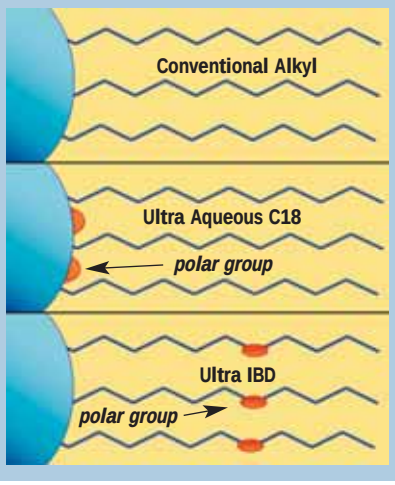


Figure 2

Hydrophobic retention for Ultra Aqueous C18 columns is approximately twice that for Ultra IBD columns, despite similar surface areas and % carbon.

Peak List:	Conc. (mg/mL)
1. uracil	0.02
2 benzene	3.00
3. naphthalene	0.50
4. biphenyl	0.06

Column:	Ultra Aqueous C18	Ultra IBD
Catalog #:	9178565	9175565
Dimensions:	150 x 4.6mm	150 x 4.6mm
Particle Size:	5µm	5µm
Pore Size:	100Å	100Å

Sample: Inj.: 1.3µL
Solvent: methanol:water (75:25, v/v)
Mobile Phase: A: water
B: methanol
Isocratic: 80%B
Flow: 1.0mL/min
Temp.: ambient
Det.: UV @ 254nm

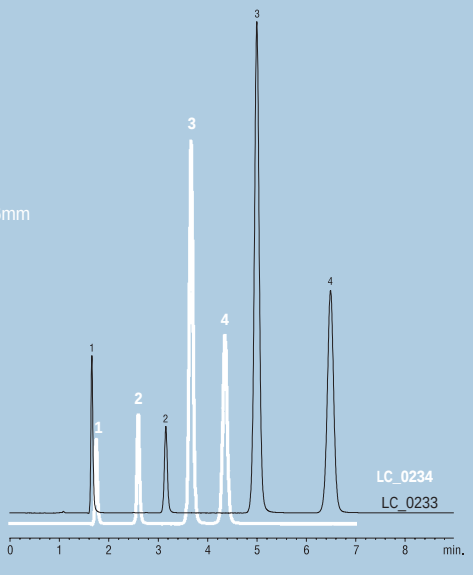
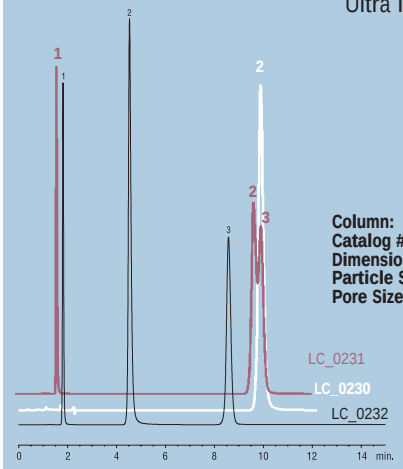


Figure 3

Ultra IBD and Ultra Aqueous C18 columns share important characteristics.

A) Excellent Deactivation for Bases

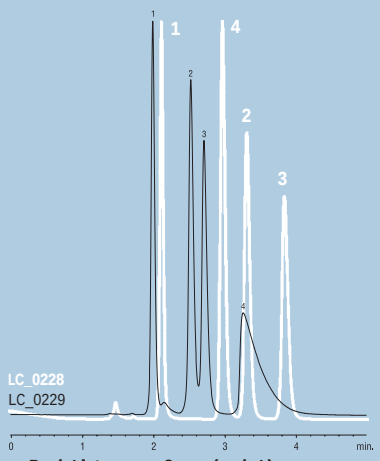


	Ultra IBD	Ultra Aqueous C18	Ultra Aqueous C18
Column:			
Catalog #:	9175565	9178565	9178565
Dimensions:	150 x 4.6mm	150 x 4.6mm	150 x 4.6mm
Particle Size:	5µm	5µm	5µm
Pore Size:	100Å	100Å	100Å

Peak List:	Conc.
1. uracil	50µg/mL
2 pyridine	0.1µL/mL
3. phenol	1.86mg/mL

Sample: Inj: 5µL
Solvent: mobile phase
Mobile phase: A: 20mM potassium phosphate, pH 7.0
B: acetonitrile
Isocratic: 20%B
Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 254nm

B) Compatible with 100% Aqueous Mobile Phases

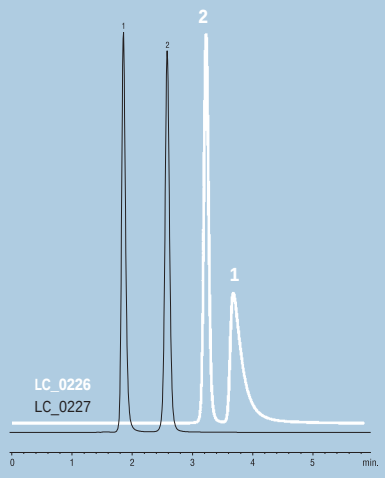


Peak List	Conc. (mg/mL)
1. glycolic acid	2.0
2. lactic acid	3.3
3. acetic acid	2.1
4. malonic acid	1.8

	Ultra IBD	Ultra Aqueous C18
Column:		
Catalog #:	9175565	9178565
Dimensions:	150 x 4.6mm	150 x 4.6mm
Particle Size:	5µm	5µm
Pore Size:	100Å	100Å

Sample: Inj.: 10µL
Solvent: mobile phase
Mobile Phase: A: 50mM potassium phosphate, pH 2.5
Isocratic: 100%A
Flow: 1.0mL/min
Temp.: ambient
Det.: UV @ 210nm

C) Complementary Selectivity



Peak List:	Conc. (mg/mL)
1. thiamin	2.5
2. ascorbic acid	2.5

	Ultra IBD	Ultra Aqueous C18
Column:		
Catalog #:	9175565	9178565
Dimensions:	150 x 4.6mm	150 x 4.6mm
Particle Size:	5µm	5µm
Pore Size:	100Å	100Å

Sample: Inj.: 1.0µL
Solvent: mobile phase
Mobile Phase: A: 50mM potassium phosphate, pH 2.5
Isocratic: 100%A
Flow: 1.0 mL/min.
Temp.: ambient
Det.: UV @ 254nm

some differences between Ultra Aqueous C18 and Ultra IBD columns, however. Note that the elution orders in Figure 3B are different, demonstrating significant differences in selectivity. Also, the malonic acid peak tails on the Ultra IBD column. We believe this is because malonic acid has two carboxylic acid groups—several dicarboxylic acids tail on Ultra IBD columns and similar columns.

Finally, we used the same 100% aqueous mobile phase to evaluate selectivity and retention for mixtures containing both an acid (ascorbic acid/Vitamin C) and a base (thiamin/Vitamin B1). Note in Figure 3C that Ultra Aqueous C18 and Ultra IBD columns produce opposite elution order, again demonstrating their complementary selectivity. Of several columns evaluated from various manufacturers, the Ultra Aqueous C18 column was the only column to exhibit significant retention of thiamin. This is consistent with the enhanced retention of pyridine shown in Figure 3A. Thiamin is barely retained by an Ultra IBD column, using the uracil peak in Figure 2 as the measure of column void volume (t_o).

Highly base-deactivated Ultra Aqueous C18 and Ultra IBD columns are powerful tools for analyzing polar compounds by reversed phase HPLC. They offer alternate selectivity to each other, as well as to conventional C18 stationary phases. Their secondary polar functionalities enhance retention of polar analytes, contribute to their unique selectivity, and make them compatible with a complete spectrum of mobile phase compositions, from 100% organic to 100% aqueous. An Ultra Aqueous C18 column is the better choice for small dicarboxylic acids, or for maximum retention of bases in a highly aqueous mobile phase.

Ordering Information | Ultra Aqueous C18 5µm Columns

	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
Length	**cat.#**	**cat.#**	**cat.#**	**cat.#**
30mm	9178531	9178532	9178533	9178535
50mm	9178551	9178552	9178553	9178555
100mm	9178511	9178512	9178513	9178515
150mm	9178561	9178562	9178563	9178565
200mm	9178521	9178522	9178523	9178525
250mm	9178571	9178572	9178573	9178575

Ordering Information | Ultra IBD 5µm Columns

	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
Length	**cat.#**	**cat.#**	**cat.#**	**cat.#**
30mm	9175531	9175532	9175533	9175535
50mm	9175551	9175552	9175553	9175555
100mm	9175511	9175512	9175513	9175515
150mm	9175561	9175562	9175563	9175565
200mm	9175521	9175522	9175523	9175525
250mm	9175571	9175572	9175573	9175575

for more info

For our complete line of HPLC columns and HPLC accessories, request the *2003 Chromatography Products Guide* (lit. cat.# 59473)

Sulfinert™- & Silcosteel®-Treated Ball & Plug Valves

Helping Complete the Inert Sample Pathway

by Gary Barone, Metals Passivation Marketing Manager

✔ No adsorption of active compounds at low ppb concentrations.
✔ Stable, flexible surface deactivation that will not crack or flake.
✔ Commonly used pathway components now available from stock.

We are pleased to introduce Restek's new Sulfinert™-treated and Silcosteel®-treated plug valves and ball valves with 1/8" and 1/4" fittings. Until now, the only way to obtain these deactivated parts was to supply Restek with disassembled valve components for custom coating. Now, the newest product offerings from our Metals Passivation Group give you the benefits of these stable, flexible surface treatments on stock items.

Sulfinert™-treated or Silcosteel®-treated valves are logical components in a system that incorporates Sulfinert™- or Silcosteel®-treated tubing and fittings. Combine deactivated valving with our other stock deactivated sample pathway components to create the most inert pathway available.

Our Sulfinert™- or Silcosteel®-treated plug valve is made by Parker, and is rated for service up to 3000psig at 21ºC. It can be used in environments having temperatures up to 205ºC.

The Sulfinert™- or Silcosteel®-treated ball valve also is a Parker valve. The construction of this valve allows us to completely coat all sample-wetted steel components. The Sulfinert™- or Silcosteel®-treated ball "floats" on dual seats to reduce operating torque and provide leak-tight bi-directional sealing. The ball valve is rated for pressures up to 1500psig at 21ºC.

How do you decide which parts you need? Use Sulfinert™-treated valves, fittings, and tubing when creating pathways for extremely low-level (i.e., parts-per-billion) organics. Also, use Sulfinert™-treated pathway components when designing a system for sulfur-, phosphorus- and/or nitrogen-containing organics. Application areas that benefit from a Sulfinert™-treated sample pathway include analyses of sulfurs in air, in natural gas, in ethylene/propylene, or in beverage-grade carbon dioxide, any flowpath for pesticides or herbicides, and chemical weapons analyzers.

Silcosteel® treatment continues to be a highly popular choice for passivating systems and flowpaths that carry target analytes at parts-per-million levels. Time-proven Silcosteel® passivation is used to deactivate components in virtually all of today's leading sampling systems and instruments.

Ordering Information | Sulfinert™- and Silcosteel®-Treated Valves

		Sulfinert™-Treated		Silcosteel®-Treated	
Fitting Type	**Size**	**qty.**	**cat.#**	**qty.**	**cat.#**
Plug Valve 	1/8"	ea.	21586	ea.	21576
	1/4"	ea.	21587	ea.	21577
Ball Valve 	1/8"	ea.	21588	ea.	21578
	1/4"	ea.	21589	ea.	21579

Ordering Information | Coiled Sulfinert™-Treated Welded 304 Grade Stainless Steel Tubing

			Price-per-foot by length			
ID	**OD**	**cat.#**	**5-24 ft.**	**25-199 ft.**	**200-399 ft.**	**>400 ft.**
0.011" (0.28mm)	0.022" (0.56mm)	22500				
0.021" (0.53mm)	0.029" (0.74mm)	22501				
0.010" (0.25mm)	1/16" (1.59mm)	22502				
0.020" (0.51mm)	1/16" (1.59mm)	22503				
0.030" (0.76mm)	1/16" (1.59mm)	22504				
0.040" (1.02mm)	1/16" (1.59mm)	22505				
0.085" (2.16mm)	1/8" (3.18mm)*	22506				
0.210" (5.33mm)	1/4" (6.35mm)*	22507				

Ordering Information | Coiled Silcosteel®-Treated Welded/Drawn 304 Grade Stainless Steel Tubing

			Price-per-foot by length			
ID	**OD**	**cat.#**	**5-24 ft.**	**25-199 ft.**	**200-399 ft.**	**>400 ft.**
0.011" (0.28mm)	0.022" (0.56mm)	20590				
0.021" (0.53mm)	0.029" (0.74mm)	20591				
0.010" (0.25mm)	1/16" (1.59mm)	20592				
0.020" (0.51mm)	1/16" (1.59mm)	20593				
0.030" (0.76mm)	1/16" (1.59mm)	20594				
0.040" (1.02mm)	1/16" (1.59mm)	20595				
0.085" (2.16mm)	1/8" (3.18mm)*	20596				
0.210" (5.33mm)	1/4" (6.35mm)*	20597				

for more info

Request the *2003 Product Guide* (lit. cat.# 59473) or visit **www.restekcorp.com** for complete listing of Silcosteel®- and Sulfinert™-treated fittings.

Paul Silvis, Restek Head Coach and Founder, Named Entrepreneur of the Year (2002)

PA Business Central chose Restek's Head Coach in the For-Profit, Large Business category. "Paul's innovative approach to business and the results speak for themselves. He created a company out of nowhere and has been successful beyond all expectations. Paul epitomizes the model by which many entrepreneurs should compare themselves," said Todd A. Erdley of Videon Central, one of this year's contest judges and a recipient of the newspaper's Entrepreneur 2001 award in the Small Business category. ***Congratulations, Paul!***

**0.020" wall thickness*

Analysis of European Organophosphorus Pesticides

Fast, Complete Resolution Using an Rtx®-CLPesticides Column

by Christopher English, Environmental Innovations Chemist

✔ Fastest analysis of 21 target OPPs and surrogates.
✔ Extremely low column bleed at temperatures >300ºC.
✔ Use Rtx®-CLPesticides columns with FPDs, NPDs, or GC/MS systems.

Our European customers provided us with a list of organophosphorus compounds and asked us to help develop a GC analysis. Restek offers the analytical reference standards, and we now have determined the chromatography column and conditions needed for a problem-free analysis of these materials.

Organophosphorus pesticide (OPP) is a general term that includes all phosphorus-containing insecticides. Because these compounds eliminate target insects effectively, then break down into non-toxic derivatives, they have replaced many of the environmentally persistent organochlorine pesticides. By 1959 over 50,000 different OPPs had been prepared. OPPs are hydrolyzed by bases and acids and are photosensitive, which makes them ideal for agricultural applications.

The main concern OPPs pose is the intact molecules' high toxicity to mammals—these chemicals are considered the most toxic of all pesticides to vertebrates. Further, at low levels, OPPs can be endocrine-disrupting compounds (EDC) - materials that interfere with hormone activities in mammals. Dimethoate and malathion have been shown to be in this category. Consequently, dimethoate and malathion are on the European Priority List; further study is required to determine if use of these two pesticides should be suspended.

We used computer modeling software to design the Rtx®-CLPesticides column for analyses of chlorinated pesticides, but this column also is well suited for analyses of electronegative species, such as OPPs. We evaluated many columns for resolving this selection of compounds, and we found that the Rtx®-CLPesticides column resolves these compounds to baseline in the least amount of time.

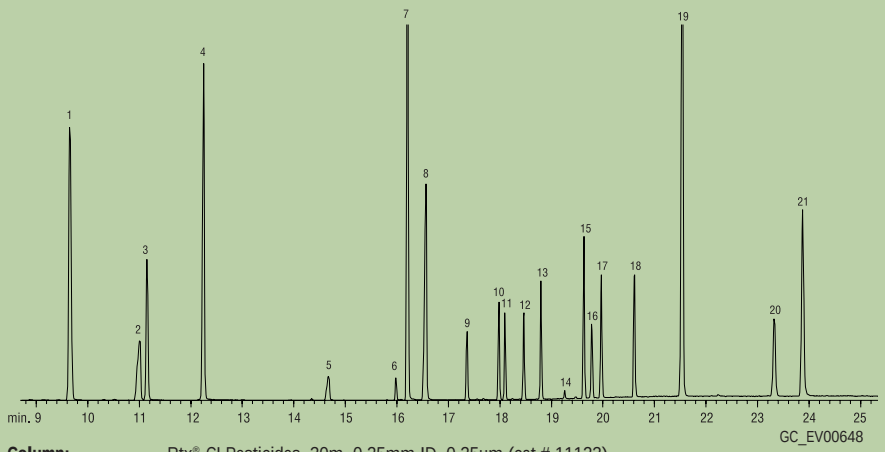
The 30m x 0.25mm x 0.25µm Rtx®-CLPesticides column configuration is best suited for GC/MS applications; a 30m x 0.32mm x 0.50µm column can be used with direct injections and FPD or NPD detection. The GC/MS analysis is shown in Figure 1. For the application shown we added the surrogates most commonly used with FPD or NPD detectors and optimized conditions so that all compounds, including the four added surrogates, are baseline resolved.

The high temperature stability of Rtx®-CLPesticides columns enabled us to bring the oven temperature to 330°C, following each analysis, to bake out high molecular weight contaminants commonly found in OPP-containing extracts. The ability to condition these columns at higher temperatures, not attainable with commonly used cyano phases, will enable you to make more injections with an Rtx®-CLPesticides column.

In most laboratories, fast, efficient sample throughput is important. In analyses of organophosphorus pesticides, an Rtx®-CLPesticides column is an important part of attaining—and maintaining—this goal.

Figure 1

An Rtx®-CLPesticides column provides sharp, fast resolution of organophosphorus pesticides.



Column:	Rtx®-CLPesticides, 30m, 0.25mm ID, 0.25µm (cat.# 11123)	
Sample:	Custom European Standard Mix	call for details
	1-bromo-2-nitrobenzene	cat.# 32279
	4-chloro-3-nitrobenzotrifluoride	cat.# 32282
	tributylphosphate	cat.# 32280
	triphenylphosphate	cat.# 32281
Inj.:	1.0µL splitless (hold 0.4 min.) 4mm double gooseneck inlet liner (cat.# 20785)	
Inj. temp.:	250°C	
Carrier gas:	helium, constant flow, 6 psi head pressure	
Flow rate:	0.75mL/min.	
Linear velocity:	28cm/sec.	
Dead time:	1.82 min. @ 80°C	
Oven temp.:	80°C (hold 1.0 min.) to 150°C @ 7°C/min. (no hold) to 280°C @ 15°C/min. (hold 7 min.)	
Det.:	Agilent 5971A GC/MS	
Transfer line temp.:	280°C	
Scan range:	35–400 amu	
Solvent Delay:	5 min.	
Tuning compound:	PFTBA	
Ionization:	EI	

Compound	Conc. on-column (ng)*	Compound	Conc. on-column (ng)*
1. 4-chloro-3-nitrobenzotrifluoride (surr.)	100	12. chlorpyrifos	10
2. methamidophos	50	13. malathion	20
3. dichlorvos	50	14. quinalphos	10
4. 1-bromo-2-nitrobenzene (surr.)	100	15. tokuthion (prothiofos)	20
5. acephate	20	16. methidathion	20
6. demeton-S-methyl	20	17. profenfos	20
7. tributylphosphate (surr.)	100	18. ethion	20
8. omethoate	100	19. triphenylphosphate (surr.)	100
9. dimethoate	20	20. azinphos-methyl	40
10. tolclofos-methyl	10	21. pyrazophos	50
11. pirimiphos methyl	10		

**This mix was prepared for FPD analyses. Peaks will be approximately equivalent in height with FPD detection (0.32mm ID column).*

Ordering Information | Rtx®-CLPesticides Columns (Fused Silica)

ID	df (µm)	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter	60-Meter
0.10mm	0.10	-60 to 310/330°C	43101				
0.18mm	0.18	-60 to 310/330°C	42101		42102		
0.25mm	0.25	-60 to 320/340°C		11120		11123	11126
0.32mm	0.50	-60 to 320/340°C		11136		11139	
0.53mm	0.50	-60 to 300/320°C		11137		11140	

Rtx®-CLPesticides columns are available in **convenient and economical kits** that include a deactivated guard column and a connector. Refer to our general catalog, or contact your local Restek representative.

What's New in '02

This Year's New Reference Materials

By Ken Herwehe, Product Marketing Manager, Analytical Reference Materials

Environmental Reference Standards

✔Haloacetic acid and methyl ester mixtures (US EPA Method 552)
- 4 mixtures, internal standard, 3 surrogate standards, each as acid and methyl ester solutions.
- Formulation compliant with 12/01 Disinfectants and Disinfection Byproducts Rule.

✔8270 MegaMix™ and 8270 Matrix Spike Mix
- MegaMix™ of 76 compounds has 18-month shelf life.
- Includes 3- and 4-methylphenol at 0.5x concentration of other components.
- Eliminates mixing/minimizes preparation time for calibration and laboratory control samples.

✔Benzidines Mix
✔8270 System Performance Check C (SPCC) Mix
✔Skinner Volatiles Mixture
✔Skinner Semivolatiles Mixture
✔8260B Acetates Mixture
✔EPA 526 Semivolatiles in Drinking Water
✔EPA 528 Phenols in Drinking Water
✔Organochlorine Pesticides Mix AB #3
✔EPA 8082 PCB Congeners Mixture
✔ChemService Pesticides
- More than 900 pesticides and pesticide metabolites.
- Same day/next day shipping with Plus 1™ Service, always.*
- Convenient and economical—one order, one call.

Underground Storage Tank Monitoring

✔Octacosane Standard
✔Certified PAH in Diesel #2
✔BTEX Standard
- *m*- and *p*-xylene at 0.5x concentration of other components.

Petroleum Reference Standards

✔ASTM-2887-01 Calibration Mixes
- Meet requirements for revision ASTM 2887-01.
- C5-C44 at equal weight/weight concentrations.

Food, Flavor and Fragrance Mixtures

✔Food Industry FAMEs Mixture
✔NLEA FAMEs Mixture
✔*cis/trans* FAMEs Mixture

Forensics and Toxicology

✔Blood Alcohol Ethanol Standard
- Eight concentrations, 0.015 g/dL to 0.30 g/dL.
- Package sizes 5x1mL, 5mL, 20mL.

✔Explosives Solutions
- 15 single component solutions for US EPA 8095 and 8331.

International Environmental Reference Standards

✔ISO/DIS 9377-4 & H53 (in hexane)
✔Diesel #2/Mineral Oil
✔Diesel #2/Motor Oil
✔Extraction Solvent Stock Solution #1
✔Extraction Solvent Stock Solution #2
✔Florisil® Cartridge Quality Control Mix with Diesel #2/Mineral Oil
✔Florisil® Cartridge Quality Control Mix with Diesel #2/Motor Oil
✔Quality Control Standard Mixture (in acetone)
✔Stearyl Stearate Test Solution
✔System Performance Test Standard Mixture (*n*-alkanes)
✔International Petroleum Reference Standards
✔Canadian PHC / Pentacontane (in hexane)
- Meets CCME 2001 Petroleum Hydrocarbons in Soil Method-Tier 1.
- Primary calibration standards for quantifying four fractions.

for more info

for more information on any reference material listed here, contact the Technical Service Team at 800-356-1688 or 814-353-1300, ext. 4, contact your local Restek representative, or visit us online at **www.restekcorp.com.**

**Orders in by 3pm Eastern Time are shipped the same day, subject to product availability.*

Certified PAHs in Diesel

- Confirms diesel #2 TPH and priority PAHs in a single analysis.
- Certificate of Analysis includes concentration of TPH and certified concentrations of individual PAHs.
- Complete data packs available.

Certified PAHs in Diesel

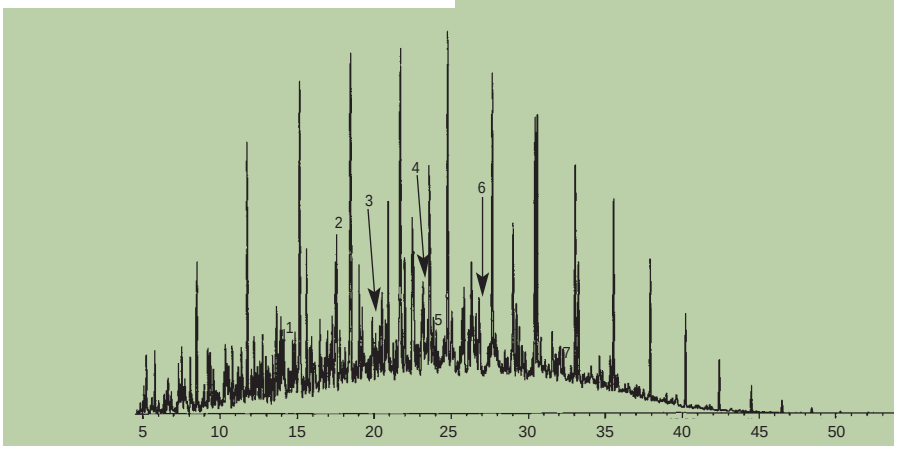
Certified PAHs	Typical Certified Conc.** (ppm)
acenaphthene	20
acenaphthylene	14
fluorene	32
1-methylnaphthalene	269
2-methylnaphthalene	180
naphthalene	90
phenanthrene	47

50,000ppm diesel #2 in methylene chloride, typical PAH concentrations listed above, 1mL/ampul

Each	5-pk.	10-pk.
31673	31673-510	—
	with data pack	
31673-500	31673-520	31773

Column: 30m, 0.25mm ID, 0.25µm Rtx®-5 with 5-meter Integra-Guard™ guard column (cat.# 10223-124)
Carrier gas: helium @ 1mL/min.
Temp. program: 75°C to 275°C @ 4°C/min.
Inj. temp: 250°C
Det. temp.: 300°C
Detector type: MSD

1. naphthalene
2. 1-methylnaphthalene
3. 2-methylnaphthalene
4. acenaphthylene
5. acenaphthene
6. fluorene
7. phenanthrene



***Concentration varies lot to lot. See Certificate of Analysis for certified concentrations.*

What's New in This Issue

Skinner Semivolatiles & VOAs, Chlorinated Pesticides, PCBs, FAMEs

NLEA FAME Mix (28 components)

- Use as a calibration standard for AOAC Method 996.06.

Compound	Conc. (%)	Compound	Conc. (%)
C4:0	1.5	C18:1(*trans*)	2.5
C6:0	1.5	C18:1(*cis*)	15.0
C8:0	2.0	C18:2(*trans*)	2.5
C10:0	2.5	C18:2(*cis*)	10.0
C11:0	2.5	C18:3	5.0
C12:0	5.0	C20:0	2.5
C13:0	2.5	C20:1	1.5
C14:0	2.5	C20:5	2.5
C14:1(*cis*-9)	1.5	C22:0	2.5
C15:0	1.5	C22:1	1.5
C16:0	10.0	C22:6	2.5
C16:1(*cis*-9)	5.0	C23:0	1.5
C17:0	2.5	C24:0	2.5
C18:0	5.0	C24:1	2.5

30mg/mL each in methylene chloride, 1mL/ampul

ea.
35078

PCB Congener Mix, Method 8082A

(19 components)

2-chlorobiphenyl (BZ #1)
2,3-dichlorobiphenyl (BZ #5)
2,2',5-trichlorobiphenyl (BZ #18)
2,4',5-trichlorobiphenyl (BZ #31)
2,2',3,5'-tetrachlorobiphenyl (BZ #44)
2,2',5,5'-tetrachlorobiphenyl (BZ #52)
2,3',4,4'-tetrachlorobiphenyl (BZ #66)
2,2',3,4,5'-pentachlorobiphenyl (BZ #87)
2,2',4,5,5'-pentachlorobiphenyl (BZ #101)
2,3,3',4',6-pentachlorobiphenyl (BZ #110)
2,2',3,4,4',5'-hexachlorobiphenyl (BZ #138)
2,2',3,4,5,5'-hexachlorobiphenyl (BZ #141)
2,2',3,5,5',6-hexachlorobiphenyl (BZ #151)
2,2',4,4',5,5'-hexachlorobiphenyl (BZ #153)
2,2',3,3',4,4',5-heptachlorobiphenyl (BZ #170)
2,2',3,4,4',5,5'-heptachlorobiphenyl (BZ #180)
2,2',3,4,4',5',6-heptachlorobiphenyl (BZ #183)
2,2',3,4',5,5',6-heptachlorobiphenyl (BZ #187)
2,2',3,3',4,4',5,5',6-nonachlorobiphenyl (BZ #206)

100µg/mL each in isooctane, 1mL/ampul

Each	5-pk.	10-pk.
32416	32416-510	—
	with data pack	
32416-500	32416-520	32516

Organochlorine Pesticide Mix AB #3

(20 components)

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

2,000µg/mL each in hexane:toluene (1:1), 1mL/ampul

Each	5-pk.	10-pk.
32415	32415-510	—
	with data pack	
32415-500	32415-520	32515

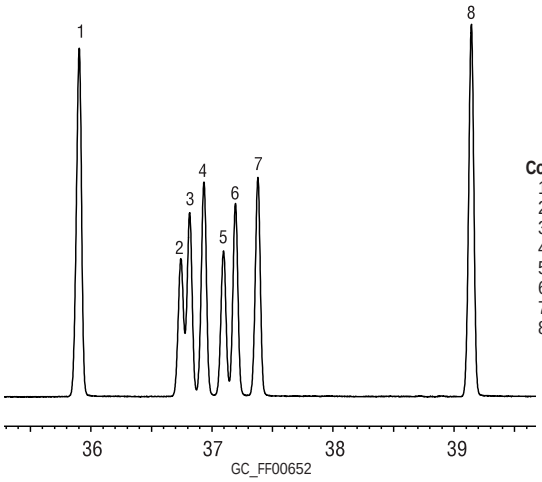
cis/*trans* FAME Mix (8 components)

- Use to identify the isomers of octadecenoic acid.

Compound	Conc. (%)
methyl elaidate (C18:1 *trans*-9)	10.0
methyl linoleate (C18:2 *cis*-9,12)	20.0
methyl oleate (C18:1 *cis*-9)	10.0
methyl petroselinate (C18:1*cis*-6)	8.0
methyl petroselaidate (C18:1*trans*-6)	8.0
methyl stearate (C18:0)	20.0
methyl transvaccenate (C18:1 *trans*-11)	12.0
methyl vaccenate (C18:1 *cis*-11)	12.0

10mg/mL total in methylene chloride, 1mL/ampul

ea.
35079



See page 11 for more information on FAME analysis

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

Compound	% in Mix
1. C18:0 methyl stearate	20.0
2. C18:1 methyl petroselaidate (*trans*-6)	8.0
3. C18:1 methyl elaidate (*trans*-9)	10.0
4. C18:1 methyl transvaccenate (*trans*-11)	12.0
5. C18:1 methyl petroselinate (*cis*-6)	8.0
6. C18:1 methyl oleate (*cis*-9)	10.0
7. C18:1 methyl vaccenate (*cis*-11)	12.0
8. C18:2 methyl linoleate (*cis*-9,12)	20.0

Skinner List - SV (33 components)

acenaphthene
anthracene
benzo(a)anthracene
benzo(b)fluoranthene
benzo(k)fluoranthene
benzo(a)pyrene
bis(2-ethylhexyl)phthalate (dioctylphthalate)
chrysene
dibenzo(a,h)acridine
dibenzo(a,h)anthracene
di-*n*-butylphthalate
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
diethylphthalate
2,4-dimethylphenol
dimethylphthalate
2,4-dinitrophenol
fluoranthene
fluorene
indene
indeno(1,2,3-cd)pyrene
1-methylnaphthalene
2-methylphenol (*o*-cresol)
3-methylphenol (*m*-cresol)*
4-methylphenol (*p*-cresol)*
naphthalene
4-nitrophenol
phenanthrene
phenol
pyrene
pyridine
quinoline

2,000µg/mL each in methylene chloride, 1mL/ampul
**(3- & 4-methylphenol at 1,000µg/mL each)*

Each	5-pk.	10-pk.
31690	31690-510	—
	with data pack	
31690-500	31690-520	31790

Skinner List - Volatiles (19 components)

benzene
2-butanone (MEK)
carbon disulfide
chlorobenzene
chloroform
1,2-dibromoethane (EDB)
1,1-dichloroethane
1,2-dichloroethane
1,4-dioxane
ethylbenzene
methyl *tert*-butyl ether (MTBE)
styrene
tetrachloroethylene
toluene
1,1,1-trichloroethane
trichloroethylene
m-xylene*
o-xylene
p-xylene*

2,000µg/mL each in methanol, 1mL/ampul
**(m- & p-xylene at 1,000µg/mL each)*

Each	5-pk.	10-pk.
30491	30491-510	—
	with data pack	
30491-500	30491-520	30591

SV System Performance Check Mix (SPCC), US EPA Method 8270C/D

(4 components)

2,4-dinitrophenol
hexachlorocyclopentadiene
4-nitrophenol
N-nitroso-di-*n*-propylamine

2,000µg/mL each in methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
31689	31689-510	—
	with data pack	
31689-500	31689-520	31789

New Ultra HPLC Column for Carbamates Analysis

Unique Stationary Phase Separates 10 Carbamates in 10 Minutes

by Greg France, HPLC Marketing Manager, Rebecca Wittrig, Ph.D., Food, Flavor, & Fragrance Innovations Chemist, and Vernon Bartlett, HPLC Innovations Manager

- ✔ Reduce analysis time by 70% versus a C18 column.
- ✔ Greatly reduce solvent purchase and disposal costs.
- ✔ Compatible with LC/MS detection.*

The traditional HPLC column for analyses of carbamates is a 250 x 4.6mm column containing a 5µm C18 packing. This column separates 10 target carbamates and an internal standard, but the price paid for the separation is steep—the analysis time is approximately 35 minutes. Several column manufacturers have managed to reduce the analysis time by packing a 4µm C18 particle in a 150mm column. This configuration reduces the analysis time to approximately 25 minutes.

Changing the physical parameters of a column is a brute force way to improve analyte separation or reduce analysis time. A better way to improve a method is to examine the analytes and determine if an alternative stationary phase or mobile phase composition would provide a better or faster separation. After the optimum stationary phase and mobile phase are selected, the analysis can be fine-tuned by adjusting the column length and/or the particle size.

With a chemistry-based approach in mind, Restek chemists developed the Ultra Carbamate column specifically for carbamates analysis. The new column has a unique stationary phase that, when bonded to 3µm silica, creates a packing that separates 10 target carbamates in under 10 minutes (Figure 1). The new stationary phase is compatible with LC/MS detection (Figure 2). The mobile phases used to obtain these separations are binary gradients consisting of water, methanol, and acetonitrile, without (Figure 1) or with ammonium formate (Figure 2).

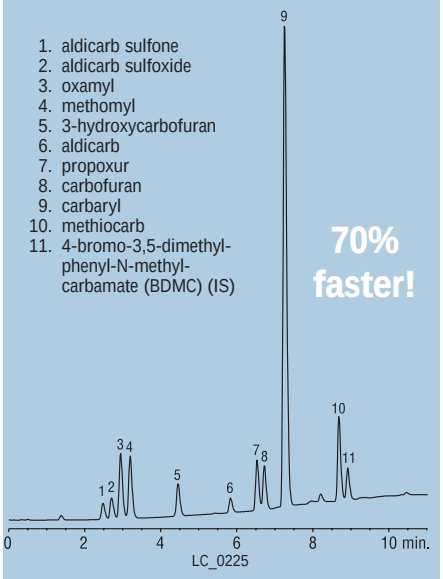
An Ultra Carbamate column can process as many as 3 to 4 samples per hour, versus less than 2 samples per hour on a traditional C18 column. In addition to increased sample throughput, faster analyses significantly reduce solvent usage—and the costs of disposing of the solvent waste after the analysis. If you have been analyzing carbamates with a general-purpose C18 column, we think you will be highly impressed with the performance of an Ultra Carbamate column—and the increased sample throughput and solvent savings will quickly pay for your new column.

Figure 1

An Ultra Carbamate column reduces analysis time by 70%.

1. aldicarb sulfone
2. aldicarb sulfoxide
3. oxamyl
4. methomyl
5. 3-hydroxycarbofuran
6. aldicarb
7. propoxur
8. carbofuran
9. carbaryl
10. methiocarb
11. 4-bromo-3,5-dimethyl-phenyl-N-methyl-carbamate (BDMC) (IS)

70% faster!

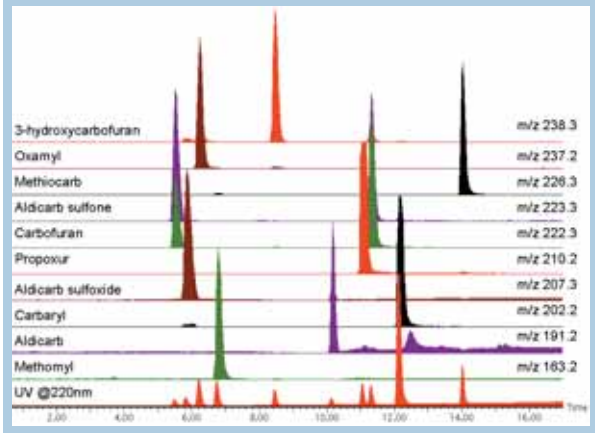


Column:	Ultra Carbamate
Catalog #:	9177355
Dimensions:	50 x 4.6mm
Particle size:	3µm
Pore size:	100Å
Conditions:	
Inj.:	5µL
Conc.:	50µg/mL
Solvent:	methanol
Restek standards:	
Catalog# 32274 (IS) and 32273 mixed 50:50	
Mobile Phase:	A: 90:10 water:methanol
	B: 90:10 methanol:acetonitrile
	Time (min): %B
	0 10
	10 90
Flow:	1.5mL/min
Temp.:	27°C
Det.:	UV @ 220nm

*Also compatible with post-column derivitization and fluorescence detection, if total system dead volume is less than 650µL, inclusive of post-column reactors.

Figure 2

An Ultra Carbamate column provides rapid, excellent results in LC/MS analyses.



Compound	Ion	Cone V
1. aldicarb sulfone	223.3	25V
2. aldicarb sulfoxide	207.3	18V
3. oxamyl	237.2*	10V
4. methomyl	163.2	15V
5. 3-hydroxycarbofuran	238.3	15V
6. aldicarb	191.2	8V
7. propoxur	210.2	18V
8. carbofuran	222.3	22V
9. carbaryl	202.2	18V
10. methiocarb	226.3	19V

*Ammonium adduct; others [M+H]+ ions.

Column:	Ultra Carbamate
Catalog #:	9177315
Dimensions:	100 x 4.6mm
Particle size:	3µm
Pore size:	100Å
Inj.:	10µL
Sample:	531.1 Carbamate Pesticide Calibration Mix (cat.# 32273)
Conc.:	100µg/mL each
Solvent:	methanol
Mobile Phase:	
A: 90:10 water:methanol + 10mM ammonium formate	
B: 10:90 acetonitrile:methanol + 10mM ammonium formate	
Gradient:	90:10 A:B to 10:90 A:B from 0-15 min.
Flow:	1mL/min. (0.75mL/min. to UV, 0.25mL/min. to MS)
Temp.:	ambient

MS Conditions:

Det.:	Micromass ZMD
Mode:	ESI+
Capillary V:	3.50
Extractor:	4.0
Ion Energy:	0.4
Multiplier:	650
Source Temp.:	100ºC
Desolv. Temp.:	250ºC
Gas Flow:	490 L/hr.

Ordering Information | 3µm Ultra Carbamate HPLC Columns

Description	Length	4.6mm ID
Ultra Carbamate	50mm	9177355
Ultra Carbamate	100mm	9177315

Internal Standard

4-bromo-3,5-dimethylphenyl-N-methylcarbamate (BDMC)

100µg/mL in methanol, 1mL/ampul

Each	5-pk.	10-pk.
32274	32274-510	—
	with data pack	
32274-500	32274-520	32374

531.1 Carbamate Pesticide Calibration Mixture (10 components)

aldicarb	3-hydroxycarbofuran
aldicarb sulfone	methiocarb
aldicarb sulfoxide	methomyl
carbaryl	oxamyl
carbofuran	propoxur (baygon)

100µg/mL each in methanol, 1mL/ampul

Each	5-pk.	10-pk.
32273	32273-510	—
	with data pack	
32273-500	32273-520	32373

Analyzing Fatty Acid Methyl Esters (FAMEs) by GC

Using Restek Capillary Columns and Analytical Reference Materials

by Rebecca Wittrig, Ph.D., Food, Flavor, & Fragrance Innovations Chemist

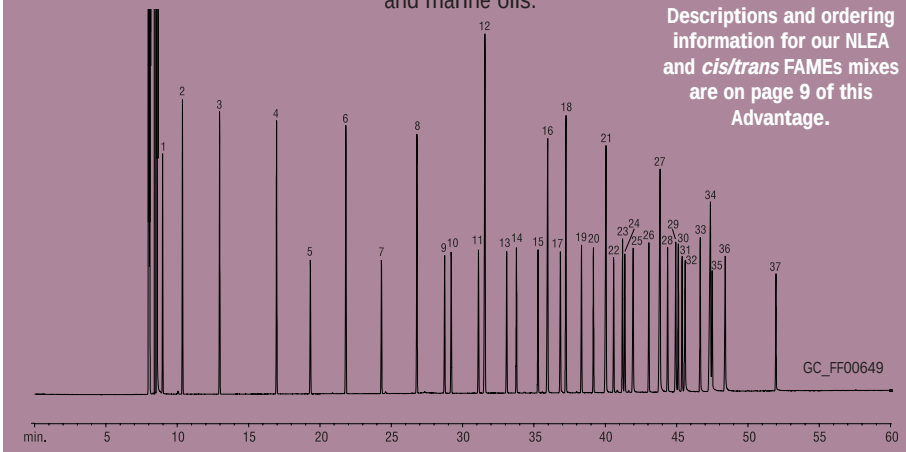
✔ Resolve individual *cis* and *trans* isomers on our new 100-meter Rt-2560 column.
✔ Separate saturated/unsaturated FAMEs on a FAMEWAX™ column.
✔ New Food Industry FAME Mix includes methyl esters of 37 common fatty acids in animal, vegetable, and marine oils.
✔ Use new qualitative *cis/trans* FAME Mix to identify C18:1 isomers.

Fatty acid methyl ester (FAME) analyses are an important tool for characterizing fats and oils and for determining the total fat content in foods. A new capillary GC column and three new reference mixtures from Restek can help you obtain the best results for these challenging assays.

Individual *cis* and *trans* isomers of unsaturated FAMEs, such as octadecenoic acid (C18:1) or octadecadienoic acid (C18:2), are resolved on an Rt-2560 column—our new 100-meter biscyanopropyl phase column. An analysis of C18:1 *cis* and *trans* isomers is shown on page 9 of this Advantage. The ability to resolve these isomers makes an Rt-2560 column the column of choice for analyzing partially hydrogenated fats and oils; the column meets the requirements of AOAC Method 996.06. Figure 1 shows a separation of 37 FAMEs commonly encountered by food quality chemists, obtained by using an Rt-2560 column.

Figure 1

37-Component Food Industry FAME Mix includes common components of animal, vegetable, and marine oils.



Descriptions and ordering information for our NLEA and *cis/trans* FAMEs mixes are on page 9 of this Advantage.

Compound	% in Mix
1. C4:0 methyl butyrate	4.0
2. C6:0 methyl hexanoate	4.0
3. C8:0 methyl octanoate	4.0
4. C10:0 methyl decanoate	4.0
5. C11:0 methyl undecanoate	2.0
6. C12:0 methyl laurate	4.0
7. C13:0 methyl tridecanoate	2.0
8. C14:0 methyl myristate	4.0
9. C14:1 methyl myristoleate (*cis*-9)	2.0
10. C15:0 methyl pentadecanoate	2.0
11. C15:1 methyl pentadecenoate (*cis*-10)	2.0
12. C16:0 methyl palmitate	6.0
13. C16:1 methyl palmitoleate (*cis*-9)	2.0
14. C17:0 methyl heptadecanoate	2.0
15. C17:1 methyl heptadecenoate (*cis*-10)	2.0
16. C18:0 methyl stearate	4.0
17. C18:1 methyl elaidate (*trans*-9)	2.0
18. C18:1 methyl oleate (*cis*-9)	4.0
19. C18:2 methyl linoleaidate (*trans*-9,12)	2.0
20. C18:2 methyl linoleate (*cis*-9,12)	2.0
21. C18:3 methyl γ-linolenate (*cis*-6,9,12)	2.0
22. C20:0 methyl arachidate	4.0
23. C20:1 methyl eicosenoate (*cis*-11)	2.0
24. C18:3 methyl linolenate (*cis*-9,12,15)	2.0
25. C21:0 methyl heneicosanoate	2.0
26. C20:2 methyl eicosadienoate (*cis*-11,14)	2.0
27. C20:3 methyl eicosatrienoate (*cis*-11,14,17)	2.0
28. C22:0 methyl behenate	4.0
29. C22:1 methyl erucate (*cis*-13)	2.0
30. C20:3 methyl eicosatrienoate (*cis*-11,14,17)	2.0
31. C20:4 methyl arachidonate (*cis*-5,8,11,14)	2.0
32. C23:0 methyl tricosanoate	2.0
33. C22:2 methyl docosadienoate (*cis*-13,16)	2.0
34. C20:5 methyl eicosapentaenoate (*cis*-5,8,11,14,17)	2.0
35. C24:0 methyl lignocerate	4.0
36. C24:1 methyl nervonate (*cis*-15)	2.0
37. C22:6 methyl docosahexaenoate (*cis*-4,7,10,13,16,19)	2.0

For analyzing mixtures of saturated and unsaturated fatty acid methyl esters, stationary phases consisting of polyethylene glycol (e.g., Carbowax®) typically are used. FAMEWAX™ columns offer excellent selectivity for analyses of polyunsaturated fatty acids. The polyethylene glycol stationary phase is especially capable of resolving Omega-3 polyunsaturated FAMEs, such as eicosapentenoic acid (C20:5) and docosahexenoic acid (C22:6). FAMEWAX™ columns combine excellent resolution of polyunsaturated FAMEs with significantly reduced analysis times, compared to traditional Carbowax® stationary phases.

In addition to our new Rt-2560 capillary column for *cis* and *trans* isomers analysis, we are introducing three new FAMEs mixtures to support food and nutraceuticals testing. Our 37-component Food Industry FAME Mix is a complete profile of the common fatty acids in animal, vegetable, and marine oils, including many polyunsaturated FAMEs, such as C20:5 and C22:6. Figure 1 is a chromatographic separation of this mix. New NLEA FAME Mix was designed with AOAC Method 996.06 in mind; it can be used as the calibration standard for this Fat by Fatty Acid Composition method. Use our qualitative *cis/trans* FAME Mix to identify the *cis* and *trans* isomers of octadecenoic acid (C18:1). For descriptions and ordering information for the latter two mixes, and an analysis of the *cis/trans* FAME Mix, see page 9.

Before you attempt to characterize a fat or oil, contact Restek for capillary columns and reference materials that can help you obtain the data you need.

Food Industry FAME Mix (37 components)

Component	Component
C4:0	C18:2(all-*cis*-9,12)
C6:0	C18:3(all-*cis* 6,9,12)
C8:0	C18:3(all-*cis* 9,12,15)
C10:0	C20:0
C11:0	C20:1(*cis*-11)
C12:0	C20:2(all-*cis* 11,14)
C13:0	C20:3 (all-*cis* 8,11,14)
C14:0	C20:3(all-*cis* 11,14,17)
C14:1(*cis*-9)	C20:4(all-*cis* 5,8,11,14)
C15:0	C20:5 (all-*cis* 5,8,11,14,17)
C15:1(*cis*-10)	C21:0
C16:0	C22:0
C16;1(*cis*-9)	C22:1(*cis* 13)
C17:0	C22:2(all-*cis* 13,16)
C17:1(*cis*-10)	C22:6 (all-*cis* 4,7,10,13,16,19)
C18:0	C23:0
C18:1(*trans*-9)	C24:0
C18:1(*cis*-9)	C24:1(*cis*-15)
C18:2(all-*trans*-9.12)	

In methylene chloride, 1mL/ampul. For concentrations, see Figure 1.

	ea.
35077	

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

Ordering Information | Rt-2560 Column (Fused Silica)

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

Ordering Information | FAMEWAX™ Columns (Fused Silica)

ID	df (µm)	temp. limits	30-Meter
0.25mm	0.25	20 to 250°C	12497
0.32mm	0.25	20 to 250°C	12498
0.53mm	0.50	20 to 250°C	12499

Improved Responses for Chlorinated Pesticides

Using a Drilled Uniliner® GC Inlet Liner and Rtx®-CLPesticides Columns

by Lydia Nolan, Environmental Innovations Chemist

✔ Less breakdown of active compounds, for more accurate results.
✔ Greater sensitivity, for lower detection limits.
✔ Minimal injection port discrimination.
✔ Liner accommodates direct or splitless injections.

Inlet activity is the primary drawback to using hot flash injection when analyzing chlorinated pesticides by GC. Residues of heavier and non-volatile materials often build up throughout the injection port, leaving a reactive surface that can cause compounds such as endrin and DDT to break down. An inlet liner that provides a press-fit connection between the column and the liner eliminates reactive surface problems associated with the bottom portion of the injection port. A physical connection between the column and the liner also improves sensitivity, by minimizing injection port discrimination.

This article summarizes the advantages of using a Restek Drilled Uniliner® inlet liner (Figure 1) and an Rtx®-CLPesticides/Rtx®-CLPesticides2 column pair for chlorinated pesticides analysis. Included are comparisons of data obtained using a Drilled Uniliner® inlet liner to data obtained using a conventional splitless inlet liner, and a splitless inlet liner packed with fused silica wool.

Figure 1

The drilled hole in a Uniliner® injection port liner makes direct injection possible with EPC systems by equalizing pressure in the injection port.

Procedures detailed in US EPA Methods 8081 and 8000 require each laboratory to document that the quantification results they generate are reliable, precise, and accurate. Beginning with a five-point calibration curve, a calibration factor is calculated for each analyte. The relative standard deviation (RSD) should be no more than 20% for each analyte. Using a Drilled Uniliner® inlet liner, mean RSD values for 20 chlorinated pesticides for the Rtx®-CLPesticides column and for the Rtx®-CLPesticides2 column were 4.1% and 4.5% respectively. High values were 12.5% and 14.2%.

A calibration standard must be analyzed and quantified regularly. The concentration of each analyte in this standard should be within ±15% of the "true" value. The accuracy of data obtained by using a Drilled Uniliner® inlet liner was tested with pesticide standard mix at the 20/40/200ng/mL concentration level (Figure 2). For either column, the mean percent difference from the "true" value for each analyte was only ±2.2%, well within the acceptable limits.

Finally, because some analytes readily break down as the injection port inlet becomes more contaminated (e.g., endrin and DDT in this analysis), a performance evaluation mix must be analyzed and breakdown for each analyte calculated. Breakdown should not exceed 15%. The Drilled Uniliner® inlet liner reduces endrin and DDT breakdown, relative to the splitless liners (Table 1), because it shields the analytes from contact with active surfaces outside the inlet liner. Wool packing in the splitless liner makes this problem worse, because it greatly increases the surface area and potential active sites.

In addition to reducing variability and increasing accuracy of calibration data, the Drilled Uniliner® inlet liner increases overall response for individual analytes, because injection port discrimination is greatly reduced. This enhances minimum detection levels, compared to standard splitless inlet liners. This is most apparent from the area counts for the last eluting analyte, decachlorobiphenyl, which were greater by 18-39%, relative to area counts for injections made on the splitless liners (Table 1).

By eliminating the bottom of the injector from the sample pathway, a Drilled Uniliner® inlet liner makes the pathway more inert. This reduces breakdown of labile analytes, such as endrin and DDT and increases accuracy and precision. For analysts using hot flash injection techniques in analyses of chlorinated pesticides, or other labile analytes, these results clearly indicate that the Drilled Uniliner® inlet liner is the liner of choice.

Table I

Lowest breakdown of endrin and DDT, and highest responses for analytes, using a Drilled Uniliner® inlet liner.

% Breakdown

Analyte	Column	Drilled Uniliner®	4mm splitless	4mm splitless with wool
Endrin	Rtx®-CLPesticides	4.4	4.7	9.8
	Rtx®-CLPesticides2	4.9	6.9	8.3
DDT	Rtx®-CLPesticides	0.2	0.3	2.6
	Rtx®-CLPesticides2	0.3	0.9	3.1

Response*

Analyte	Column	Drilled Uniliner®	4mm splitless with wool	4mm splitless
Tetrachloro-*m*-xylene (TCMX)	Rtx®-CLPesticides	147	111	106
	Rtx®-CLPesticides2	191	167	162
Decachloro-biphenyl (DCB)	Rtx®-CLPesticides	150	119	108
	Rtx®-CLPesticides2	209	177	166

*Mean response (n=2); value in table x 10^3 = response units.

Organochlorine Pesticide Mix AB #2

(20 components)

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

In hexane:toluene (1:1), 1mL/ampul

Each	5-pk.	10-pk.
32292	32292-510	—
	with data pack	
32292-500	32292-520	32392

Pesticide Performance Evaluation Mix w/Surrogates (8 components)

04.1 and 3/90 SOW

α-BHC	1µg/mL
β-BHC	1
γ-BHC (lindane)	1
4,4'-DDT	10
decachlorobiphenyl	2
endrin	5
methoxychlor	25
2,4,5,6-tetrachloro-*m*-xylene	2

In hexane, 1mL/ampul

Each	5-pk.	10-pk.
32074 $28.90	32074-510	—
	with data pack	
32074-500 $39.20	32074-520	32174

Pesticide Surrogate Mix

04.1, 3/90, 4/89, and 2/88 SOW

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

Each	5-pk.	10-pk.
32000 $18.60	32000-510	—
	with data pack	
32000-500 $28.90	32000-520	32100

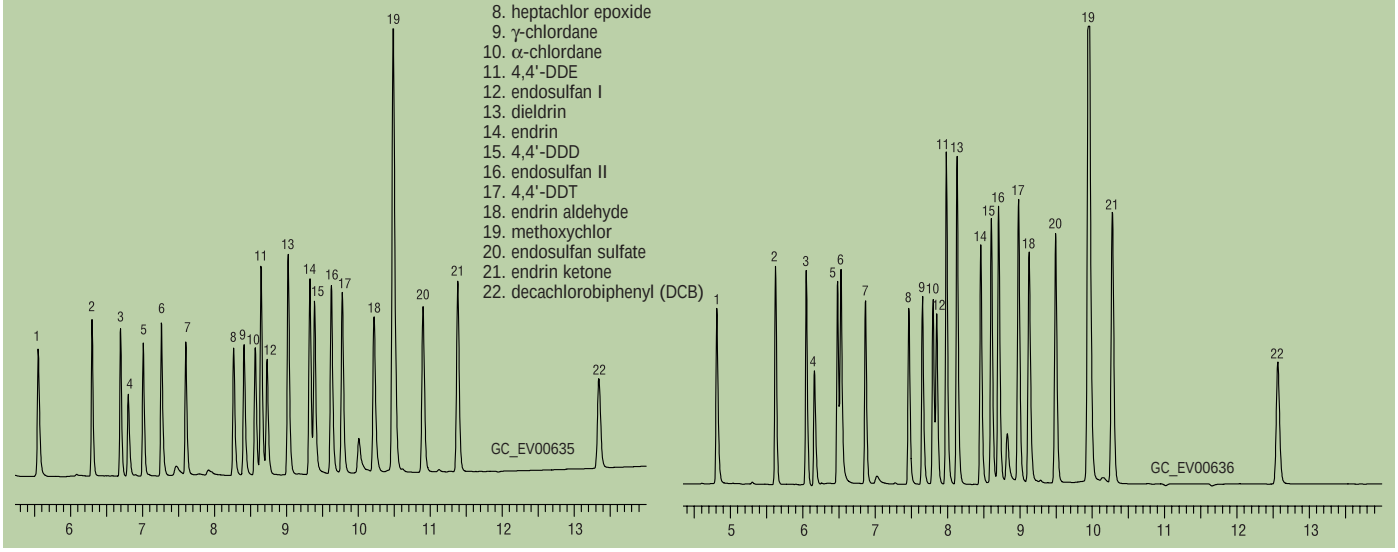
Figure 2

Chlorinated pesticides show excellent response using the Drilled Uniliner® inlet liner and Rtx®-CLPesticides columns—even at the 20/40/200ng/mL concentration level.

Column:	Rtx®-CLPesticides 30m, 0.32 ID, 0.50µm (cat.# 11139)
Sample:	20–400µg/mL Organochlorine Pesticide Mix AB #2 (cat.# 32292) in hexane:toluene (1:1) 200µg/mL Pesticide Surrogate Mix (cat.#32000) in acetone
Inj.:	1.0µL direct, open-top drilled Uniliner® inlet liner (cat.# 21054)
Inj. temp.:	225°C
Carrier gas:	helium, constant pressure
Linear velocity:	53cm/sec. @ 110°C
Oven temp.:	110°C (hold 1min.) to 245°C @ 20°C/min. to 310°C (hold 1 min.)
Det.:	ECD @ 330°C

Column:	Rtx®-CLPesticides2 30m, 0.32 ID, 0.25µm (cat.# 11324)
Sample:	20–400µg/mL Organochlorine Pesticide Mix AB #2 (cat.# 32292) in hexane:toluene (1:1) 200µg/mL Pesticide Surrogate Mix (cat.#32000) in acetone
Inj.:	1.0µL direct, open-top drilled Uniliner® inlet liner (cat.# 21054)
Inj. temp.:	225°C
Carrier gas:	helium, constant pressure
Linear velocity:	53cm/sec. @ 110°C
Oven temp.:	110°C (hold 1min.) to 245°C @ 20°C/min. to 310°C (hold 1 min.)
Det.:	ECD @ 330°C

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



Ordering Information | Rtx®-CLPesticides Columns (Fused Silica)

ID	df (µm)	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter	60-Meter
0.10mm	0.10	-60 to 310/330°C	43101				
0.18mm	0.18	-60 to 310/330°C	42101		42102		
0.25mm	0.25	-60 to 320/340°C		11120		11123	11126
0.32mm	0.50	-60 to 320/340°C		11136		11139	
0.53mm	0.50	-60 to 300/320°C		11137		11140	

Ordering Information | Rtx®-CLPesticides2 Columns (Fused Silica)

ID	df (µm)	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter	60-Meter
0.10mm	0.10	-60 to 310/330°C	43301		43302		
0.18mm	0.14	-60 to 310/330°C	42301		42302		
0.25mm	0.20	-60 to 320/340°C		11320		11323	11326
0.32mm	0.25	-60 to 320/340°C		11321		11324	
0.53mm	0.42	-60 to 300/320°C		11337		11340	

DI Liners for Agilent 5890 & 6890 GCs (For 0.25/0.32/0.53mm ID Columns)	ID*/OD & Length (mm)	cat.#/price ea.	cat.#/price 5-pk.
Drilled Uniliner®	4.0 ID 6.3 OD x 78.5	21054	21055
Siltek™ Drilled Uniliner®	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5
Siltek™ 1mm Drilled Uniliner®	1.0 ID 6.3 OD x 78.5	21390-214.1	21391-214.5

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

**Nominal ID at syringe needle expulsion point.*

for more info

for additional data from these analyses, request Applications Note 59487.

Rtx®-CLPesticides columns are available in **convenient and economical kits** that include a deactivated guard column and a connector. Refer to our general catalog, or contact your local Restek representative.

Restek's Customer Commitment

Plus 1™ PLUS ONE

Plus 1™ means that we will surpass your expectations every time you contact us. Looking for the solution to your tough analytical problem or placing a late-day order? Contact us to experience Plus 1™ service today!

Peak Performers

GC/MS Tools and Supplies

by Donna Lidgett, GC Accessories Marketing Manager

MSD Conversion Fitting

- A flat, soft aluminum sealing ring deforms and butt-seals against the MSD interface.
- A standard Vespel® ferrule seals the column and 1/16-inch stainless steel nut.
- Fitting is constructed of nickel-plated brass for longevity and softness.
- Use any standard Vespel® or Vespel®/graphite 1/16-inch ferrule.
- Includes a 1/16-inch stainless steel nut and two replacement sealing rings. Order ferrules separately.

The butt-seal in Agilent's MSD interface also seals the MSD source to the capillary column. This system is prone to leakage. Restek's MSD conversion fitting is designed with two separate seals to reduce the chance of leaks: a crunch washer seals the MSD conversion fitting to the source, and a ferrule seals the capillary tubing to the conversion fitting.

Description	qty.	cat.#
MSD Conversion Fitting	ea.	21314
MSD Conversion Fitting Replacement Ring Seals	2-pk.	21313

MSD Source Nut

The nut bore has been changed from 0.8mm to 1.2mm to permit easy removal of ferrules with a standard tapered-needle file (cat.# 20106). The nuts match the manufacturer's original part specifications and are made of brass to prevent thread-stripping on the transfer line.
(Similar to Agilent part # 05988-20066.)

Description	qty.	cat.#
(Detector) MSD Source Nut	2-pk.	20643

Capillary Installation Tool for Agilent 5973 MS

new!

- Pre-seats ferrule onto column for consistent installations.
- Made from high-quality stainless steel.

Install the nut and ferrule onto the column, then insert the column through the installation tool, exposing several centimeters at the exit end.

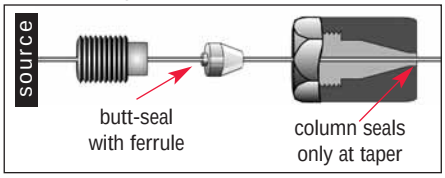
Tighten the nut.

Score and remove the exposed end of the column making sure of a clean, square cut, then loosen the nut.

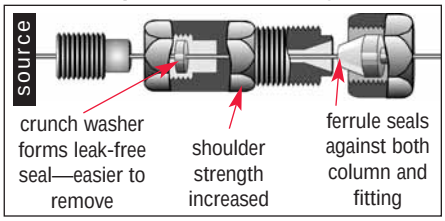
The ferrule will be properly seated and should remain in place when light force is applied. Install the column into the GC/MS interface.

Description	Similar to Agilent part #	qty.	cat.#
Capillary Installation Tool for Agilent 5973 MS	G1099-20039	ea.	21894

Agilent MSD Interface



Improved Restek Design



Ion Source Cleaning Powder

new!

- Use to clean the ion source when you encounter poor sensitivity and inadequate abundances at high masses.
- Clean surfaces that contact the sample or ion beam with a slurry of this powder and reagent-grade methanol on a cotton swab, or an abrasive paper.

(Similar to Agilent part# 8660-0791.)

Description	qty.	cat.#
Aluminum Oxide Powder	1 kg.	22685

Rough Pump Oil #19 for MSD Pumps, Oil Vacuum Pump

new!

- Formulated from crude oil stocks known for durability and line lubricating qualities.
- Use in Agilent 5973/5972/5971/ and GCD mass spec systems, or in other manufacturers' MSD systems that require rough pump oil.
- Replace oil in the foreline rough pump every six months (average use).

(Similar to Agilent part# 6040-0834.)

Description	qty.	cat.#
Rough Pump Oil for MSD Pumps	1 liter	22687

Autosampler Supplies for Agilent 7673A & 7673B

✔ Meet OEM specifications.

Autosampler Syringe Pulley Belt

new!

Description	qty.	cat.#
Autosampler Syringe Pulley Belt for Agilent 7673A and 7673B	ea.	22695

Plunger Motor Belt

Description	Similar to Agilent part #	qty.	cat.#
Plunger Motor Belt for Agilent 7673A and 7673B	1500-1010	ea.	22692

cool tools

For Easier GC & HPLC Maintenance Try These New Tools from Restek

by Brad Rightnour and Michael Goss, Instrument Innovations Team

Injector Wrench for Agilent 5890/6890/6850 GCs

- Use to remove the septum nut and weldments during GC maintenance.
- High-quality stainless steel construction.

(Similar to Agilent part #19251-00100.)
cat.# 22065, (ea.) $28

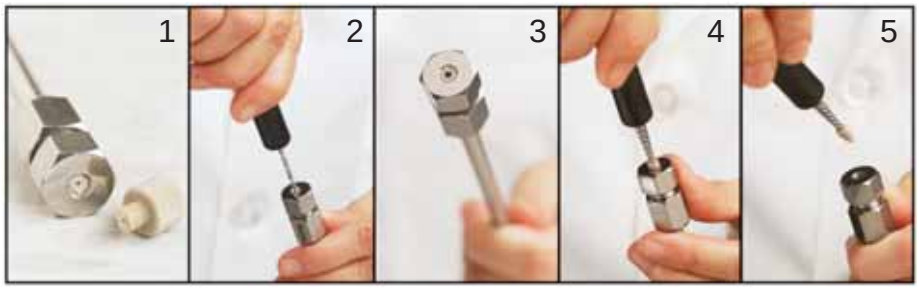
Use the smaller end to remove the septum nut.

Use the larger end to tighten the split/splitless weldment nut.

PEEK® Fitting Extractor

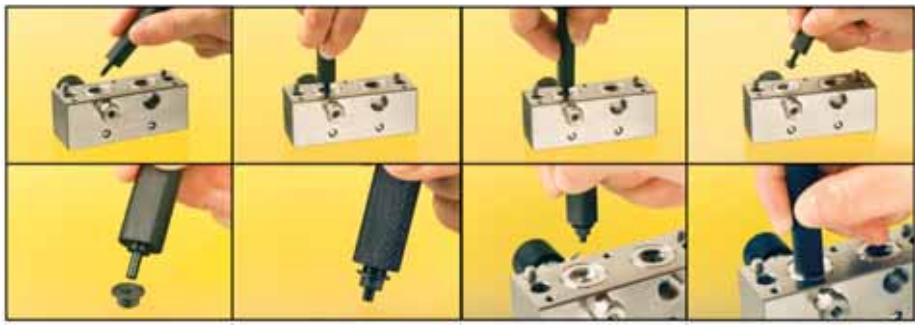
1. PEEK® nut or column plug has snapped off in the analytical HPLC column.
2., 3. Use the hand drill to enlarge the bore in the fitting.
4. Turn the reverse threaded, tapered bit into the fitting in a counterclockwise direction.
5. The bit will grab the PEEK® fitting and remove it from the column—saving your column.

cat.# 25325, (ea.)



HPLC Piston Seal Insertion Tool

Top: Using the tool to remove the old piston seal from the housing.
Insert the threaded end of the tool into the exposed seal and turn in a clockwise direction until the tool grips the seal. Pull it straight out of the housing.
Bottom: Using the tool to insert a new seal.
Insert the smooth end of the tool into the new piston seal. Firmly push the seal straight into the pump housing. The stepped tip of the tool ensures a precise depth fit into the pump housing. This prevents expansion/deformation of the seal on insertion and helps maximize the life of the seal.
cat.# 21356, (ea.)



GC Accessories Organizer

- Ideal for keeping GC accessories and supplies easy to find.
- Built-in syringe and vial holders.
- Mounts on the GC for easy access.
- For Agilent 5890/6890 and Varian GCs.
- Includes all mounting hardware.

cat.# 22681, (ea.)

GC accessories and supplies not included; to order items shown, please refer to our catalog.

Correction—please note:

In the formula on page 8 of the technical guide *Operating Hints for Using Split/Splitless Injectors* included with this *Advantage*, the calculation for P should read:
P = absolute column headpressure = gauge pressure (atm) + 1atm

the RESTEK Advantage

Innovators of High Resolution Chromatography Products

New Pinnacle™ DB HPLC Columns

new!

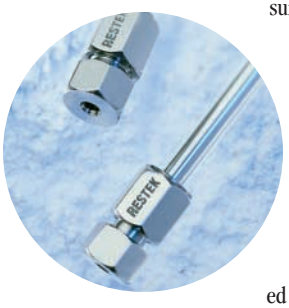
Superior Performance for Basic Compounds

by Greg France, HPLC Products Marketing Manager

✔ Silica manufactured in Restek facilities, for total control of product quality.
✔ Unique manufacturing process ensures sharp, symmetric peaks for basic analytes.
✔ Columns available in narrow bore through preparative-scale formats.

In 2001 our Pinnacle II™ line of columns put Restek among the select few HPLC column manufacturers who manufacture their own silica. We believe that to truly control HPLC variables, a manufacturer must control the entire column manufacturing process, beginning at the initial step—making a well-characterized, consistently-performing silica.

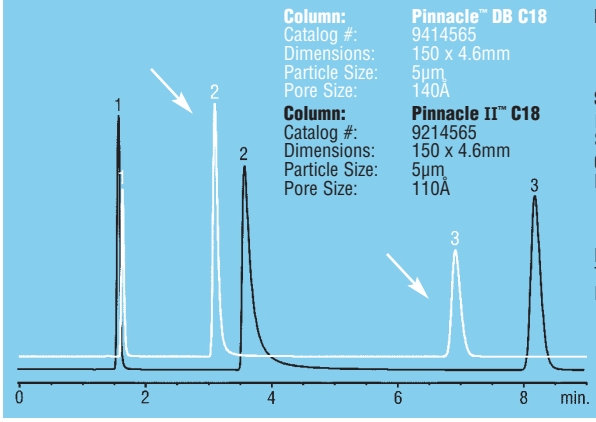
Now, we are pleased to introduce a new line of HPLC columns, Pinnacle™ DB columns, prepared from our newest silica support. Pinnacle™ DB silica is a highly base-deactivated silica, suitable for a wide range of challenging applications. It is ideal for analyses of basic compounds, or mixtures of varied functionality. Part of our manufacturing process for Pinnacle™ DB silica is a unique processing step that creates a base-deactivated particle with excellent performance characteristics. The base deactivation and attractive mass transfer capabilities of the particles enable Pinnacle™ DB silica to resolve and elute basic compounds (e.g., pharmaceuticals) without severe peak tailing—with minimal or no need for mobile phase modifiers such as tetrabutylammonium (TBA). Physical characteristics of Pinnacle™ DB silica are listed in Table I (page 2).



Column manufacturers often use an analysis of a pyridine/phenol test mix to demonstrate a column's separation capabilities and indicate the peak shape that can be anticipated for basic compounds. Figure 1 shows pyridine/phenol separations from a Pinnacle™ DB C18 and a non-base-deactivated C18 column. The sharp, symmetric peaks from the Pinnacle™ DB C18 column—without a mobile phase modifier—are what an analyst can expect for many pharmaceutical or other basic analytes. Figure 2 shows an array of basic pharmaceutical compounds analyzed, with excellent results, on a Pinnacle™ DB C18 column. Note the consistent peak symmetry. Often, neutral or acidic compounds can be present with basic pharmaceutical compounds as impurities or degradation products. Pinnacle™ DB columns are highly suited to these challenges as well. Along with the monomeric C18 bonded phase, the Pinnacle™ DB line currently

Figure 1

Pinnacle™ DB C18 columns provide sharp, symmetric peaks for basic analytes, without a mobile phase modifier.



Column:	**Pinnacle™ DB C18**
Catalog #:	9414565
Dimensions:	150 x 4.6mm
Particle Size:	5µm
Pore Size:	140Å
Column:	**Pinnacle II™ C18**
Catalog #:	9214565
Dimensions:	150 x 4.6mm
Particle Size:	5µm
Pore Size:	110Å

Peak List:	**Conc.**
1. uracil	5.0µg/mL
2. pyridine	0.1µL/mL
3. phenol	1.86mg/mL
Sample:	
Inj.:	5.0µL
Sample Diluent:	mobile phase
Conditions:	
Mobile Phase:	A: 20mM potassium phosphate, pH 7.0 B: acetonitrile Isocratic: 80%A:20%B
Flow:	1.0mL/min
Temp.:	ambient
Det.:	UV @ 254nm

LC_0248
LC_0247

in this issue

RESTEK
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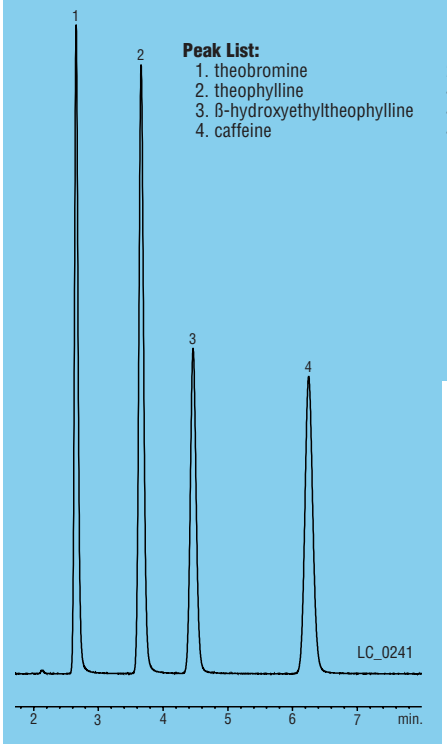
2003 vol. 1

Figure 2

Excellent peak shapes for basic pharmaceuticals help ensure accurate quantification.



Peak List:	Conc.
1. theobromine	225µg/mL
2. theophylline	418µg/mL
3. ß-hydroxyethyltheophylline	418µg/mL
4. caffeine	400µg/mL

Sample:
Inj.: 2µL
Sample Diluent: mobile phase

Column: **Pinnacle™ DB C18**
Catalog #: 9414565
Dimensions: 150 x 4.6 mm
Particle Size: 5µm
Pore Size: 140Å

Conditions:
Mobile Phase: A:20 mMolar KH_2PO_4 buffer, pH 3.0
B:acetonitrile
Isocratic: 90%A:10%B
Flow: 1.0 mL/min
Temp.: 27°C
Det.: UV @ 254nm

Figure 3

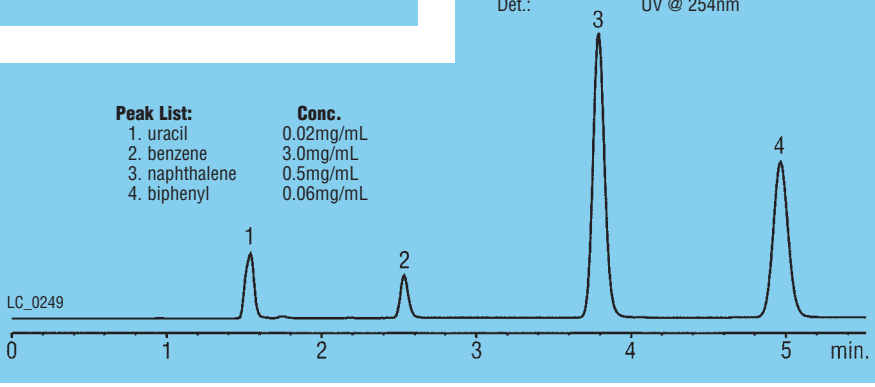
Every Pinnacle™ DB column must pass stringent efficiency criteria.

Sample: **Reversed Phase Test Mix**
Inj.: 5µL
Sample Diluent: methanol:water (75:25 v/v)

Column: **Pinnacle™ DB C18**
Catalog #: 9414565
Dimensions: 150 x 4.6mm
Particle Size: 5µm
Pore Size: 140Å

Conditions:
Mobile Phase: A: water
B: methanol
Isocratic: 20%A:80%B
Run Time: 8 min.
Flow: 1.0mL/min
Temp.: ambient
Det.: UV @ 254nm

Peak List:	Conc.
1. uracil	0.02mg/mL
2. benzene	3.0mg/mL
3. naphthalene	0.5mg/mL
4. biphenyl	0.06mg/mL



Pinnacle™ DB C18 5µm Columns

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#	price
30mm	9414531	9414532	9414533	9414535	
50mm	9414551	9414552	9414553	9414555	
100mm	9414511	9414512	9414513	9414515	
150mm	9414561	9414562	9414563	9414565	
200mm	9414521	9414522	9414523	9414525	
250mm	9414571	9414572	9414573	9414575	

Pinnacle™ DB C8 5µm Columns

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#	price
30mm	9413531	9413532	9413533	9413535	
50mm	9413551	9413552	9413553	9413555	
100mm	9413511	9413512	9413513	9413515	
150mm	9413561	9413562	9413563	9413565	
200mm	9413521	9413522	9413523	9413525	
250mm	9413571	9413572	9413573	9413575	

Pinnacle™ DB Cyano 5µm Columns

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#	price
30mm	9416531	9416532	9416533	9416535	
50mm	9416551	9416552	9416553	9416555	
100mm	9416511	9416512	9416513	9416515	
150mm	9416561	9416562	9416563	9416565	
200mm	9416521	9416522	9416523	9416525	
250mm	9416571	9416572	9416573	9416575	

Pinnacle™ DB Silica 5µm Columns

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#	price
30mm	9410531	9410532	9410533	9410535	
50mm	9410551	9410552	9410553	9410555	
100mm	9410511	9410512	9410513	9410515	
150mm	9410561	9410562	9410563	9410565	
200mm	9410521	9410522	9410523	9410525	
250mm	9410571	9410572	9410573	9410575	

Table I

Physical characteristics of Pinnacle™ DB silica.

Particle:	5µm, spherical
Pore size:	140Å
Pore volume:	0.65mL/g
Carbon load:	C18 - 11%
	C8 - 6%
	cyano - 4%

includes a C8 and a cyano bonded phase, and bare silica. Each of the bonded phases is endcapped.

To ensure the same high quality and reliability as offered by all other Restek HPLC columns, we established demanding quality control procedures to ensure each column performs as expected. Figure 3 is an example analysis of the test mix we use to individually quality check every Pinnacle™ DB column.

The Pinnacle™ DB line is available in a wide range of column dimensions, from 1mm narrow bore columns through 50mm preparative-scale columns. Please call our Technical Service Team for additional information about this new column line, or call our Customer Service representatives to place an order.

To order preparative-scale Pinnacle™ DB columns, please contact our Technical Service Team at 800-356-1688 or 814-353-1300, ext. 4 or contact your local Restek Representative.

new! EZ No-Vent™ GC Column-Mass Spectrometer Connector

Change Columns in Minutes, Without Venting

by Christopher English, Environmental Innovations Chemist, & Brad Rightnour, Instrument Innovations Manager

✔ Easy to install and maintain—no special tools or extra plumbing required.
✔ Gold plated for inertness.
✔ 100µm ID transfer line keeps analytes focused.
✔ Lower cost than other "no-vent" fittings.

We designed our new EZ No-Vent™ GC column-mass spectrometer connector to be simple and easy to use. After studying user feedback concerning our EZ-Vent™ 2000 connector, we re-engineered the connector fitting for even better performance.

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 1 shows the fitting installed and ready for use.

We tested the EZ No-Vent™ connector, using highly volatile gaseous sample components that are very susceptible to tailing in the presence of dead volume. We operated the system as a purge-and-trap GC/MS system, initially with a split at the injection port and the column inserted directly into the MS interface. Next, we included the EZ No-Vent™ connector at the MS interface. We anticipated that any dead volume in this fitting would produce significant tailing and broader peaks, relative to the direct connection. Peak shape was excellent using the new fitting.

Similarly, we used an application-specific test to evaluate the ability of the MS to stabilize after we changed columns without venting. Again we used a purge-and-trap system, halogenated volatiles as the sample, and an Rtx®-624 column to separate the analytes. We acquired Figure 2a at 08:12 AM, then changed the column. We acquired Figure 2b 76 minutes later, at 09:28 AM. Note the excellent peak shapes and responses. In the interval between the two analyses we verified MSD tuning, and the system passed bromofluorobenzene (BFB) criteria. Subsequent detailed investigations have established that the EZ No-Vent™ connector will allow several column changes in a single day, with no harm to the MS or loss of data quality.

If you're tired of waiting for your MS to stabilize, we highly recommend you use an EZ No-Vent™ connector. It will reduce your MS downtime, saving you money, and increase your sample throughput—making you money.

for more info
request lit. cat.# 59498.

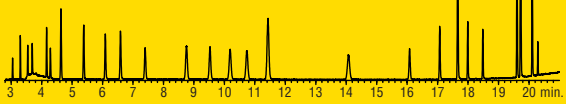
Figure 1
MS transfer line installed in an EZ No-Vent™ connector. Connection can be made quickly, without special tools.

Figure 2
Acquire a sample within two hours of changing columns, using an EZ No-Vent™ connector.

Rtx®-624 60m, 0.25mm ID, 1.4µm (cat# 10969)
Halogenated Volatiles @ 200 ppb in 5mL/RO water.
502.2 Calibration Mix#1 (gases) cat# 30042
502.2 Calibration Mix#2 cat# 30043
BTEX standard cat# 30213

Restek *Innovation!*

2a—acquired at 8:12am



GC_EV00659c

Inj.: purge & trap
GC: Agilent 6890
Inj. temp.: 300°C
Carrier gas: helium, constant flow
Flow rate: 1.0mL/min.
Oven temp.: 60°C (hold 15 min.) to 220°C @ 30°C/min. (hold 1 min.)
Det: Agilent 5973 GC/MS
Transfer line temp.: 280°C
Scan range: 35-550 amu
Tune: BFB
Ionization: EI

2b—acquired at 9:28am
Ez-No Vent™, acquired 76 min. after installing new column

GC_EV00659d

Description	qty.	cat.#	price
EZ No-Vent™ Connector Kit for Agilent 5971/5972 and 5973 GC/MS (Kit includes: EZ No-Vent™ Connector, 0.4mm ID ferrules for connecting capillary column, 0.4mm ID ferrules for connecting transfer line, 100µm deactivated transfer line (3 ft.), and EZ No-Vent™ column plug and nut.)	kit	21323	
Replacement ferrules for connecting capillary column to EZ No-Vent™: 0.4mm ID	2-pk.	21015	
0.5mm ID	2-pk.	21016	
Replacement ferrules for connecting transfer line to EZ-No Vent™: 0.4mm ID	2-pk.	21043	
Replacement deactivated transfer line: 100µm ID	3 ft.	21018	
Replacement EZ No-Vent™ Column Nut	5-pk.	21900	
Replacement EZ No-Vent™ Plug	2-pk.	21915	
Open-end Wrenches (1/4" x 5/16")	2-pk.	20110	

Did you know?

Restek offers supplies and innovative tools for your MS. Refer to the Instrument Supplies section of the annual *Chromatography Products Guide* (lit. cat.# 59473).

GC/MS Screening of Semivolatile Organic Compounds in Drinking Water

Using New Restek Reference Materials and a Capillary Column with Optimized Dimensions

by Katia May, Ph.D., R&D Chemist, and Christopher English, Environmental Innovations Chemist

✔ Full complement of new reference materials for EPA Method 526: calibration standard, internal standard, surrogate standard.
✔ Rtx®-5Sil MS column offers low GC/MS bleed and excellent inertness.
✔ Styrene/divinylbenzene extraction disks for sample preparation.

The US EPA recently developed GC/MS methodology for screening finished drinking water for selected semivolatile organic compounds not addressed by the Safe Drinking Water Act (SDWA). These Unregulated Contaminant Monitoring Rule (UCMR) List 2 contaminants are part of a screening survey established to determine whether in the future these contaminants should be regulated by standard drinking water methods. Method 526 is applicable for 11 of the 15 contaminants on List 2.* Compounds monitored by Method 526 are effectively extracted from water, using 47mm polystyrene divinylbenzene (SDVB) solid phase sorbent, and are sufficiently volatile and thermally stable for GC. The minimum reporting level (MRL) concentration for UCMR List 2 is 0.5µg/L—the value of the lowest concentrations at which precision and accuracy determinations were made during method development.

After careful review of the method we have prepared a new Restek calibration standard, as recommended in Method 526, that includes all 11 semivolatile organic compounds at 200µg/mL each in ethyl acetate. We also developed internal and surrogate standards, and we offer a GC/MS tuning solution at a convenient 2500µg/mL concentration in methylene chloride (SV Tuning Compound, cat.# 31001; see page 7 of this newsletter). This set of Restek reference materials will meet all chemical standards needs for Method 526.

The majority of the target compounds analyzed through EPA Method 526 are pesticides. Low-level injections reduce sensitivity for many of these compounds, due to degradation or irreversible adsorption in the injection port. Cyanazine, 2,4,6-trichlorophenol, and prometon, for example, are susceptible to adsorption or thermal degradation in the GC inlet. To circumvent inlet problems, we use a deactivated, Drilled Uniliner® liner in the inlet, to significantly reduce sample exposure to the hot metal surfaces of the injection port.**

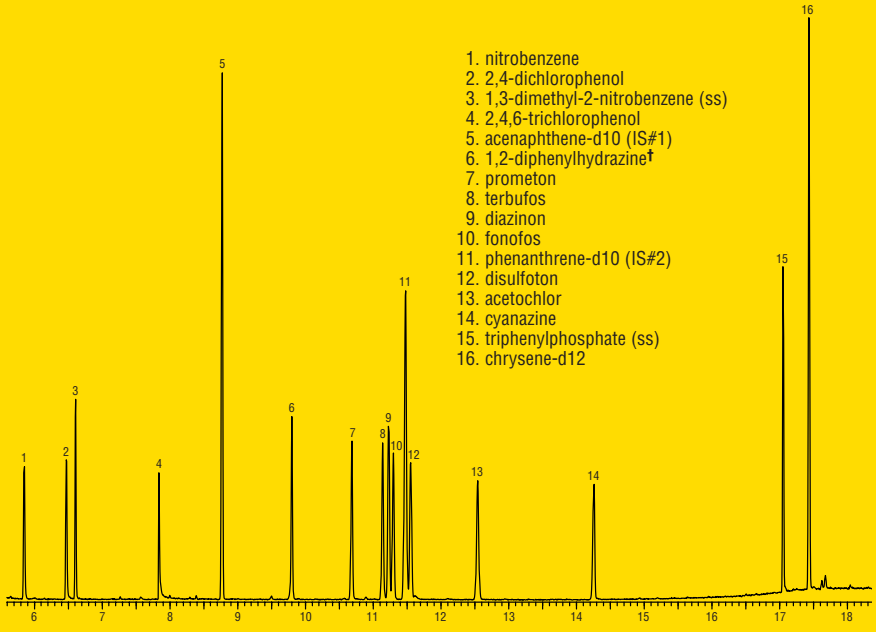
In addition, when analyzing pesticides there are demands on the capillary GC column for higher inertness and lower GC/MS bleed: active sites in the inlet liner or column can cause complete loss of prometon and excessive tailing of 2,4-dichlorophenol and 2,4,6-trichlorophenol peaks. Our silarylene polymer-based Rtx®-5Sil MS column provides optimal separation of the new reference materials and exhibits very low column bleed, compared to traditional phenyl/methyl phases. The 30m, 0.25mm ID, 0.25µm column (cat.# 12723) used to obtain Figure 1 is an optimal combination of internal diameter and film thickness, making it the choice for analyzing many semivolatile compounds. It also is an excellent choice for EPA Method 8270. The thin phase film reduces analysis time, and high temperature stability (330/350ºC) enables an analyst to use a rapid temperature program during the analysis, and to bake high boiling contaminants out of the column after each analysis, without significant bleed.

We highly recommend these new reference materials, a Drilled Uniliner® inlet liner, and an Rtx®-5Sil MS column to anyone conducting analyses according to Method 526.

See product listing on the next page.

Figure 1

An inert, low-bleed Rtx®-5Sil MS column is an effective analytical tool for unregulated drinking water contaminants.



Rtx®-5Sil MS, 30m, 0.25mm ID, 0.25µm (cat# 12723)
US EPA Method 526 analytes, 1µL, 10ppm (20ppm IS)
Semivolatile Calibration Mix, EPA 526 cat.# 31691
Surrogate Standard Mix, EPA 526 cat.# 31693
Internal Standard Mix, EPA 526 cat.# 31692

Inj.:	1.0µL splitless (hold 0.3 min.), 4mm Drilled Uniliner® (cat.# 21055)
GC:	Agilent 6890
Inj. temp.:	300°C
Carrier gas:	helium, constant flow
Flow rate:	0.8 mL/min.
Oven temp.:	50°C (hold 1 min.) to 200°C @ 20°C/min. (hold 5 min.) to 310°C @ 30°C/min. (hold 3 min.)
Det:	Agilent 5973 GC/MS
Transfer line temp.:	280°C
Scan range:	35-550 amu
Solvent delay:	5.5 min.
Tune:	DFTPP
Ionization:	EI

† *Mix component 1,2-diphenylhydrazine is oxidized to azobenzene, the analyte in the figure, on injection onto the heated column.*

For information about UCMR limits, see http://www.epa.gov/safewater/methods/unregtbl.html
For more information about Drilled Uniliner® inlet liners, see page 13.

Resprep™ Resin SPE Disks

- For chlorinated, benzidene-containing, or nitrogen-containing pesticides.
- Meet description in EPA Methods 515.2, 553, and 526.
- 47mm glass fiber embedded with styrene/DVB resin.

Description	qty.	cat.#	price
Resprep™ Resin SPE Disks	20-pk.	26023	

Diskcover™-47 Extraction Disk Holder

- Compatible with most vacuum manifold systems that accept ⅛-inch male luer fittings.
- Sample can be automatically introduced via ⅛-inch Teflon® tubing or by the optional Diskcover™-47 reservoir.

Description	qty.	cat.#	price
Diskcover™-47	ea.	24020	
Diskcover™-47	6-pk.	24021	

Refer to our* Chromatography Products Catalog *(lit. cat.# 59473) for manifolds.

Semivolatile Calibration Mix, EPA 526

(11 compounds)

acetochlor
cyanazine
diazinon
2,4-dichlorophenol
1,2-diphenylhydrazine
disulfoton
fonofos
nitrobenzene
prometon
terbufos
2,4,6-trichlorophenol

200µg/mL each in ethyl acetate, 1mL/ampul

Each	5-pk.	10-pk.
31691	31691-510	—
	w/data pack	
31691-500	31691-520	31791

Internal Standard Mix, EPA 526

acenaphthene-d10
chrysene-d12
phenanthrene-d10

500µg/mL each in acetone, 1mL/ampul

Each	5-pk.	10-pk.
31692	31692-510	—
	w/data pack	
31692-500	31692-520	31792

Surrogate Standard Mix, EPA 526

2-nitro-*m*-xylene
triphenylphosphate

500µg/mL each in acetone, 1mL/ampul

Each	5-pk.	10-pk.
31693	31693-510	—
	w/data pack	
31693-500	31693-520	31793

Ordering Information | Drilled Uniliner® Inlet Liners

DI Liners for Agilent 5890 & 6890 GCs (For 0.25/0.32/0.53mm ID Columns)	ID*/OD & Length (mm)	cat.#/price ea.	cat.#/price 5-pk.
Drilled Uniliner®	4.0 ID 6.3 OD x 78.5	21054	21055
Siltek™ Drilled Uniliner®	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5
Siltek™ 1mm Drilled Uniliner®	1.0 ID 6.3 OD x 78.5	21390-214.1	21391-214.5

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

**Nominal ID at syringe needle expulsion point.*

Ordering Information | Rtx®-5Sil MS Columns (Fused Silica)

(Similar selectivity to Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

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
0.32mm	0.10	-60 to 330/350°C	12706	12709
	0.25	-60 to 330/350°C	12721	12724
	0.50	-60 to 330/350°C	12736	12739
	1.00	-60 to 325/350°C	12751	12754
0.53mm	0.50	-60 to 320/340°C	12737	12740
	1.00	-60 to 320/340°C	12752	12755
	1.50	-60 to 310/330°C	12767	12770

Leak Detective™ II

A Compact, Sensitive Leak Detector for Every Analyst

✔ Affordable thermal conductivity leak detector—every analyst should have one.
✔ Compact, ergonomic design is easy to hold and operate.
✔ Detects helium, hydrogen, and nitrogen at 1x10^{-4}cc/sec. or at an absolute concentration as low as 100ppm.*

Gas leaks in your GC system can increase detector noise, cause baseline instability, waste carrier gas, and damage valuable analytical columns. Leak checks should be a regular part of your GC maintenance program. The new Leak Detective™ II electronic leak detector is the affordable solution for detecting gas leaks. It will identify minute gas leaks that might go undetected by liquid leak detectors.

The Leak Detective™ II electronic leak detector incorporates microchip technology and a new design, to give you better sensitivity and faster response time in a more compact unit. An auto-zero feature allows you to instantly zero the leak detector with a push of a button, and the ergonomic design brings all the controls to your fingertips for easy use. The unit responds in less than two seconds to trace leaks of gases with thermal conductivities different than air. Helium, hydrogen, and nitrogen can be detected at 1x10^{-4}cc/sec or at an absolute concentration as low as 100ppm.* Leaks are indicated by an audible alarm, as well as by an LED readout. For easy, sensitive, and reliable leak detection, order a new Leak Detective™ II electronic leak detector today.

Description	qty.	cat.#	price
Leak Detective™ II Leak Detector	ea.	20413	

**Never use liquid leak detectors on a capillary system because liquids can be drawn into the column.*
Caution: NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.

Simplify Analyses of Permanent Gases and Light Hydrocarbons

new! RESTEK Exclusive!

Using ShinCarbon ST Micropacked Columns

by Barry Burger, Petroleum Chemist, and Neil Mosesman, GC Columns Marketing Manager

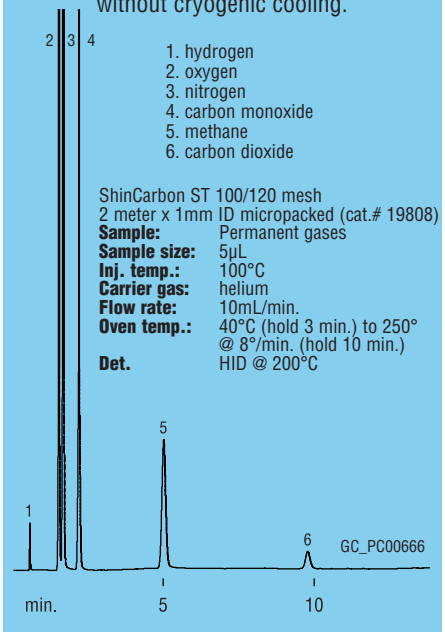
- ✔ Separate permanent gases, including CO/CO_2, in 10 minutes, without cryogenic cooling.
- ✔ Rapid separations of permanent gas / light hydrocarbon mixtures.
- ✔ Excellent compatibility with most GC detectors—minimal bleed, minimal baseline rise.
- ✔ Pre-conditioned, less than 30 minutes to stabilize.

Analyzing the permanent gases oxygen, nitrogen, methane, carbon monoxide, and carbon dioxide has been virtually impossible for a single gas chromatography (GC) or gas solid chromatography (GSC) column, without sub-ambient temperatures. Porous layer open tubular (PLOT) or molecular sieve-packed columns are capable of separating the small molecules, such as oxygen and nitrogen, but adsorb larger-molecule gases, such as carbon dioxide. Porous polymer-packed columns, such as Hayesep® Q, D, or A or Porapak® Q, can be used to analyze methane, carbon monoxide, and carbon dioxide, but column lengths in excess of six meters (20ft.) and sub-ambient conditions are required to separate oxygen and nitrogen.

Now, Restek's new ShinCarbon ST material, a high surface area carbon molecular sieve (~1500 m^2/g), is the ideal medium for separating gases and highly volatile compounds by GSC. A 2-meter by 1mm ID micropacked column containing ShinCarbon ST separates the permanent gases in 10 minutes, without need for cryogenic cooling (Figure 1).

Figure 1

A ShinCarbon ST micropacked column separates permanent gases in 10 minutes, without cryogenic cooling.



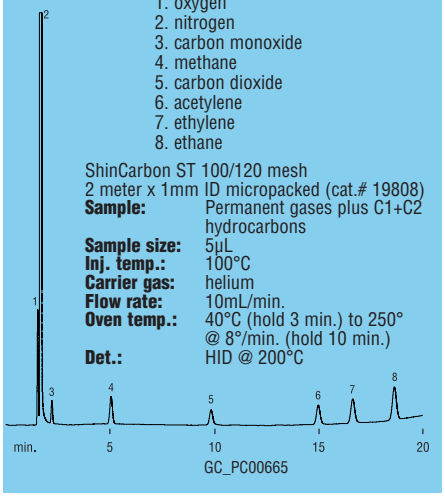
1. hydrogen
2. oxygen
3. nitrogen
4. carbon monoxide
5. methane
6. carbon dioxide

ShinCarbon ST 100/120 mesh
2 meter x 1mm ID micropacked (cat.# 19808)

Sample:	Permanent gases
Sample size:	5µL
Inj. temp.:	100°C
Carrier gas:	helium
Flow rate:	10mL/min.
Oven temp.:	40°C (hold 3 min.) to 250° @ 8°/min. (hold 10 min.)
Det.	HID @ 200°C

Figure 2

ShinCarbon ST columns rapidly separate light hydrocarbon / permanent gas mixtures.



1. oxygen
2. nitrogen
3. carbon monoxide
4. methane
5. carbon dioxide
6. acetylene
7. ethylene
8. ethane

ShinCarbon ST 100/120 mesh
2 meter x 1mm ID micropacked (cat.# 19808)

Sample:	Permanent gases plus C1+C2 hydrocarbons
Sample size:	5µL
Inj. temp.:	100°C
Carrier gas:	helium
Flow rate:	10mL/min.
Oven temp.:	40°C (hold 3 min.) to 250° @ 8°/min. (hold 10 min.)
Det.:	HID @ 200°C

In addition to providing a breakthrough in analyses of permanent gases, ShinCarbon ST columns can be used to separate light hydrocarbon / permanent gas mixtures. Figure 2 shows an analysis of permanent gases plus acetylene, ethylene, and ethane, completed in less than 20 minutes. Other potential applications for ShinCarbon ST include analyses of low molecular weight sulfur compounds or Freon® fluorocarbons.

ShinCarbon ST is a highly stable material. Its 330ºC upper temperature limit minimizes bleed and baseline rise during temperature programming, making the material compatible with most detection systems used for gas analysis, including TCD or HID. All ShinCarbon ST columns are fully conditioned in an oxygen/moisture free environment to prevent contamination. This minimizes stabilization time (less than 30 minutes) when installing a new column which, in turn, minimizes downtime.

The unique properties of ShinCarbon ST make it an ideal packing material for analyses of gases and highly volatile compounds, including permanent gases, low molecular weight hydrocarbons and sulfur compounds, and Freon® gases. The rapid, above-ambient analyses these columns provide will be a great convenience. Excellent thermal stability of the high surface area carbon, combined with careful conditioning during column manufacture, ensures low-bleed operation and rapid stabilization when installing a new column. Custom-made ShinCarbon ST columns are available on request.

Ordering Information | ShinCarbon ST 100/120 Micropacked Columns

OD	ID	1-Meter	2-Meter
1/16"	1.0mm	19809	19808
0.95mm	0.75mm	19810	—

Ordering Information | ShinCarbon ST 80/100 Packed Columns

OD	ID	2-Meter
1/8" SilcoSmooth™	2.0mm	80486-xxx

Ordering Information | Installation Kits

	for 0.75mm ID col.	for 1mm ID col.
For valve applications	21062	21065
For split applications	21063	—
For all Agilent GCs	21064	—
For direct injections	—	21066

Column Configurations
Add the appropriate suffix to the catalog number when ordering packed columns. Contact us for custom configurations.

General Configuration: Suffix -800

Agilent 5880, 5890, 5987, 6890: Suffix -810

Varian 3700, Vista Series, FID: Suffix -820

PE 900-3920 Sigma 1,2,3: Suffix -830

PE Auto System 8300, 8400, 8700 (Not On-Column): Suffix -840

Refer to our catalog or website for
Scott gas standards for permanent gases and light hydrocarbons

GC/MS Screening for Phenols

Using an Optimized Analysis

by Christopher English, Environmental Innovations Chemist, and Katia May, Ph.D., R&D Chemist

✔ Full complement of reference materials for US EPA Method 528.
✔ Fortification solution formulated based on MS sensitivity to each analyte.
✔ Rtx®-5Sil MS column allows phenols identification at 5ng on-column.

EPA Method 528 is a solid phase extraction (SPE)/gas chromatography/mass spectrometry screening method developed to measure 12 environmentally important phenols in drinking water and determine whether to regulate these contaminants in order to protect public health. Method 528 is an improvement over previous methods because MS affords positive identification of all 12 phenols without need for confirmation.*

Method 528 detection limits for phenols range from 0.02-0.58µg/L—concentrations below those needed for drinking water monitoring, based on current health effect information. Figure 1 illustrates an analysis of the 12 phenols, at a concentration of 5ng/L. Several critical steps enable us to obtain excellent peak shape and sensitivity at this low concentration:

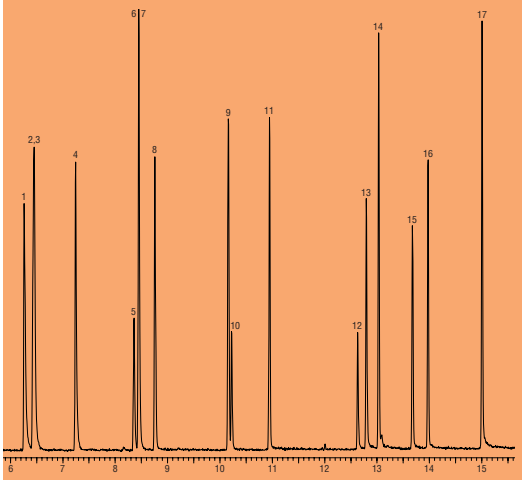
1. Phenols tend to break down in the injection port and exhibit excessive tailing and poor sensitivity. We minimized contact with metal surfaces by using a Drilled Uniliner® liner.** A press-tight seal between the column and the liner eliminates sample contact with the inlet seal. This improves peak shape and increases sensitivity, relative to other hot injection techniques.
2. Reducing the injection port temperature from 330°C to 220°C also contributed to transfer of the phenols to the column with minimal thermal degradation.
3. A pulsed splitless injection (50psi, 0.5 min.) rapidly transfers the analytes to the column inlet.
4. We set the initial temperature to 40°C to prevent excessive tailing for the early eluting phenols.
5. All 12 phenols elute during the first temperature ramp of 12°C/min.; the second ramp to 300°C bakes out high molecular weight contamination. The Rtx®-5Sil MS column performs at this high temperature with low bleed and excellent inertness.***

We have developed a full set of standards for Method 528—calibration standard, internal standard, analyte fortification solution, surrogate standard, and GC/MS tune check solution. MS sensitivity to four of the phenols is significantly less than for the others, so we incorporate these at a higher concentration, 500µg/mL, in the fortification solution. Some of the substituted phenols exist as isomers that have similar mass spectra, but can be differentiated by retention time. The only coelutions are deuterated surrogates, which do not share common ions with the target analytes.

Because of the potential problems associated with these analytes, Method 528 calls for a low bleed column that provides adequate analyte separation. Rtx®-5Sil MS column performance is characterized by low bleed, excellent inertness, and high maximum operating temperature. A Drilled Uniliner® liner significantly reduces the loss of active compounds and ensures more precise results.

Figure 1

Excellent sensitivity and peak shape for phenols at 5ng on-column, using an Rtx®-5Sil MS column and Restek reference materials.



GC_EV00664

1. phenol
2. 2-chlorophenol-3,4,5,6-d4 (SS)
3. 2-chlorophenol
4. 2-methylphenol
5. 2-nitrophenol
6. 2,4-dimethylphenol-3,5,6-d3 (SS)
7. 2,4-dimethylphenol
8. 2,4-dichlorophenol
9. 4-chloro-3-methylphenol
10. 1,2-dimethyl-3-nitrobenzene (IS#1)
11. 2,4,6-trichlorophenol
12. 2,4-dinitrophenol
13. 4-nitrophenol
14. 2,3,4,5-tetrachlorophenol (IS#2)
15. 2-methyl-4,6-dinitrophenol
16. 2,4,6-tribromophenol (SS)
17. pentachlorophenol

Rtx®-5Sil MS 30m, 0.25mm ID, 0.25µm (cat# 12723)
US EPA Method 528 analytes, 1µL, 5ppm
Phenol Calibration Mix 1, EPA 528 (cat# 31694)
Internal Standard Mix, EPA 528 (cat# 31696)
Surrogate Standard Mix, EPA 528 (cat# 31697)

Inj.:	1.0µL pulsed splitless (hold 0.5 min.), 4mm Drilled Uniliner® (cat.# 21055), pulsed pressure 50psi for 0.5 min.
GC:	Agilent 6890
Inj. temp.:	220°C
Carrier gas:	helium, constant flow
Flow rate:	1.3 mL/min.
Oven temp.:	40°C (hold 1 min.) to 220°C @ 12°C/min. (hold 0 min.) to 300°C @ 30°C/min (hold 1 min.)
Det:	Agilent 5973 GC/MS
Transfer line temp.:	280°C
Scan range:	35-550 amu
Solvent Delay:	5.5 min.
Tune:	DFTPP
Ionization:	EI

Phenol Calibration Mix 1, EPA 528

4-chloro-3-methylphenol
2-chlorophenol
o-cresol
2,4-dichlorophenol
2,4-dimethylphenol
2,4-dinitrophenol
2-methyl-4,6-dinitrophenol
2-nitrophenol
4-nitrophenol
pentachlorophenol
phenol
2,4,6-trichlorophenol

2,000µg/mL each in methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
31694	31694-510	
	w/data pack	
31694-500	31694-520	31794

Internal Standard Mix, EPA 528

3-nitro-*o*-xylene	1,000µg/mL
2,3,4,5-tetrachlorophenol	2,000µg/mL

In methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
31696	31696-510	—
	w/data pack	
31696-500	31696-520	31796

Phenols Fortification Mix, EPA 528

4-chloro-3-methylphenol	100µg/mL
2-chlorophenol	100
o-cresol	100
2,4-dichlorophenol	100
2,4-dimethylphenol	100
2,4-dinitrophenol	500
2-methyl-4,6-dinitrophenol	500
2-nitrophenol	100
4-nitrophenol	500
pentachlorophenol	500
phenol	100
2,4,6-trichlorophenol	100

In methanol, 1mL/ampul

Each	5-pk.	10-pk.
31695	31695-510	
	w/data pack	
31695-500	31695-520	31795

Surrogate Standard Mix, EPA 528

2-chlorophenol-d4	1,000µg/mL
2,4-dimethylphenol-d3	1,000
2,4,6-tribromophenol	2,000

In methanol, 1mL/ampul

Each	5-pk.	10-pk.
31697	31697-510	
	w/data pack	
31697-500	31697-520	31797

SV Tuning Compound

decafluorotriphenylphosphine (DFTPP)

2,500µg/mL in methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
31001	31001-510	
	w/data pack	
31001-500	31001-520	31101

** Requirements for testing and analysis are outlined in Number 40 Code of Federal Regulations (CFR), Chapter 1, Part 141.40.*
***For more information about Drilled Uniliner® inlet liners, see page 13.*
****Rtx®-5Sil MS columns are listed on page 5.*

New Reference Materials for Environmental and Fragrance Analyses

new!

by Katia May, Ph.D., R&D Chemist, and Rebecca Wittrig, Ph.D., Food, Flavor, & Fragrance Innovations Chemist

Approximate Boiling Point/Carbon Number Distribution Marker Stock Standard: Method TNRCC 1005 for TPH

- ✔ Easily determine the retention time window for each boiling point range.
- ✔ Prepared in *n*-pentane, according to EPA requirements.

Complete set of Restek standards for Method TNRCC 1005 also includes: TPH Locator Mix (cat.# 31482), TX TPH Calibration Mix (cat.# 31483), TX TPH Matrix Spike Mix (cat.# 31484).

TNRCC 1005 Retention Time Markers Mix

n-hexane (C6)
n-dodecane (C12)
n-octacosane (C28)
n-pentatriacontane (C35)

200µg/mL each in pentane, 1mL/ampul

Each	5-pk.	10-pk.
31698	31698-510	—
	w/data pack	
31698-500	31698-520	31798

System Evaluation Mix for Organochlorine Pesticides Analyses: US EPA Methods 508/508.1, 608, 617, 625, 1618, 1656, 8080A/8081, 8250A/8270B, CLP

- ✔ Designed for daily assessment of system performance.
- ✔ Reveals active sites in the injection port and/or GC column.
- ✔ Prepared in MTBE—low expansion volume helps minimize backflash.

Daily testing for DDT and endrin degradation, a requirement of US EPA Methods 508/508.1, 608, 617, 625, 1618, 1656, 8080A/8081, 8250A/8270B, and CLP, typically is performed by injecting the calibration mix and checking for degradation products. Because we designed this new reference material specifically for degradation testing, it provides better quality control.

Organochlorine Pesticide System Evaluation Mix

4,4'-DDT 200µg/mL
endrin 100µg/mL

In MTBE, 1mL/ampul

Each	5-pk.	10-pk.
32417	32417-510	—
	w/data pack	
32417-500	32417-520	32517

Aldehydes/Ketones DNPH Standard for US EPA Method TO-11A and 8315

- ✔ Use with HPLC analysis of carbonyl compounds in air.
- ✔ Convenient 15µg/mL concentration, similar to the concentration of interest in most ambient air work.
- ✔ Certificate of Analysis lists both aldehyde/ketone and -DNPH derivative concentrations.

We offer this new fifteen-component standard for Method TO-11A and Method 8315. A 150 x 4.6 mm Ultra C18 HPLC column (cat.# 9174565) provides fast, reliable separations of formaldehyde, many other aldehydes, and ketones.

Aldehyde-Ketone-DNPH TO-11A Calibration Mix

acetaldehyde-DNPH
acetone-DNPH
acrolein-DNPH
benzaldehyde-DNPH
n-butyraldehyde-DNPH
crotonaldehyde-DNPH
2,5-dimethylbenzaldehyde DNPH
formaldehyde-DNPH
hexaldehyde-DNPH
isovaleraldehyde-DNPH
propionaldehyde-DNPH
m-tolualdehyde-DNPH
o-tolualdehyde-DNPH
p-tolualdehyde-DNPH
valeraldehyde-DNPH

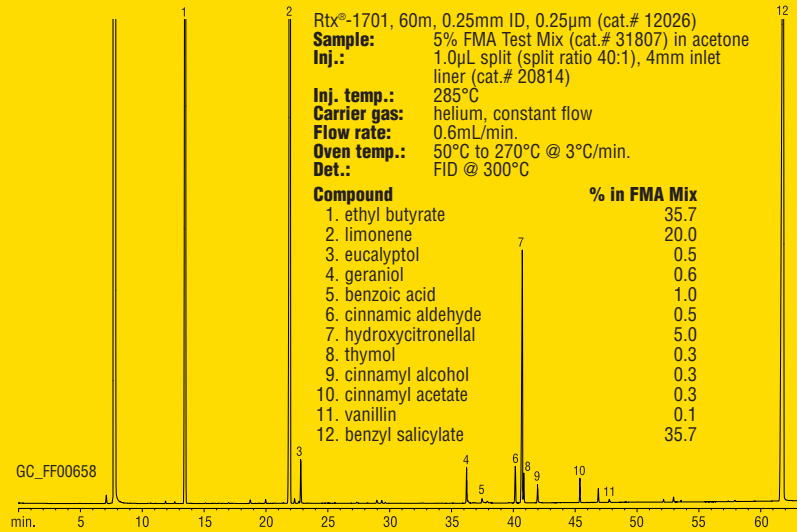
15µg/mL each in acetonitrile, 1mL/ampul

Each
31808 $50

New Fragrance Materials Association GC Performance Evaluation Test Mixture

- ✔ Performance evaluation for essential oils and fragrance chemicals.
- ✔ System Suitability Mixture for GC systems and analytical columns.
- ✔ Convenient 0.5mL quantity for easy dilution to recommended working solution.

Figure 1
New test mix for essential oils and fragrance chemicals.



The Fragrance Materials Association (FMA) has proposed a standardized method that calls for analyzing essential oils by capillary GC on both polar and nonpolar analytical columns. A performance evaluation mixture should be used to determine the condition of the chromatographic system, to aid in the detection of inlet problems, stationary phase degradation, loss of resolution, changes in sensitivity, and the presence of reactive sites in the sample pathway. We have developed a performance evaluation test mixture consistent with the mixture proposed by the FMA. The required 5% test solution can be conveniently made by diluting the entire 0.5mL of neat mixture to 10mL with acetone. The working solution will be stable for up to one week if transferred to a dark container and stored refrigerated.

Fragrance Materials Association Test Mix

benzyl salicylate	362 parts*	geraniol	6 parts
cinnamic aldehyde	5 parts	hydroxycitronellal	50 parts
cinnamic alcohol	3 parts	d-limonene	200 parts
cinnamyl acetate	3 parts	thymol crystal	3 parts
ethyl butyrate	362 parts	vanillin	1 part
eucalyptol	5 parts	benzoic acid	1% of mix

Neat, 0.5mL in an amber ampul

Each	5-pk.
31807	31807-510

**parts per thousand*

New Performance Test Mixtures for Liquid Chromatography

by Vernon Bartlett, HPLC Innovations Manager, and Katia May, Ph.D., R&D Chemist

new!

Column Performance Test Mixture

✔ Five-component mix for characterizing HPLC column parameters.
✔ Simple, easy, reliable approach to QC evaluations or column classification.

The National Institute of Standards and Technology (NIST) has formulated a mixture of five organic compounds that is highly effective for characterizing HPLC columns. The primary column parameters measured by using the mixture are: efficiency, void volume, methylene selectivity, retentiveness, and activity toward chelators and organic bases. Results obtained by using the mix can be used for column classification, column selection during method development, for monitoring column performance over time, or for quality control during column manufacturing.

We follow the NIST method for producing this mix, and we test our material against the NIST 870 standard. Figure 1 shows the chromatographic profiles for the Restek reference material and the NIST 870 standard are nearly identical.

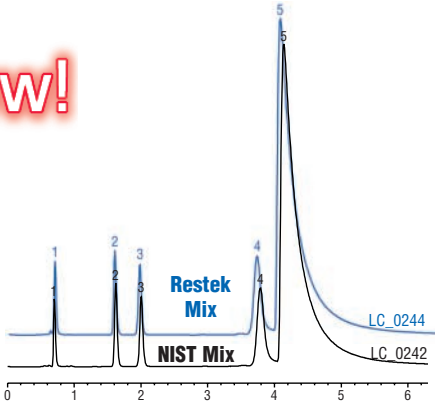
HPLC Performance Test Mix

amitriptyline hydrochloride	2800µg/mL	quinizarin	94
ethylbenzene	1700	toluene	1400
		uracil	28

In methanol, 1mL/ampul

Each	5-pk.	10-pk.
31699	31699-510	—
	w/data pack	
31699-500	31699-520	31799

Figure 1
Restek HPLC Column Performance Test Mixture matches NIST 870 standard.



Peak List: **Conc. (µg/mL)**
1. uracil 28
2. toluene 1400
3. ethylbenzene 1700
4. quinizarin 94
5. amitriptyline 2800

Sample: HPLC Performance Text Mix (cat.# 31699)
Inj.: 5.0µL
Sample diluent: methanol

Column: **Pinnacle II™ C18**
Catalog #: 9214565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 110Å

Conditions:
Mobile phase: A: 5mM potassium phosphate, pH 7.0
B: MeOH
20%A: 80%B
Flow: 2.0mL/min.
Temp.: 23°C
Det.: UV @ 254nm

HPLC Operational Qualification Test Mix for UV Detector Linearity

✔ Simple test of a detector's ability to produce a linear response to varied concentrations.
✔ Concentrations suitable for clear indication of linear range.

Regulatory documentation for installation qualification (IQ), operation qualification (OQ), and performance qualification (PQ) verifies the system's performance for regulatory agencies. An important part of OQ for HPLC instruments is detector linearity. Analyzed compounds can vary in concentration, so it is important that the detector produce linear responses to concentration variations. Response should be proportional to the concentration of the analyte at constant injection volume.

For this purpose, Restek chemists developed a new analytical standards kit consisting of 1mL each of five aqueous solutions of caffeine at concentrations of 5, 25, 125, 250 and 500 µg/mL. These solutions are convenient for linearity measurements, and can be used to generate simple plots of response versus concentration. Further, the correlation coefficient between sample concentration and response is easily calculated. Our Certificate of Analysis includes caffeine concentration (5–500µg/mL), calculated variance in preparing each mixture, a linearity plot, and coefficient of determination (r^2) for the linear plot.

HPLC OQ Linearity Test Mix Kit

Caffeine at five concentrations in a five ampul kit at 5.0, 25.0, 125.0, 250.0, 500.0 µg/mL in water. Used to determine UV detector linearity. Sold only as a kit.

Contains 1mL each of these mixtures.

Kit
31805

Sugar Column Performance Qualification Check Mix

✔ For simple sugar analysis by HPLC with refractive index detection.
✔ Use with a silica column with amino functionality, as recommended by AOAC International.
✔ Use as a calibration standard for AACC Method 80-04.
✔ Offered dry, for enhanced stability.

AOAC International recommends an HPLC method using refractive index (RI) detection and an amino-based stationary phase for analyses of simple sugars, i.e., the monosaccharides fructose and glucose and the disaccharides sucrose, maltose, and lactose.

The performance qualification test (PQ), part of laboratory instrument validation, determines the precision of the HPLC system. Usually the PQ test results show the retention time variations. Our new performance check mix for PQ of HPLC / RI consists of five simple sugars in varied concentrations. Because sugar solutions are subject to bacteriological degradation, we prepare the reference material in water, lyophilize it, and pack it dry in small (4mL) vials, for enhanced stability. To perform the performance test, dissolve the material in 1mL acetonitrile/water (75:25, v/v).

Figure 2 shows an excellent resolution of the simple sugars, achieved by using a Pinnacle II™ Amino HPLC column (cat.# 9217365)—a propylamino stationary phase bonded to 3µm silica. Because we manufacture this new column from in-house synthesized materials, we have strict control of column-to-column reproducibility.

Sugar Column Performance Check Mix

glucose	2.0µg/mL*	maltose	4.5
fructose	2.1	sucrose	4.0
lactose	4.4		

Dry components in 4mL screw-cap vial.

Each
31809

**Final concentration when reconstituted in 1mL acetonitrile:water (75:25) v/v.*

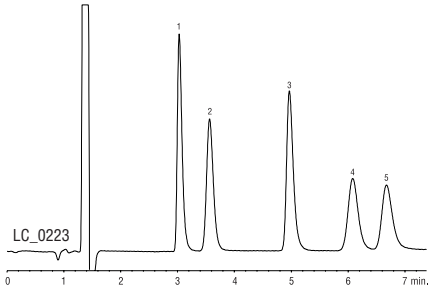
Figure 2
Excellent performance for simple sugars, using a Pinnacle II™ Amino HPLC column.

Peak List: **Conc.** (mg/mL)
1. fructose 2.0
2. glucose 2.1
3. sucrose 4.0
4. maltose 4.5
5. lactose 4.4

Sample: Sugar Column Performance Check Mix (cat.# 31809)
Inj.: 5µL

Column: **Pinnacle II™ Amino**
Catalog #: 9217365
Dimensions: 150 x 4.6mm
Particle size: 3µm
Pore size: 110Å

Conditions:
Mobile phase: water:acetonitrile (25:75, v/v)
Flow: 1.5 mL/min.
Temp.: 35°C
Det.: refractive index @ 35°C



Silcosteel®-CR Tubing & Fittings

Enhanced Resistance to Inorganic Acids

by Gary Barone, Metals Passivation Group Product Marketing Manager

- ✔ Use to resist hydrochloric, sulfuric, and nitric acids.
- ✔ Reduces system maintenance and downtime.
- ✔ Tubing and many fittings in stock; custom coating service available.

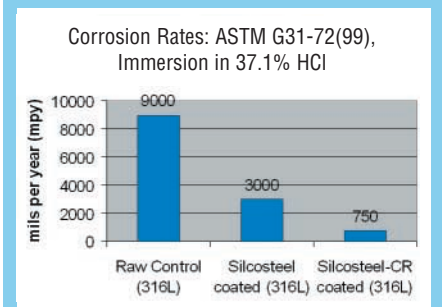
In our continuing program of developing surface technologies, Restek introduces this line of highly corrosion resistant fittings and tubing. Silcosteel®-CR* coating is optimized to enhance resistance to hydrochloric, sulfuric, and nitric acids (Figure 1).

Tubing and fittings used in the presence of these destructive acids require frequent inspection and replacement. The Silcosteel®-CR coating, applied to widely-used Parker A-lok fittings and seamless 316L stainless steel tubing, adds an order of magnitude of protection against corrosive attack.

Silcosteel®-CR treatment involves a unique deposition process that completely covers the exposed surface of the tubing or fitting and prevents contact between the acids and the surface. The Silcosteel®-CR coating itself is insoluble in these acids, and its application provides an unmatched layer of protection to stainless steel.

Custom Silcosteel®-CR coating service is available. Please contact our technical service department or your local Restek representative to inquire about having your items Silcosteel®-CR protected.

Figure 1
Silcosteel®-CR coated 316L stainless steel shows greater than 10-fold better resistance to HCl, compared to untreated stainless steel, and a 4-fold improvement, compared to Silcosteel®-coated stainless steel.



Ordering Information | Silcosteel®-CR Seamless 316L Stainless Steel Tubing

ID	OD	cat.#	Price-per-foot by length 5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
0.055" (1.40mm)	1/8" (3.18mm)**	22896				
0.180" (4.57mm)	1/4" (6.35mm)**	22897				

***0.035" wall thickness*

Ordering Information | Silcosteel®-CR Fittings

Description	qty.	cat.#	price
1/16" Union	ea.	22863	
1/8" Union	ea.	22864	
1/4" Union	ea.	22865	
1/16" Tee	ea.	22866	
1/8" Tee	ea.	22867	
1/4" Tee	ea.	22868	
1/8" to 1/16" Reducer	ea.	22869	
1/4" to 1/16" Reducer	ea.	22870	
1/4" to 1/8" Reducer	ea.	22871	
1/8" Cross	ea.	22872	
1/4" Cross	ea.	22873	
1/16" Elbow	ea.	22874	
1/8" Elbow	ea.	22875	
1/4" Elbow	ea.	22876	
1/16" Plug	ea.	22877	
1/8" Plug	ea.	22878	
1/4" Plug	ea.	22879	
1/8" to 1/16" Tube End Reducer	ea.	22880	
1/4" to 1/16" Tube End Reducer	ea.	22881	
1/8" to 1/4" Tube End Reducer	ea.	22882	
1/4" to 1/8" Tube End Reducer	ea.	22883	
1/8" Port Connector	ea.	22884	
1/4" Port Connector	ea.	22885	
1/8" to 1/4" Port Connector	ea.	22886	
1/8" to 1/8" Male NPT Union	ea.	22887	
1/4" to 1/4" Male NPT Union	ea.	22888	
1/16" to 1/8" Male NPT Union	ea.	22889	
1/8" to 1/4" Male NPT Union	ea.	22890	
1/4" to 1/8" Male NPT Union	ea.	22891	
1/8" to 1/8" Female NPT Union	ea.	22892	
1/4" to 1/4" Female NPT Union	ea.	22893	
1/4" to 1/8" Female NPT Union	ea.	22894	
1/8" to 1/4" Female NPT Union	ea.	22895	

**Patent pending.*

Please note: An extra charge is applied for cutting Silcosteel®-CR tubing. The charge is calculated from the total number of pieces produced from cutting, for each line item, as follows:

5 to 15 pieces $50 additional
16 to 30 pieces $100 additional
31 to 75 pieces $150 additional
76 to 99 pieces $200 additional
100 to 200 pieces $250 additional

New Restek Catalog

By the time you receive this Advantage, the corners of your 2003 Restek catalog should already be dog-eared. Bigger and better than last year, it features chromatography columns, accessories, and reference chemicals introduced throughout 2002 in the *RESTEK Advantage*, plus many other items to make life in your laboratory easier. And, it's not too soon to start thinking of good, practical gifts for your co-workers—the 2003 holiday season will be here before you know it!

If you haven't received your catalog, it's lost in the mail, or your colleagues have it. Call or fax us, and we'll be sure you get one (lit. cat.# 59473).

new! Reproducible Analyses of Polynuclear Aromatic Hydrocarbons Using Pinnacle II™ PAH HPLC Columns and Restek Reference Standards

by Terrence S. Reid, Innovations Team Chemist

✔ Special-purpose HPLC columns resolve 16 PAHs to baseline.
✔ In-house-manufactured silica ensures reproducible column performance.
✔ Three reference standards—choose the concentrations best suited to your need.

Mutagenic, carcinogenic, and pervasive in the modern environment, polynuclear aromatic hydrocarbons (PAHs) are emitted by many sources of combustion, including heating devices that burn fossil fuels and internal combustion engines in motor vehicles. Because they are universal, PAHs are analyzed in many air, water, and soil matrices. Environmental samples often contain complex mixtures of PAHs that are difficult to resolve, due to considerable structural similarity among the various analytes. Reversed phase HPLC is the favored approach for analyses of PAHs because this technique can discriminate among the closely related compounds, on the basis of molecular shape. US Environmental Protection Agency Method 610, for example, calls for reversed phase HPLC for analyses of the 16 PAHs listed in Figure 1.

We developed the newest addition to our Pinnacle II™ HPLC product line, the Pinnacle II™ PAH column, specifically for these demanding analyses. The Pinnacle II™ PAH stationary phase is a specialized polymeric C18 bonding that uses unique shape selectivity to provide baseline resolution of all 16 PAHs in EPA Method 610. Further, we make Pinnacle II™ PAH columns using silica we manufacture ourselves, at our Bellefonte, PA facility. By making our own silica, we have greater control over both quality and reproducibility. Pinnacle II™ PAH columns provide the same selectivity as our original Pinnacle™ PAH columns, but greater reproducibility.

Lot-to-lot reproducibility of performance for Pinnacle II™ PAH columns is excellent, as shown in Figure 1. Each of the three lots of columns represented has provided baseline resolution of the 16 PAHs listed in Method 610, using a simple, linear water/acetonitrile mobile phase gradient.

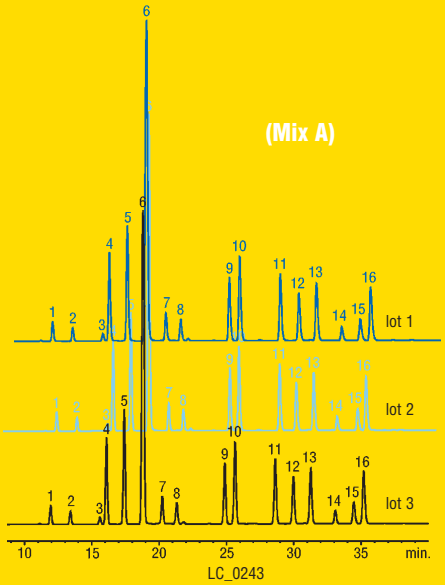
In addition to these special-purpose HPLC columns, Restek manufactures three analytical reference mixes for EPA Method 610. The mixes differ in concentrations, but each contains all 16 listed PAHs, packaged in convenient 1mL ampuls. The sample used to obtain Figure 1 is 610 PAH Calibration Mix A.

If you are analyzing PAHs, the information presented here shows Restek can provide both the reliable, cost-effective HPLC columns and the chemical standards you need.

Figure 1

Pinnacle II™ PAH columns provide reproducible, baseline resolution of the 16 PAHs listed in US EPA Method 610.

Peak List:	Ret. Time (min.) Lot 1	Lot 2	Lot 3	Conc. (µg/mL)
1. naphthalene	11.95	12.17	11.80	100
2. acenaphthylene	13.41	13.65	13.26	100
3. acenaphthene	15.61	15.89	15.45	100
4. fluorene	16.09	16.31	15.92	100
5. phenanthrene	17.41	17.61	17.23	50
6. anthracene	18.80	18.92	18.61	100
7. fluoranthene	20.23	20.39	20.03	50
8. pyrene	21.31	21.46	21.11	50
9. benzo(a)anthracene	24.88	24.87	24.65	50
10. chrysene	25.64	25.54	25.40	50
11. benzo(b)fluoranthene	28.62	28.53	28.35	50
12. benzo(k)fluoranthene	29.97	29.74	29.70	50
13. benzo(a)pyrene	31.26	31.02	30.97	50
14. dibenzo(a,h)anthracene	33.09	32.74	32.81	50
15. benzo(ghi)perylene	34.46	34.24	34.16	50
16. indeno(1,2,3-cd)pyrene	35.19	34.85	34.91	50



Sample:
Inj.: 610 PAH Calibration Mix A (cat.# 31264), 5µL
Sample diluent: methylene chloride:acetonitrile (1:9, v/v)

Column:
Catalog #: 9219563
Dimensions: 150 x 3.2mm
Particle size: 5µm
Pore size: 110Å

Conditions:
Mobile phase: A: water; B: acetonitrile

Time:	%B
0	40
30	100
40	100
41	40
51	40

Flow: 0.5mL/min
Temp.: 30°C (or ambient)
Det.: UV @ 254nm

Pinnacle II™ PAH 5µm Columns

Length	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#	price
100mm	—	9219513	9219515	
150mm	—	9219563	9219565	
200mm	—	9219523	9219525	
250mm	9219572	9219573	9219575	

SV Calibration Mix #5 (16 components)

3/90 and 4/89 SOW

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

2,000µg/mL each in methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
31011	31011-510	—
w/data pack		
31011-500	31011-520	31111

610 PAH Calibration Mix A

(16 components)

	1000µg/mL		500
acenaphthene		chrysene	
acenaphthylene	1000	dibenzo(a,h)anthracene	500
anthracene	1000	fluoranthene	500
benzo(a)anthracene	500	fluorene	1000
benzo(a)pyrene	500	indeno(1,2,3-cd)pyrene	500
benzo(b)fluoranthene	500	naphthalene	1000
benzo(k)fluoranthene	500	phenanthrene	500
benzo(ghi)perylene	500	pyrene	500

In methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
31264	31264-510	—
w/data pack		
31264-500	31264-520	31364

610 PAH Calibration Mix B

(16 components)

	1000µg/mL		200
acenaphthene		benzo(ghi)perylene	
acenaphthylene	2000	chrysene	100
anthracene	100	dibenzo(a,h)anthracene	200
benzo(a)anthracene	100	fluoranthene	200
benzo(a)pyrene	100	fluorene	200
benzo(b)fluoranthene	200	indeno(1,2,3-cd)pyrene	100
benzo(k)fluoranthene	100	naphthalene	1000
phenanthrene	100	pyrene	100

In methylene chloride:methanol (1:1), 1mL/ampul

Each	5-pk.	10-pk.
31455	31455-510	—
w/data pack		
31455-500	31455-520	31555

High-Resolution Analysis of Fatty Acid Methyl Esters (FAMEs)

Using an Rt-2560 Capillary GC Column to Resolve *cis* and *trans* Isomers

by Rebecca Wittrig, Ph.D., Food, Flavor & Fragrance Innovations Chemist

✔ Highly polar Rt-2560 column meets analysis requirements of AOAC Method 996.06.
✔ Column suitable for determining fatty acid composition or total *trans* fat.
✔ Reference materials formulated for calibrating the GC system and identifying isomers.

Modern requirements for characterizing fats and oils and determining the total fat content in foods call for highly efficient separations offered by capillary GC columns. A properly chosen column can provide accurate information about complex fat or oil samples, e.g., total fat content, *trans* fat content, or total omega-3 polyunsaturated fatty acid content. Carbowax®-type (polyethylene glycol) stationary phases typically are used for separating, identifying, and quantifying saturated and unsaturated fatty acid methyl esters (FAMEs). More polar biscyanopropyl phases, typically in longer columns, are needed to resolve *cis* and *trans* isomers of polyunsaturated components, for determining total fat content, or for quantifying total *trans* fat.

Individual *cis* and *trans* FAME isomers are effectively resolved on a 100-meter Rt-2560 column, making this the column of choice for analyzing partially hydrogenated oils. The highly polar biscyanopropyl phase gives the selectivity needed for resolving these isomers, such as the *cis* and *trans* forms of C18:1. The *trans* isomers elute before the *cis* isomers, opposite of their elution order on Carbowax®-based phases such as Rtx®-Wax or FAMEWAX®.

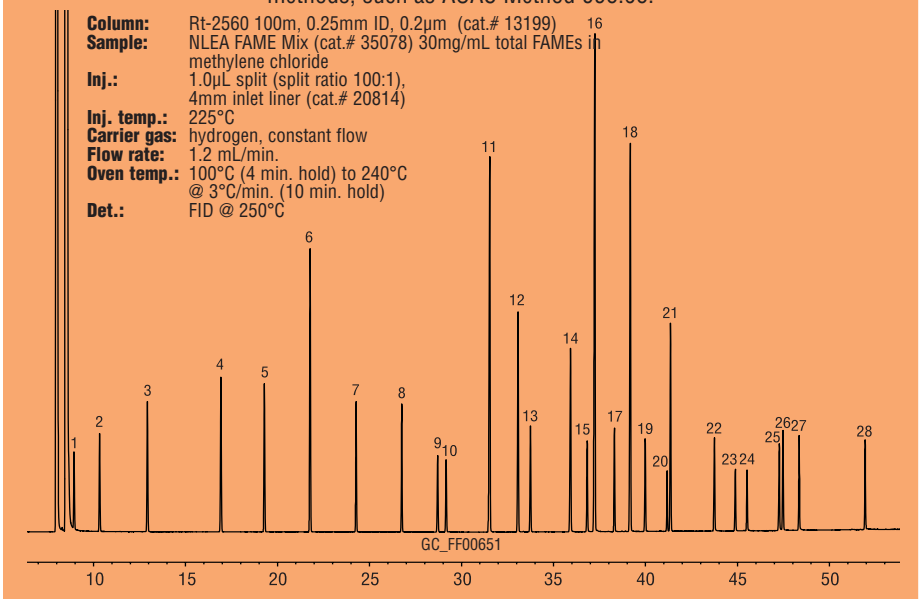
AOAC Method 996.06, the specified method for determining the total fat content of a food for nutritional labeling purposes, calls for determining total fat content based on fatty acid content, after the fatty acids are converted to methyl esters.[1] The 100-meter Rt-2560 column meets the requirements of this procedure. An Rt-2560 column also allows quantification of total *trans* content. Note that Rtx®-2330 columns, our slightly less polar 90% biscyanopropyl phase columns, also resolve *cis* and *trans* FAME isomers. On Rtx®-2330 columns, as on Rt-2560 columns, the *trans* forms of the FAMEs elute before the *cis* forms.

To calibrate the GC system for assays of this type, use a FAME mixture such as our Food Industry FAME Mix, a 37-component mixture of FAMEs typically encountered in vegetable, animal, and marine fats and oils, or our 28-component NLEA FAME Mix (Figure 1). Each of these standards includes a gravimetric certificate of analysis to help ensure accurate quantification. To ensure correct identifications of individual *cis* and *trans* isomers of C18:1, use our *cis/trans* Isomer Mix.

Whatever your fat or oil analysis requirements, Restek can provide high-performance analytical columns and reference standards that will help you to accurately characterize your materials.

Figure 1

NLEA FAME Mix contains the components needed to standardize fat-by-fatty acid composition methods, such as AOAC Method 996.06.



1. C4:0 methyl butyrate
2. C6:0 methyl heanoate
3. C8:0 methyl octanoate
4. C10:0 methyl decanoate
5. C11:0 methyl undecanoate
6. C12:0 methyl laurate
7. C13:0 methyl tridecanoate
8. C14:0 methyl myristate
9. C14:1 methyl myristoleate (*cis*-9)
10. C15:0 methyl pentadecanoate
11. C16:0 methyl palmitate
12. C16:1 methyl palmitoleate (*cis*-9)
13. C17:0 methyl heptadecanoate
14. C18:0 methyl stearate
15. C18:1 methyl elaidate (*trans*-9)
16. C18:1 methyl oleate (*cis*-9)
17. C18:2 methyl linoelaidate (*trans*-9,12)
18. C18:2 methyl linoleate (*cis*-9,12)
19. C20:0 methyl arachidate
20. C20:1 methyl eicosenoate (*cis*-11)
21. C18:3 methyl linolenate (*cis*-9,12,15)
22. C22:0 methyl behenate
23. C22:1 methyl erucate (*cis*-13)
24. C23:0 methyl tricosanoate
25. C24:0 methyl lignocerate
26. C20:5 methyl eicosapentaenoate (*cis*-5,8,11,14,17)
27. C24:1 methyl mervonate (*cis*-15)
28. C22:6 methyl docosahexaenoate (*cis*-4,7,10,13,16,19)

NLEA FAME Mix (28 components)

Compound	Qty. (%)	Compound	Qty. (%)
C4:0	1.5%	C18:1(*trans*)	2.5%
C6:0	1.5%	C18:1(*cis*)	15.0%
C8:0	2.0%	C18:2(*trans*)	2.5%
C10:0	2.5%	C18:2(*cis*)	10.0%
C11:0	2.5%	C18:3	5.0%
C12:0	5.0%	C20:0	2.5%
C13:0	2.5%	C20:1	1.5%
C14:0	2.5%	C20:5	2.5%
C14:1(*cis*-9)	1.5%	C22:0	2.5%
C15:0	1.5%	C22:1	1.5%
C16:0	10.0%	C22:6	2.5%
C16;1(*cis*-9)	5.0%	C23:0	1.5%
C17:0	2.5%	C24:0	2.5%
C18:0	5.0%	C24:1	2.5%

30mg/mL total in methylene chloride, 1mL/ampul

	ea.
35078	

Food Industry FAME Mix (37 components)

Compound	Qty. (%)	Compound	Qty. (%)
C4:0	4.0	C18:2(all-*cis*-9,12)	2.0
C6:0	4.0	C18:3(all-*cis* 6,9,12)	2.0
C8:0	4.0	C18:3(all-*cis* 9,12,15)	2.0
C10:0	4.0	C20:0	4.0
C11:0	2.0	C20:1(*cis*-11)	2.0
C12:0	4.0	C20:2(all-*cis* 11,14,)	2.0
C13:	2.0	C20:3 (all-*cis* 8,11,14)	2.0
C14:0	4.0	C20:3(all-*cis* 11,14,17)	2.0
C14:1(*cis*-9)	2.0	C20:4(all-*cis* 5,8,11,14)	2.0
C15:0	2.0	C20:5(all-*cis* 5,8,11,14,17)	2.0
C15:1(*cis*-10)	2.0	C21:0	2.0
C16:0	6.0	C22:0	4.0
C16;1(*cis*-9)	2.0	C22:1(*cis* 13)	2.0
C17:0	2.0	C22:2(all-*cis* 13,16)	2.0
C17:1(*cis*-10)	2.0	22:6(all-*cis*4,7,10,13,16,19)	2.0
C18:0	4.0	C23:0	2.0
C18:1(*trans*-9)	2.0	C24:0	4.0
C18:1(*cis*-9)	4.0	C24:1(*cis*-15)	2.0
C18:2(all-*trans*-9.12)	2.0		

30mg/mL total in methylene chloride, 1mL/ampul

	ea.
35077	

cis/trans FAME Mix (8 components)

Compound	Qty. (%)
methyl elaidate (C18:1 *trans*-9)	10.0%
methyl linoleate (C18:2 *cis*-9,12)	20.0%
methyl oleate (C18:1 *cis*-9)	10.0%
methyl petroselinate (C18:1*cis*-6)	8.0%
methyl petroselaidate (C18:1*trans*-6)	8.0%
methyl stearate (C18:0)	20.0%
methyl transvaccenate (C18:1 *trans*-11)	12.0%
methyl vaccenate (C18:1 *cis*-11)	12.0%

10mg/mL total in methylene chloride, 1mL/ampul

	ea.
35079	

Ordering Information | Rt-2560 Column (Fused Silica)

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

1. *Official Methods of Analysis,* 17th edition, AOAC International, 2000.

Improved Responses for Active Analytes

Using a Drilled Uniliner® GC Inlet Liner

by Gary Stidsen and Lydia Nolan, Innovations Team

✔ Less breakdown of active compounds, for more accurate results.
✔ Greater sensitivity, for lower detection limits.
✔ Minimal injection port discrimination.

Articles on pages 4 and 7 of this *Advantage* describe analyses of phenols and active semivolatile organic compounds that are notoriously difficult to analyze, particularly at low concentrations. Low-level injections reduce sensitivity for these compounds, due to degradation or irreversible adsorption in the injection port. Residues of heavier and non-volatile materials often build up at the bottom of the injection port, leaving a reactive surface that can cause active compounds to break down.

Figure 1

Reduce the loss of active analytes—use a Drilled Uniliner® inlet liner.

Table I

Lowest breakdown of endrin and DDT, and highest responses for analytes, using a Drilled Uniliner® inlet liner.

% Breakdown

Analyte	Column	Drilled Uniliner®	4mm splitless	4mm splitless with wool
Endrin	Rtx®-CLPesticides	4.4	4.7	9.8
	Rtx®-CLPesticides2	4.9	6.9	8.3
DDT	Rtx®-CLPesticides	0.2	0.3	2.6
	Rtx®-CLPesticides2	0.3	0.9	3.1

Response— Mean response; value in table x 10^3 = response units.

Analyte	Column	Drilled Uniliner®	4mm splitless with wool	4mm splitless
Tetrachloro-*m*-xylene (TCMX)	Rtx®-CLPesticides	147	111	106
	Rtx®-CLPesticides2	191	167	162
Decachloro-biphenyl (DCB)	Rtx®-CLPesticides	150	119	108
	Rtx®-CLPesticides2	209	177	166

DI Liners for Agilent 5890 & 6890 GCs (For 0.25/0.32/0.53mm ID Columns)	ID*/OD & Length (mm)	cat.#/price ea.	cat.#/price 5-pk.
Drilled Uniliner®	4.0 ID 6.3 OD x 78.5	21054	21055
Siltek™ Drilled Uniliner®	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5
Siltek™ 1mm Drilled Uniliner®	1.0 ID 6.3 OD x 78.5	21390-214.1	21391-214.5

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

**Nominal ID at syringe needle expulsion point.*

To circumvent this problem, we use a deactivated, Drilled Uniliner® inlet liner (Figure 1) in the inlet, in place of a standard splitless inlet liner, to minimize sample exposure to the hot metal surfaces of the injection port. A Drilled Uniliner® liner makes a press-fit connection between column and liner, eliminating the primary source of analyte breakdown. This physical connection between column and liner also improves sensitivity, by minimizing injection port discrimination. Further, with a Drilled Uniliner® inlet liner less of the injected sample is vented, should you need to switch from splitless to split mode to sweep remaining solvent from the inlet. This too contributes to greater sensitivity.

We tested the performance of a system that included a Drilled Uniliner® inlet liner, using a mixture of chlorinated pesticides at 20, 40, and 200ng/mL concentrations (*Restek Advantage* 2002v4). The percent difference between the observed value and the "true" value for each analyte ranged from 0% to a maximum of only 6.4%. The system that included the Drilled Uniliner® inlet liner reduced endrin and DDT breakdown, relative to systems that included splitless liners (Table 1), because a splitless liner allows the analytes to contact active surfaces in the inlet. Wool packing in the splitless liner aggravates this problem, by greatly increasing the surface area within the liner and introducing additional active sites.

In addition to reducing variability and increasing accuracy of calibration data, a Drilled Uniliner® inlet liner increased overall response for individual analytes, enhancing minimum detection levels compared to standard splitless inlet liners. In the pesticides test, area counts for the last eluting peak, decachlorobiphenyl, were greater by 18-39% when a Drilled Uniliner® inlet liner was used, relative to area counts for injections made on splitless liners (Table 1).

By eliminating the bottom of the injector from the sample pathway, a Drilled Uniliner® inlet liner makes the pathway more inert. This can increase accuracy and precision, and reduce breakdown of active analytes, relative to hot flash injection techniques. If you are conducting analyses of phenols, chlorinated pesticides, or other active analytes, these results clearly show that a Drilled Uniliner® inlet liner could be the liner of choice.

Restek's Customer Commitment
Plus 1™ PLUS ONE

Plus 1™ means that we will surpass your expectations every time you contact us. Looking for the solution to your tough analytical problem or placing a late-day order? Contact us to experience Plus 1™ service today!

Visit us at Pittcon® 2003!

Booth #6151

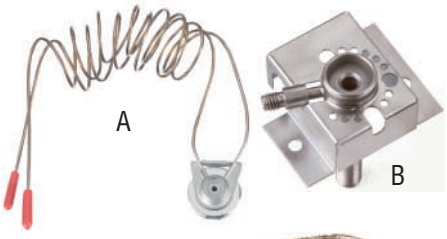
See our website for more information about
• Technical presentations
• Workshops

Peak Performers

Injection Ports for Agilent GCs

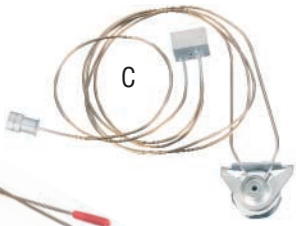
by Donna Lidgett, GC Accessories Marketing Manager

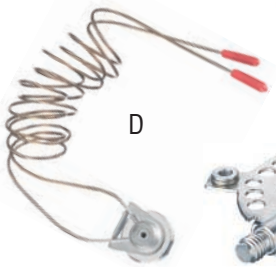
Is it time to replace your injector? Restek's high-quality stainless steel split/splitless injectors are direct replacements for Agilent 5890 and 6890/6850 GCs. Our replacement parts meet or exceed Agilent original equipment performance. For a truly inert sample pathway, we offer Silcosteel®-treated injectors. Silcosteel® treatment passivates the metal surface to deliver superior performance.



Direct Replacement Split/Splitless Injection Port for Agilent 5890 GCs

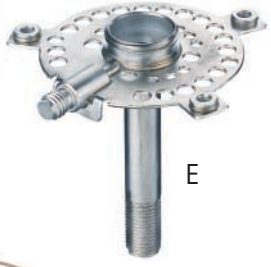
Description	Similar to Agilent part #	qty.	cat.#	price
A) Replacement Weldment*	19251-60575	ea.	20265	
Silcosteel® Weldment*	19251-60575**	ea.	20267	
B) Replacement Shell Weldment	19251-80570	ea.	20266	
Silcosteel® Shell Weldment	19251-80570**	ea.	20268	
O-rings for Agilent Trap Fittings	5180-4181	25-pk.	22064	

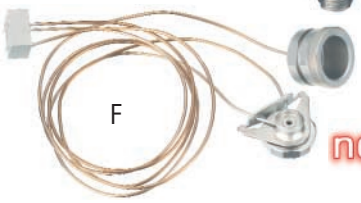




Direct Replacement Split/Splitless Injection Port for Agilent 6890/6850 GCs

Description	Similar to Agilent part #	qty.	cat.#	price
C) Replacement Weldment with EPC	G1544-60575	ea.	22674	
Silcosteel® Weldment with EPC	G1544-60575**	ea.	22672	
D Replacement Weldment*	19251-60575	ea.	20265	
Silcosteel® Weldment*	19251-60575**	ea.	20267	
E) Replacement Shell Weldment	G1544-80570	ea.	22673	
Silcosteel® Shell Weldment	G1544-80570**	ea.	22671	
F) Optional Split/Splitless Weldment (for use with large canister type filter)	G1544-60585	ea.	22686	
Silcosteel® Optional Split/Splitless Weldment (for use with large canister type filter)	G1544-60585**	ea.	22670	

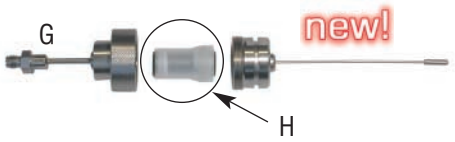




Replacement Chemical Trap for Agilent GCs

- Easy to install.
- Replaces original equipment.

Description	Similar to Agilent part #	qty.	cat.#	price
G) Optional Split Vent Trap Assembly (for use with large canister-type filter)	G1544-60610	kit	23031	
H) Replacement Traps (2) and O-rings (4)	G1544-80530	kit	23032	



**For use with manual flow or EPC on Agilent 5890 GCs. For use with manual flow only on Agilent 6890/6850 GCs.*
***Coated with Restek's exclusive Silcosteel® coating for increased inertness.*

HOT tech tip

To eliminate possible leaks and damage to the weldment, always replace split vent line, nut, and ferrule when installing a new shell weldment.

Injector Wrench for Agilent 5890/6890/6850 GCs

- Use to remove the septum nut and weldments during GC maintenance.
- High-quality stainless steel construction.
- Meets original equipment specifications.

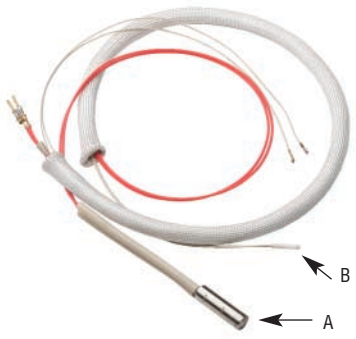
Description	Similar to Agilent part #	qty.	cat.#	price
Injector Wrench for Agilent 5890/6890/6850 GCs	19251-00100	ea.	22065	

Heater Cartridge & PRT Sensor for Agilent 5890 GCs

new!

- Use with 5890 FID and split/splitless weldments.
- Meets or exceeds OEM specifications.

Description	Similar to Agilent part #	qty.	cat.#	price
Injector/FID Heater/PRT Sensor Assembly	05890-61140	ea.	22068	
A) Injector/FID Heater	19231-60620	ea.	22069	
B) Injector/FID PRT Sensor	19231-60660	ea.	22070	



cool tools

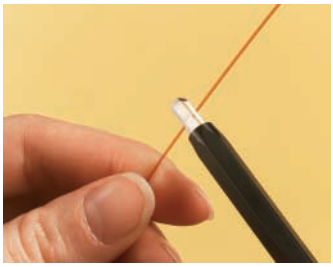
For Easier GC Maintenance Try These Tools from Restek

by Brad Rightnour and Michael Goss, Instrument Innovations Team

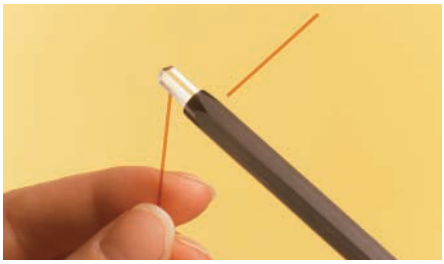
Sapphire Scribe

- Four long-lived sapphire cutting edges.
- Produces a clean, square cut in fused silica tubing.
- Clips in shirt or lab coat pocket—no hunting for a scribe when you need one.

cat.# 20182, (ea.)



One quick stroke...

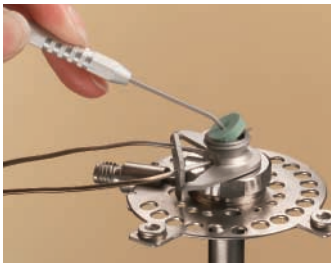


...and tap leaves a clean, square end.

Septum Puller

- Ideal to keep on hand in your laboratory—can be used in so many different ways.
- Hooked end can be used for removing septa and O-rings; sharp, pointed end works well for removing stuck ferrule fragments.

cat.# 20117, (ea.)



Remove septa without damaging an expensive weldment.

Dislodge a stuck ferrule quickly and easily - without scoring the fitting.

Mini Air Sampling Canisters

Available with Sulfinert™ Treatment

by Donna Lidgett, Air Monitoring Products Marketing Manager

✔ Ideal for indoor air, personal, emergency response, or soil gas sampling.
✔ 400cc or 1000cc.
✔ Available with quick-connect fitting (compatible with sampling and analysis instruments) or diaphragm valve.

These small canisters are designed for controlled sampling, such as personal air sampling, as an alternative to tube and pump samplers. The 1000cc canister is suitable for sampling volatile organic compounds in air according to US EPA Methods TO-14 and TO-15.

Restek offers these products in stainless steel or with Sulfinert™ coating for greatest inertness. We continue to offer passive coating technologies that are unmatched in the air sampling industry—try a Sulfinert™-treated canister and achieve the ultimate in analyte stability.

new!

Miniature Air Sampling Canisters with Quick Connect Stem Fitting

Description	Volume	qty.	cat.#	price
Electro-Polished Miniature Canister with Quick-Connect Stem Fitting	400cc	ea.	24188	
Sulfinert™-Coated Miniature Canister with Quick-Connect Stem Fitting	400cc	ea.	24189	
Sulfinert™-Coated Miniature Canister with Sulfinert™-Treated Quick-Connect Stem Fitting	400cc	ea.	24190	
Electro-Polished Miniature Canister with Quick-Connect Stem Fitting	1000cc	ea.	24194	
Sulfinert™-Coated Miniature Canister with Quick-Connect Stem Fitting	1000cc	ea.	24195	
Sulfinert™-Coated Miniature Canister with Sulfinert™-Treated Quick-Connect Stem Fitting	1000cc	ea.	24196	

Miniature Air Sampling Canisters with Metal-Seated Diaphragm Valve

Description	Volume	qty.	cat.#	price
Electro-Polished Miniature Canister with Metal-Seated Diaphragm Valve	400cc	ea.	24191	
Sulfinert™-Coated Miniature Canister with Metal-Seated Diaphragm Valve	400cc	ea.	24192	
Sulfinert™-Coated Miniature Canister with Sulfinert™-Treated Diaphragm Valve	400cc	ea.	24193	
Electro-Polished Miniature Canister with Metal-Seated Diaphragm Valve	1000cc	ea.	24197	
Sulfinert™-Coated Miniature Canister with Metal-Seated Diaphragm Valve	1000cc	ea.	24198	
Sulfinert™-Coated Miniature Canister with Sulfinert™-Treated Diaphragm Valve	1000cc	ea.	24199	

Helpful information about air sampling!

- ✔ Assembling a sampling train.
- ✔ Field sampling.
- ✔ Cleaning and certifying the sampling train and canister.

Request our new, free technical guide, A Guide to Passive Air Sampling, Using Canisters (lit. cat.#59977A).

RESTEK Behind the Scenes

Recommendations for Living, from the Dalai Lama

We couldn't persuade our colleagues to share their New Year's resolutions, so we went with an expert's advice. More in our next *Advantage*!

1. Take into account that great love and great achievements involve great risk.
2. When you lose, don't lose the lesson.
4. Remember that not getting what you want is sometimes a wonderful stroke of luck.
7. When you realize you've made a mistake, take immediate steps to correct it.
8. Spend some time alone every day.
10. Remember that silence is sometimes the best answer.
14. Share your knowledge. It's a way to achieve immortality.
15. Be gentle with the earth.
16. Once a year, go someplace you've never been before.
17. Remember that the best relationship is one in which your love for each other exceeds your need for each other.

Roger Greene Returns from Service in Kuwait

Restek gratefully salutes Roger, personal trainer, who has just returned from a tour of duty with the US Air Force 913th Security Force Squadron, in Kuwait. A letter to Restek from the Air Force expressed thanks to Roger for his service. We thank you too, Roger.

Other Restekians also have completed tours with our armed forces: Ken Herwehe, Tad Lucas, Alvira Peak, and Pete Zucco.

New Literature

- ✓ Acrylamide Analysis by GC
  Applications Note (lit. cat.# 59485)
- ✓ ASTM D-6352-98 Simulated Distillation
  Applications Note (lit. cat.# 59479)
- ✓ Better Responses for Chlorinated Pesticides by GC
  Applications Note (lit. cat.# 59487)
- ✓ Reference Materials for Florida UST
  Fast Facts (lit. cat.# 59395)
- ✓ Ultra Aqueous C18 HPLC Columns
  Fast Facts (lit. cat.# 59371)
- ✓ Vespel® Ring Inlet Seal—Fast Facts (lit. cat.# 59470)
- ✓ Instrument Innovations—Flyer (lit. cat.# 59278A)
- ✓ Syringe and Vial Essentials—Flyer (lit. cat.# 59225C)
- ✓ Air Monitoring Products
  Mini catalog (lit. cat.# 59661A)
- ✓ Alcoholic Beverage Components by GC
  Technical guide (lit. cat.# 59462)
- ✓ Guide to Direct Injections
  Technical guide (lit. cat.# 59882A)
- ✓ Guide to Passive Air Sampling with Canisters
  Technical guide (lit. cat.# 59977A)
- ✓ Guide to Split/Splitless Injections
  Technical guide (lit. cat.# 59880A)
- ✓ HPLC Column Selection
  Wall chart (lit. cat.# 59454)

Restek Supports Region's Cyclists

The Nittany Velo Club is a bicycle racing club/team whose membership includes just about every "serious" cyclist in Centre County, PA. For the past two years, Restek has sponsored the NVC, helping to defray the costs of traveling to races, entry fees, jerseys, equipment, etc., and helping the club stage their own cycling events and activities. The NVC performs about 500 man-hours of trail maintenance work in Rothrock State Forest annually and organizes the "Mount Nittany Classic," a 70 mile road race that will be held on April 27, 2003, on a circuit of Centre County back roads in the shadow of Mount Nittany. If you're in the neighborhood, stop by to watch—or join the race.

Want to Help Military Families Overseas?

Living overseas on military pay is not easy, and these families don't receive Sunday newspaper coupon inserts to help them stretch their budgets. Here's an easy way to help those that are serving away from home: Visit www.siteforsavings.com/content_mas/hlphand.htm for details, then clip the manufacturers' coupons from your newspaper inserts and send the ones you won't use to one of the addresses listed on the website—they are accepted in military commissaries, which carry many of the same brands offered in the States. If you have coupons that recently expired, they're still good—the commissaries will accept coupons up to six months after the expiration date.

ISO 9001 REGISTERED

Lit. Cat.# 59494-INT

Please direct your comments on this publication to Carrie Sprout, Graphic Designer, at carrie@restekcorp.com or call 814-353-1300, ext. 2151.

the RESTEK Advantage

Innovators of High Resolution Chromatography Products

Secure, Reliable Column-to-Column Connections

With a Vu2 Union™ Capillary GC Column Connector•

Restek Innovation!

By Mike Goss, Instrument Innovations Team, and Donna Lidgett, GC Accessories Marketing Manager

- ✔ Reliable seal integrity under rapid temperature changes or other stress.
- ✔ Easy to use.
- ✔ Visually confirm the seal.
- ✔ Fits all fused silica tubing from 0.33mm - 0.74mm OD.*

Our new Vu2 Union™ connector combines the simplicity of our Press-Tight® union with the durability of a metal union, to reliably couple an analytical column to a transfer line, a guard column, or another analytical column. The columns cannot unexpectedly disconnect if the connector is bumped or vibrated, or after repeated cycles to temperatures as high as 400ºC.

How does a Vu2 Union™ connector work?

A Press-Tight® union in the Vu2 Union™ connector joins the fused silica ends together (Figure 1); the ferrule and knurled nut at each end of the connector hold the tubing in place via a secondary seal between the ferrule and the Press-Tight® union. The knurled nuts apply pressure to the ferrules, to make a leak-tight seal. These ultra-strong connections will not unexpectedly disconnect when subjected to temperature changes, vibrations, or other stresses normally encountered in GC analyses. The open design allows visual evaluation of the seal between the column and the Press-Tight® union, to confirm the connection. The connector is designed to hang from the column cage, to minimize stress on the connections.

Who will benefit from using Vu2 Union™ connectors?

Any analyst using guard columns, transfer lines, or restrictor tubing, or performing a dual-column analysis with columns connected in series, or seeking to repair a broken column will find a Vu2 Union™ connector the simple, reliable, easy-to-use solution to their connection need (Figure 2, page 2).

•Patent pending.
**Restek 0.1mm - 0.53mm ID tubing.*

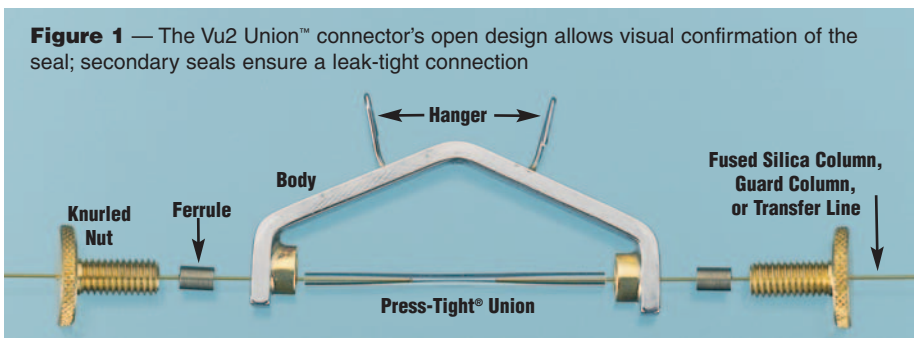


Figure 1 — The Vu2 Union™ connector's open design allows visual confirmation of the seal; secondary seals ensure a leak-tight connection

When should you use a Vu2 Union™ connector?

Use a Vu2 Union™ connector when you:

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

in this issue

FREE

Vespel® Ring Inlet Seals. See Page 19.

RESTEK
800-356-1688 ★ 814-353-1300
www.restekcorp.com

2003 vol. 2
INTERNATIONAL

Vu2 Union™ Connector Kits

Each kit includes: Vu2 Union™ body, 2 Press-Tight® unions, 2 knurled nuts, and 4 ferrules. Change column diameters - even mix column diameters - in the Vu2 Union™ body in any of the kits simply by ordering appropriate ferrules for the columns you wish to connect.

Description	Fits Column ID	qty.	cat.#
Vu2 Union™ Connector	0.15–0.25mm	kit	21105
Vu2 Union™ Connector	0.28/0.32mm	kit	21106
Vu2 Union™ Connector	0.45/0.50 & 0.53mm	kit	21107
Knurled nut		2-pk.	21108

Universal Press-Tight® Connectors

- Connect guard columns to analytical columns.
- Repair broken columns.
- Connect column outlets to transfer lines.

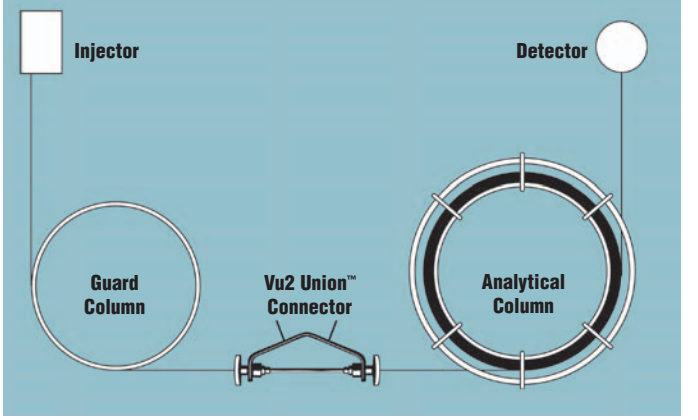
qty.	cat.#
5-pk.	20400
25-pk.	20401
100-pk.	20402

Deactivated, Universal Press-Tight® Connectors

- High-temperature silanization for excellent inertness.
- Ideal for trace analysis of active compounds.
- Ideal for analysis of pesticides, semivolatile pollutants, or clinical/forensic samples.

qty.	cat.#
5-pk.	20429
25-pk.	20430
100-pk.	20431

Figure 2 — A guard column connected to an analytical column by a Vu2 Union™ connector.



Graphite Ferrules for Vu2 Union™ Connectors

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

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

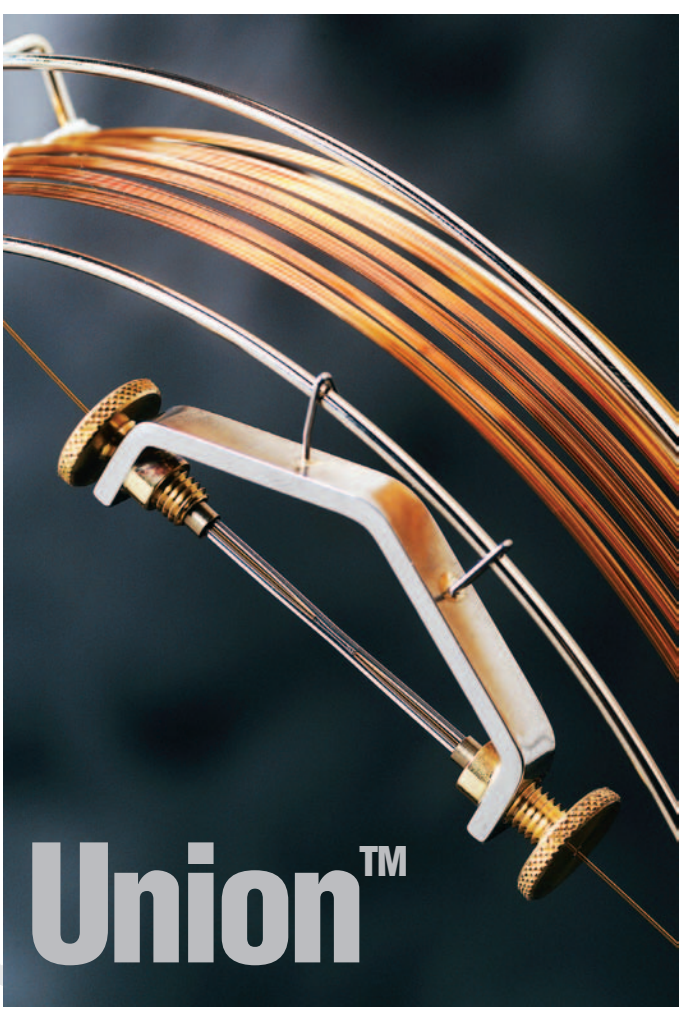
Intermediate-Polarity Deactivated Guard Columns & Transfer Lines

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.
0.10mm	0.363 ± 0.012mm	10041	
0.15mm	0.363 ± 0.012mm	10042	
0.18mm	0.37 ± 0.04mm	10046	
0.25mm	0.37 ± 0.04mm	10043	10043-600
0.28mm	0.37 ± 0.04mm	10003	10003-600
0.32mm	0.45 ± 0.04mm	10044	10044-600
0.45mm	0.69 ± 0.04mm	10005	10005-600
0.53mm	0.69 ± 0.05mm	10045	10045-600

Nominal ID	Nominal OD	10-Meter	10-Meter/6-pk.
0.25mm	0.37 ± 0.04mm	10049	10049-600
0.32mm	0.45 ± 0.04mm	10048	10048-600
0.53mm	0.69 ± 0.05mm	10047	

Polar Deactivated Guard Columns & Transfer Lines

Nominal ID	Nominal OD	5-Meter	10-Meter
0.25mm	0.37 ± 0.04mm	10065	10068
0.32mm	0.45 ± 0.04mm	10066	10069
0.53mm	0.69 ± 0.05mm	10067	10070



Efficient Analysis of Water-Miscible Solvents in Cleaning Products

Using an Rtx®-VMS Capillary GC Column

By Rebecca Wittrig, Ph.D., Senior Innovations Chemist

- ✔ Higher initial oven temperatures allow greater sample throughput.
- ✔ Excellent selectivity for resolving closely-related alcohols and other solvents.
- ✔ Consistent column-to-column performance for quality control analyses.

Consumers use a wide range of products to promote personal hygiene, improve personal appearance, and reduce levels of microorganisms in the home environment. Their choices make the cleaning and personal care products industry a multi-billion dollar industry.[1] The Soap and Detergent Association (SDA)[2] groups soaps and detergents into four general categories: personal cleansing, laundry, dishwashing, and household cleansing. As with all other consumer products, there is a need to test both raw materials and final products in each of these categories. Composition and quality control analyses for many of these products can be performed by gas or liquid chromatography. For example, volatile components, such as alcohols, can be monitored by using an Rtx®-VMS capillary GC column.

Various ingredients are needed in cleaning and personal care products, to solubilize soils, wet surfaces, mask odors, provide color, or perform other functions. Solvents are included in these products primarily to dissolve organic soils. Aside from safety considerations, the main criterion for a solvent used in cleaning products is miscibility with water, as the solvent must form a solution with other water-soluble components. Solvents that meet the criteria for use in consumer cleaning products include alcohols and glycols. In addition to water miscibility, these solvents clean without leaving residue, making them especially useful in products designed for cleaning environmental surfaces, such as glass cleaners.

Water-soluble solvents in cleaning products are analyzed by gas chromatography to ensure product quality and to further new product development. A 60m x 0.25mm ID x 1.4µm film Rtx®-VMS capillary GC column is an excellent choice for analyzing a wide range of cleaning solvents (Figure 1). These columns exhibit excellent selectivity for closely related alcohols, such as ethanol, isopropanol, *tert*-butanol, and *n*-butanol. An Rtx®-VMS column is compatible with higher initial oven temperatures—note the 60°C starting temperature in Figure 1—allowing greater sample throughput due to faster oven stabilization time. Analyses of typical consumer cleaning products, an all-purpose cleaner and a glass cleaner, are shown in Figure 2.

Coming soon!
New technical guide:
Analyzing Cleaning and Personal Care Products by Gas and Liquid Chromatography

Summary
An Rtx®-VMS capillary column makes these analyses faster, with highly reliable results, and is an excellent choice for this application.

References
1. Branna, T. *The I&I Market. Happi,* Nov. 2000.
2. The Soap and Detergent Association. Website: www.sdahq.org

Ordering Information | Rtx®-VMS Columns

Rtx®-VMS columns are listed on page 4.

Figure 1 — Commonly used cleaning solvents on an Rtx®-VMS column. Unique selectivity allows a 60°C starting temperature, for fast sample throughput.

1. methanol
2. ethanol
3. *iso*-propanol
4. *tert*-butanol
5. *n*-propanol
6. *iso*-butanol
7. methyl Cellosolve®
8. *n*-butanol
9. Cellosolve®
10. ethylene glycol
11. butyl Cellosolve®
12. limonene
13. dipropylene glycol monomethyl ether (DPGMME)
14. diethylene glycol ethyl ether (DEGEE)
15. phenyl Cellosolve®
16. tripropylene glycol monomethyl ether (TPGMME)

Rtx®-VMS, 60m, 0.25mm ID, 1.4µm (cat.# 19916)
Sample: 1% each solvent in methanol
Inj.: 1.0µL split (split ratio 100:1), 4mm inlet liner (cat.# 20814)
Inj. temp.: 250°C
Carrier gas: hydrogen, constant flow
Flow rate: 1.5 mL/min.
Oven temp.: 60°C (1 min. hold) to 138°C @ 4°C/min., to 145°C @ 2°C/min., to 210°C @ 15°C/min., to 230°C @ 5°C/min.
Det.: FID @ 250°C

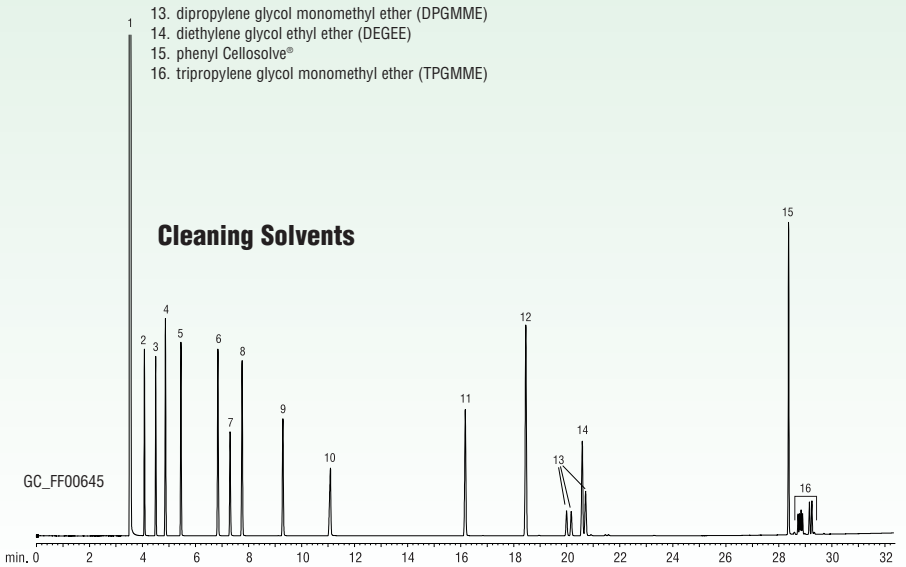
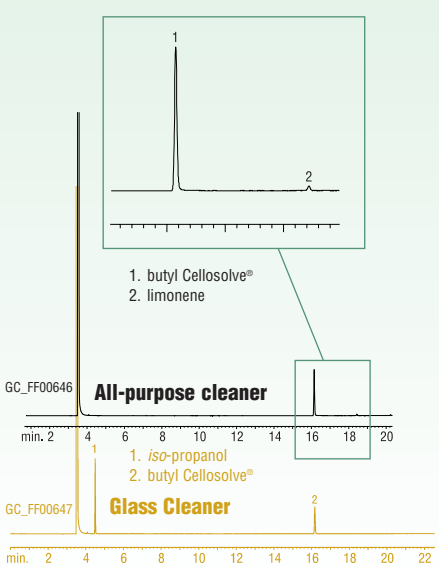


Figure 2 — Use an Rtx®-VMS column to quantify a wide range of cleaning solvents.

Rtx®-VMS, 60m, 0.25mm ID, 1.4µm (cat.# 19916)
Sample: Approx. 5% sample in methanol
Inj.: 1.0µL split (split ratio 100:1), 4mm inlet liner (cat.# 20814)
Inj. temp.: 250°C
Carrier gas: hydrogen, constant flow
Flow rate: 1.5 mL/min.
Oven temp.: 60°C (1 min. hold) to 138°C @ 4°C/min., to 145°C @ 2°C/min., to 210°C @ 15°C/min., to 230°C @ 5°C/min.
Det.: FID @ 250°C



Resolve Trace Oxygenates from a Gasoline/Water Composite

Using an Rtx®-VMS Capillary GC Column

By Christopher English, Environmental Innovations Chemist

- ✔ High accuracy—oxygenate recoveries better than 90%.
- ✔ Resolve oxygenates from potentially interfering gasoline components and volatile target compounds, by US EPA Method 8260.
- ✔ High speed—30-minute cycle time.

With the elimination of lead from gasolines, oxygen-containing compounds have become important performance-enhancing components. Oxygenated compounds most commonly added to gasoline are methanol, ethanol, *tert*-butanol (TBA), methyl *tert*-butyl ether (MTBE), diisopropylether (DIPE), and ethyl-*tert*-butylether (ETBE). Of these, MTBE is the primary additive. Contamination of ground and surface water with these and other gasoline components is a major concern. Identifying and quantifying the oxygenates from among the highly concentrated hydrocarbons in a gasoline/water matrix is a challenging task. Some compounds (e.g., MTBE and TBA) coelute on many capillary GC column stationary phases and share ions used for identification by MS.

Our investigations, and others, show that US EPA Method 8260, a purge and trap / capillary GC / mass spectrometry method, is the most reliable method for detecting oxygenated components in complex gasoline/water samples, regardless of the concentration of the gasoline.[1] In the United States, the oxygenates have not been written into any US EPA Method, with the exception of MTBE in Method 524.2. The ethers can be concentrated by purge and trap, but this approach has not been validated in any SW-846 method. Methanol and ethanol are poorly suited to analysis by purge and trap techniques. In Method 8015, a flame ionization detector (FID) is used to match a known pattern of gasoline with an unknown sample containing peaks that fall within the gasoline pattern range. This method can be used to identify oxygenates by retention time, but the high probability of misidentifications dictates confirmation on a second column. Method 8021 is specifically for analysis of aromatic and halogenated volatiles, with detection by photoionization detector (PID). This is the least desirable of the potential methods for monitoring oxygenates, because the PID is very sensitive to double bonds, but is much less sensitive to oxygenates. Our analysis of a gasoline composite standard, for example, produced a false positive for diisopropyl ether. Using GC/MS for confirmation, the compound was identified as 2-methyl-1-pentene.[2] Despite this problem, many state GRO methods use PID for the analysis of MTBE.

We evaluated the performance of four stationary phases for recovery of oxygenates, verifying passing criteria using modified EPA Method 5030B and Method 8260.[3] Non-oxygenated gasoline samples were spiked with low (ppb) levels of oxygenates to determine if operating conditions were appropriate for separating and detecting the target compounds in the presence of high concentrations of gasoline hydrocarbons. Purge and trap conditions in Method 5030B were modified for concentrating the oxygenates: we replaced the standard ambient purge with a 40°C purge. When possible, GC oven conditions were optimized for each stationary phase, to overcome coelutions of analytes that share ions (e.g., TBA and MTBE).

The instrument was calibrated using a 5-point curve. We calculated response factors (RFs) & relative standard deviations (RSDs) for the target compounds in Method 8260, then added all of the target compounds and the correct Method 8260 internal and surrogate standards to our calibration mix (84 additional target compounds), to ensure there were no coelutions of 8260 target compounds with the oxygenates. Of the columns used in this investigation, a 30-meter, 0.25mm ID, 1.4µm film Rtx®-VMS column proved best for identifying and quantifying oxygenates in a gasoline/water mix.

Figure 1 shows an analysis of a 1ppm non-oxygenated gasoline standard in water, spiked with 5ppb of each of the oxygenates, and illustrates the value of the Rtx®-VMS column in identifying and quantifying oxygenates in high levels of gasoline hydrocarbons.

The inset to the center in Figure 1 shows a portion of the total ion chromatogram with the extracted ion chromatogram for the oxygenates to scale. The inset to the center is an enlargement of the extracted ion chromatogram for the oxygenates; the clean peaks indicate that there is no interference from non-target gasoline fragmentation ions. TBA and MTBE are well resolved using the 35°C initial temperature. The column elutes the methyl-naphthalenes in less than 23 minutes, with a cycle time of 30 minutes. Using average response factors calculated from the calibration curve, we determined that oxygenate recoveries were better than 90%.[4]

This investigation established that an Rtx®-VMS column resolves oxygenates from potentially interfering gasoline components and Method 8260 target compounds. It is well suited to resolving the expanding Method 8260 target compound list, and can be used to identify low levels of analytes in contaminated/complex matrixes. An Rtx®-VMS column is the clear choice for the most demanding volatile organics analysis.

References

1. Happel, A.M., E.H. Beckenbach, R.U. Halden, *An Evaluation of MTBE Impacts to California Groundwater Resources* Lawrence Livermore National Laboratory, UCRL-AR-130897 (1988). http://www-erd.llnl.gov/mtbe/pdf/mtbe.pdf
2. C. English, C. Cox, F. Dorman, D. Patwardhan, *The Analysis of Gasoline Oxygenates Using a New Capillary Column Stationary Phase*, Pittsburgh Conference 2001, Session 199 (poster). http://www.restekcorp.com/2001/1868P.pdf
3. U.S. Environmental Protection Agency, *Volatile Organic Compounds by Gas Chromatography/Mass Spectroscopy (GC/MS): Capillary Column Technique Method 8260*. July 1992 Revision 0, US EPA Office of Solid Waste. Washington, D.C.
4. C.M. English, F.L. Dorman, G.B. Stidsen, *The Analysis of Gasoline Oxygenates by EPA Method 8260B* Pittsburgh Conference 2003, Session 590-6P (poster). http://www.restekcorp/pittcon2003.htm#slides

For more details of this work, see reference 4.

Ordering Information | Rtx®-VMS (Fused Silica)

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter
0.25mm	1.40	-40 to 240/260°C	19915	19916	
0.32mm	1.80	-40 to 240/260°C	19919	19920	
0.45mm	2.55	-40 to 240/260°C	19908	19909	
0.53mm	3.00	-40 to 240/260°C	19985	19988	19974
ID	**df (µm)**	**temp. limits**	**20-Meter**	**40-Meter**	
0.18mm	1.00	-40 to 240/260°C	49914	49915	

California Oxygenates Mix

diisopropyl ether	2,000µg/mL
ethyl-*tert*-butyl ether	2,000
tert-amyl methyl ether	2,000
tert-butyl alcohol	10,000
methyl *tert*-butyl ether	2,000

In P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30465	30465-510	—
	w/data pack	
30465-500	30465-520	30565

8260B MegaMix™ Calibration Mix

(76 + 1 components)

*2,000µg/mL each in P&T methanol, 1mL/ampul**

Each	5-pk.	10-pk.
30475	30475-510	—
	w/data pack	
30475-500	30475-520	30575

*2-chloroethyl vinyl ether provided in a separate ampul.

Figure 1 — Detect trace oxygenates, using an Rtx®-VMS column.

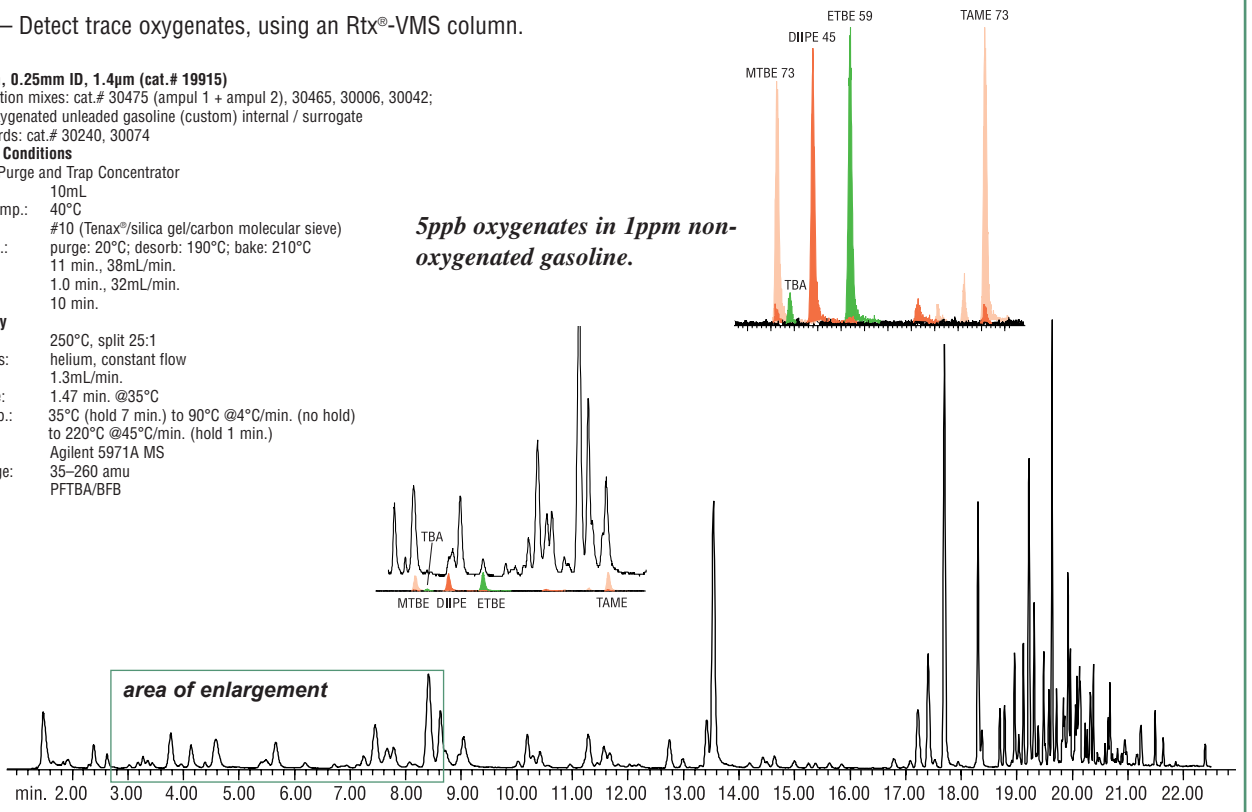
Rtx®-VMS, 30m, 0.25mm ID, 1.4µm (cat.# 19915)
Sample: calibration mixes: cat.# 30475 (ampul 1 + ampul 2), 30465, 30006, 30042; non-oxygenated unleaded gasoline (custom) internal / surrogate standards: cat.# 30240, 30074

Purge and Trap Conditions

O.I. 4560 Purge and Trap Concentrator	
Sample:	10mL
Sample Temp.:	40°C
Trap:	#10 (Tenax®/silica gel/carbon molecular sieve)
Trap Temp.:	purge: 20°C; desorb: 190°C; bake: 210°C
Purge:	11 min., 38mL/min.
Desorb:	1.0 min., 32mL/min.
Bake:	10 min.

Chromatography

Inj. Temp.:	250°C, split 25:1
Carrier Gas:	helium, constant flow
Flow Rate:	1.3mL/min.
Dead Time:	1.47 min. @35°C
Oven Temp.:	35°C (hold 7 min.) to 90°C @4°C/min. (no hold) to 220°C @45°C/min. (hold 1 min.)
Det.:	Agilent 5971A MS
Scan Range:	35–260 amu
Tune:	PFTBA/BFB



GC_EV00679

Acknowledgement: purge and trap courtesy of O.I. Analytical.

US EPA Underground Storage Tank (UST) Monitoring Program

Reference publications for recommended methods

- ✔ Helpful checklists for the latest state and EPA UST methods:
  - *– analytical reference materials*
  - *– sample preparation supplies*
  - *– chromatography columns and accessories*
- ✔ Conveniently organized by method—easy setup / easy reorder of consumables

In the late 1980s the US Environmental Protection Agency (US EPA) established the Office of Underground Storage Tanks (OUST) to enforce federal laws on environmental contamination from petroleum products. Underground Storage Tank (UST) systems installed before December 22, 1988 had no protection against spills or overfills, and were likely to corrode and leak. OUST mandated that by December 22, 1998, all UST systems were to be prevented from contaminating nearby groundwater and soil. Existing systems were to be protected from spills, overfills, and corrosion, or replaced with new systems incorporating such protection.

Many of the unprotected UST systems have been properly treated, but the need for monitoring UST systems persists. OUST has been actively enforcing federal UST regulations.

OUST has recommended specific EPA methods for UST applications. A majority of the states still use these methods, but many states have developed methods of their own for UST analysis.

To help laboratories comply with and use the appropriate analytical procedures, Restek has been active in following EPA and state guidance. Based on our knowledge and experience with these methods, our chemists have developed lists of the appropriate chromatographic tools, and formulated analytical reference products, to help ensure successful analyses. Comprehensive listings of quality chromatographic columns, analytical reference materials, and sample preparation products for latest state methods are featured in an expanding group of Restek publications. In these listings, you will find everything you need to quickly set-up or reorder consumables for UST methods.

Request comprehensive product listings for latest state UST methods

Reference Standards
- Pattern Recognition Standards
  - GRO – gasoline composites, including composite weathered gasoline standards
  - DRO - including a composite arctic range diesel standard
  - RRO - motor oil composites, including composite used motor oil
  - Weathered fuels and oils
  - Mineral oils
  - Military fuels
- State-specific UST calibration formulations
- Retention time marker standards
- Internal standards
- Surrogate standards
- Reference materials conveniently organized by method number
- Custom mixtures available - ask us for a quote

For copies of these publications, fill out the reply card at the center of this ***Advantage***. Select from these current UST publications: AK - CA - FL - IA - MA - TX - WI - Northwest (WA / OR) or USEPA

For custom mixes, use the quote form in our general catalog, or on our web site (www.restekcorp.com), or fax 814-355-2895.

Remember: we will be happy to provide a quote on any custom reference mixture, column, or other consumable you need!

Improved Resolution of Dioxins and Furans by GC-High-Resolution Mass Spectrometry,

Using an Rtx®-Dioxin Capillary Column

By Frank L. Dorman, Ph.D., Director of Technical Development

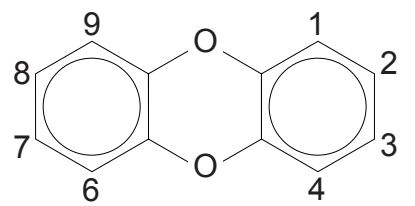
- ✔ Improved separation of dioxin and furan congeners, compared to 5% diphenyl columns.
- ✔ Greater thermal stability than 5% diphenyl columns or high-cyano confirmation columns.
- ✔ May eliminate confirmation analysis.

Gas chromatographic analysis coupled to high-resolution mass spectrometry is a common method of evaluating environmental samples for dioxins and furans. Dioxins and furans are monitored due to the toxicity of congeners that have chlorine substitution at positions 2, 3, 7, and/or 8 (Figure 1). In the US, the most common analysis methods for these compounds are USEPA methods 1613 and 8290, but the analysis is performed similarly in many countries. The overall goal of the analysis is to accurately quantify the 17 toxic dioxin and furan congeners by separating them from 119 other congeners.

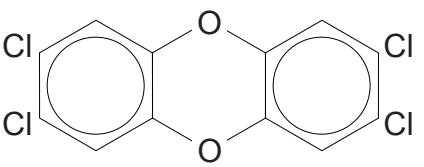
In order to achieve the desired separation, most methods describe an initial analysis on a 5% diphenyl/95% dimethyl polysiloxane stationary phase. If 2-, 3-, 7-, and/or 8-substituted congeners are detected in this analysis, most methods require a confirmatory analysis on a stationary phase that separates these congeners from the less toxic congeners. While no single column has been universally agreed upon as the best confirmation column, most analysts use a high-cyano stationary phase. While these columns offer better separation of the 2-, 3-, 7-, 8-substituted congeners, analysts using any of them must contend with poor thermal stability (maximum operating temperatures of approximately 250ºC) and poor column lifetimes, compared to 5% diphenyl-type columns used for the primary analysis. The difficulty with using the results from 5% diphenyl columns is that there are several known coelutions of environmentally-occurring 2-, 3-, 7-, 8-substituted congeners with less toxic congeners. This leads to falsely high values for the toxic congeners on the 5% diphenyl column, and to unnecessary confirmatory analysis.

An ideal stationary phase for this application would combine excellent separation, high thermal stability and, thereby, long column lifetime. With these goals in mind, Restek has developed the Rtx®-Dioxin column. The new, proprietary stationary phase, specifically developed for dioxins/furans analysis, is stable to temperatures above 425ºC. When coated onto high-temperature fused silica tubing, the thermal limit of the column is a function of the polyimide outer coating: 380ºC. Not only is this is a major improvement over the thermal stability of high-cyano phases, it is an improvement over the capabilities of 5% diphenyl phases as well.

Figure 1 — Chlorine substitution in the basic dioxin structure creates 136 congeners.



General structure for dioxin congeners



3Å by 8Å molecule fits PAH receptors, makes 2,3,7,8-tetrachlorodibenzodioxin especially toxic

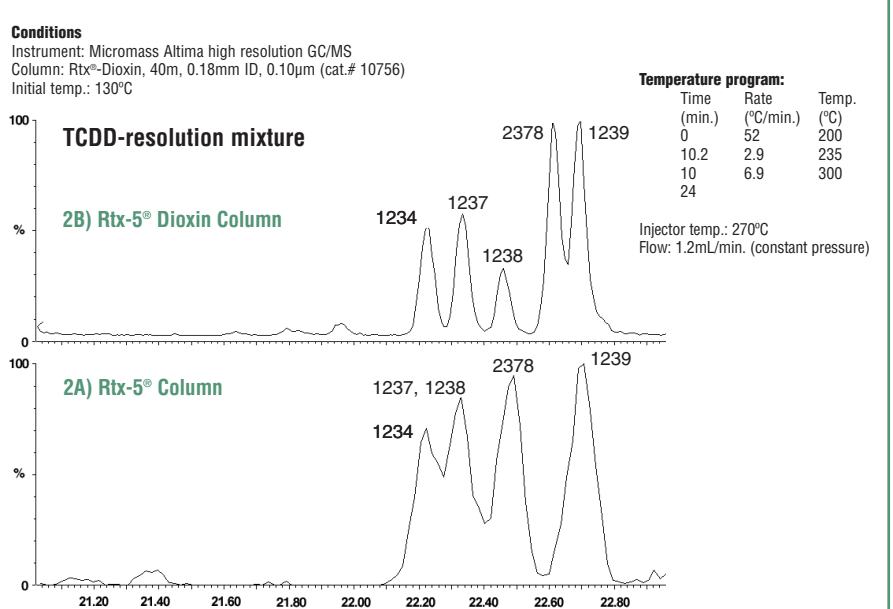
An Rtx®-Dioxin column better separates the dioxin and furan congeners, compared to 5% diphenyl columns. Most analysts experienced with dioxin and furan separations are familiar with the 4-peak tetrachlorodibenzodioxin mass pattern from a 5% diphenyl column, as shown in Figure 2A. An Rtx®-Dioxin column separates all five components in the resolution check mixture for this mass window—a significant improvement (Figure 2B). Note that 1,2,3,7- TCDD and 1,2,3,8-TCDD are tentatively identified; reference materials for individual congeners are not available.

Because few of the individual dioxin and furan congeners are available as reference materials, analysis of fly ash extracts is the accepted test of whether a column resolves the toxic congeners from the non-toxic congeners. In analyses of three fly ash extracts used in a recent international round-robin study, data from an Rtx®-Dioxin column agreed to within ±10% of the "true" values for all 2-, 3-, 7-, 8-substituted congeners, except for one penta- and one hexa-furan. This also is a significant improvement over 5% diphenyl column performance for the primary analysis. Table 1 (page 7) summarizes data for 2,3,7,8-tetrachlorodibenzofuran from an Rtx®-Dioxin column, a 5% diphenyl column, and a high-cyano column, compared to median and mean from an international round-robin study. Excellent agreement between the median and the mean, and between the Rtx®-Dioxin column and the study data, gives confidence in the proximity to the "true" value. Further work toward optimizing flow and oven temperature is in progress, to determine if the Rtx®-Dioxin column could eliminate the need for high-cyano phases for confirmation.

In summary, the new Rtx®-Dioxin column is a significant improvement over 5% diphenyl columns commonly used in the primary analysis for dioxins and

Continued on page 7

Figure 2 — Rtx®-Dioxin column separates all five components in the resolution check mixture.



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

Dioxins and Furans by GC-High Resolution MS... Continued from page 6

furans. The column also shows potential as a replacement for high-cyano confirmation phases with poor thermal stability and short lifetimes. We continue to work to optimize a temperature program that resolves all of the toxic congeners, with a goal of eliminating the need for confirmation.

If you are involved in the analysis of dioxins and furans, and would like additional information about Rtx®-Dioxin columns, please contact Frank Dorman at 1-800-356-1688, ext. 2186, or by e-mail at frank@restekcorp.com

Acknowledgements
Chromatography courtesy of Karen MacPherson and Eric Reiner, Ontario Ministry of the Environment, Etobicoke, ON, Canada.
Reference materials courtesy of Brock Chittam, Wellington Laboratories, Guelph, ON, Canada.

Table 1 — Excellent agreement between Rtx®-Dioxin column and round-robin study data.

	Column / 2,3,7,8-tetrachlorodibenzofuran (pg/g)				
Sample	DB-5*	DB-225**	Rtx®-Dioxin	Median***	Mean***
Fly Ash A	250	21	30	28	32
Fly Ash B	2100	300	378	390	390
Fly Ash C	170	19	28	27	32

*5% diphenyl column.
**high-cyano column.
***n > 110 laboratories.

Ordering Information | Rtx®-Dioxin Columns

ID	df (µm)	temp. limits	40-Meter	60-Meter
0.18mm	0.10	-60°C to 380°C	10756	—
0.25mm	0.15	-60°C to 380°C	—	10755

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Marcel Dekker, 2003, 448 pp.
ISBN 0-8247-0950-0
cat.# 21467

Dioxin 2003

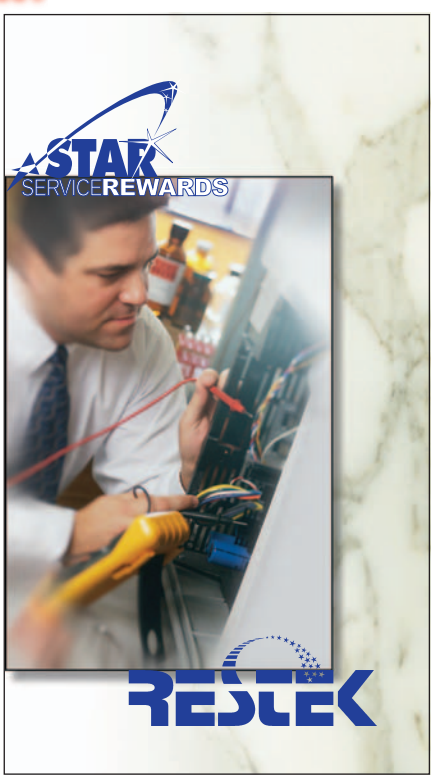
The annual meeting of world experts on these important and controversial materials, the 23rd International Symposium on Halogenated Organic Pollutants and Persistent Organic Pollutants, will be held at the Westin Copley Place Hotel, Boston, MA, August 24-29, 2003.

Specialists in dioxin research will make more than 500 presentations and discuss current knowledge. "Hot topics" sessions will focus on endocrine disrupters, Arctic POPs, neurotoxicity, ultimate trace method, and more.

For details, visit www.dioxin2003.org or contact Laura Biringer, 617-262-3424 / Lbiringer@mpwi.org

Dioxin 2003

new!



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Restek Corporation has formed an alliance with some of the premiere independent instrument service providers in the United States, with a goal of bringing you the finest chromatography operating supplies, equipment service, and applications support available.

We are pleased to introduce a new program that pays you for using Restek products, by reducing your costs for quality instrument service: STAR Service Rewards. Similar to our popular Restek Wizard Dollar program, STAR Service Rewards pays you one STAR Point for every $50 of Restek products you purchase. Redeem STAR Points with participating STAR member service providers for selected service, equipment, and training products. You get the finest chromatography operating supplies from Restek and high quality instrument service from your preferred service provider. STAR Service Rewards is one more example of why Restek is the company chromatographers trust for complete chromatography support.

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Faster Separations and Greater Sensitivity

Using Restek HPLC Columns and Fast LC Cartridge Columns

By Vernon Bartlett, Innovations Team Manager, Terrence S. Reid, HPLC Applications Chemist, and Rebecca Wittrig, Ph.D., Senior Innovations Chemist

- ✔ Save time—significant increases in sample throughput; fast re-equilibration in gradient analyses.
- ✔ Save money—reduced solvent consumption reduces purchase and disposal costs.
- ✔ Good screening technique for unknown analytes.
- ✔ Excellent for LC/MS applications.

Analysis time for many HPLC separations can be drastically reduced by using fast LC. In the example separations we show here, analysis times of 20 to 40 minutes by conventional HPLC are reduced by 60 to 75%. Further, because columns employed in fast LC analyses typically are less than 100mm long, analytes spend less time in the column. Consequently, these dramatically reduced analysis times are accompanied by improved sensitivity, due to reduced band spreading.

Fast LC analyses can be performed using either cartridge-style fast LC columns or short, conventional design HPLC columns, typically containing 3µm silica-based packings. In addition to the performance improvements attributable to short columns and small packing particles, gains also can be realized by using optimized, highly selective stationary phases. Selective phases improve separation among sample components with minimal changes in mobile phase strength. Analysts who reduce mobile phase strength in attempts to improve selectivity and/or retention often find k' is increased drastically, and analysis time is unacceptably prolonged.

There are several precautions to observe before using fast LC columns and methods. Critical separations are more sensitive to system volume; evaluate tubing lengths and system component specifications with a goal of minimizing internal volume. Also, highly selective stationary phases can be required for difficult separations (e.g., structural isomers)—we recommend discussing your intended application with our Technical Service group before ordering columns and attempting a new analysis. Finally, fast LC is not recommended for normal phase separations, nor for ion-pairing separations when gradients are required.

Figure 1 — *Digitalis* derivatives separated in 3 minutes on a fast LC cartridge.

Peak List:	Conc.	Ret. Time (min.)
1. digoxigenin	100µg/mL	0.40
2. gitoxigenin	100µg/mL	0.80
3. digoxin	100µg/mL	1.10
4. gitoxin	~10µg/mL	2.20
5. digitoxin	100µg/mL	2.60

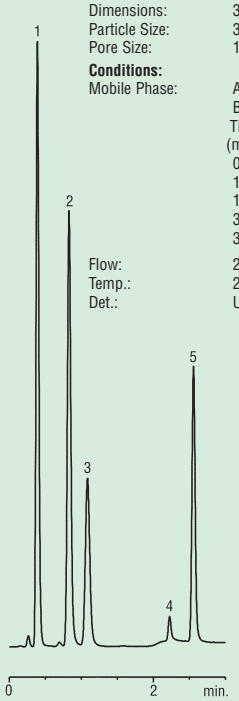
Sample:
Inj.: 10µL
Sample Diluent: water:acetonitrile (80:20 v/v)

Column: Ultra Alkaloids Fast LC Cartridge
Catalog #: custom column
Dimensions: 30 x 4.0mm
Particle Size: 3µm
Pore Size: 100Å

Conditions:
Mobile Phase: A: water
B: acetonitrile

Time (min.)	%B
0.0	20
1.5	20
1.51	35
3.0	35
3.1	20

Flow: 2.0mL/min
Temp.: 27°C
Det.: UV @ 230nm



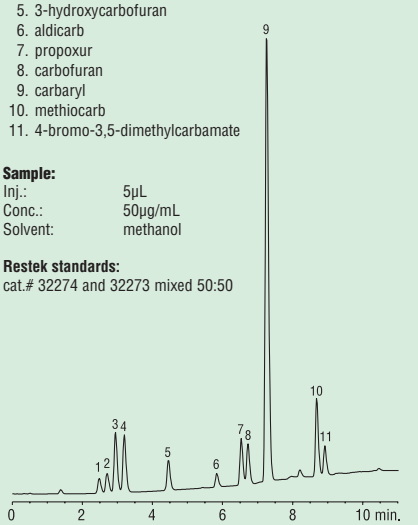
LC_0250

Figure 2 — Separate carbamates and re-equilibrate in 13 minutes, using a short Ultra Carbamate column.

Peak List:
1. aldicarb sulfone
2. aldicarb sulfoxide
3. oxamyl
4. methomyl
5. 3-hydroxycarbofuran
6. aldicarb
7. propoxur
8. carbofuran
9. carbaryl
10. methiocarb
11. 4-bromo-3,5-dimethylcarbamate

Sample:
Inj.: 5µL
Conc.: 50µg/mL
Solvent: methanol

Restek standards:
cat.# 32274 and 32273 mixed 50:50



LC_0225

Column: Ultra Carbamate
Catalog #: 9177355
Dimensions: 50 x 4.6mm
Particle size: 3µm
Pore size: 100Å

Conditions:
Mobile Phase: A: 90:10 water:methanol
B: 90:10 methanol:acetonitrile

Time (min):	%B
0	10
10	90

Flow: 1.5mL/min
Temp.: 27°C
Det.: UV @ 220nm

Fast LC Separation of *Digitalis* Derivatives
Figure 1 shows an analysis of *Digitalis* extracts and derivatives on an experimental 30mm x 4.0mm Ultra Alkaloids Fast LC cartridge column, using a simple water:acetonitrile mobile phase gradient. The analysis is completed in 3 minutes, with excellent separation of the sample components. This is dramatically reduced analysis time, relative to the cumbersome analysis on a 30cm C18 column. The fast LC approach, using this specialized stationary phase, can be applied to purification and analysis of digoxin-labeled materials in investigations of biological activity, and is perfect for high-speed cleaning validations.

Fast LC Separation of Carbamates
Figures 2 and 3 (page 9) illustrate fast LC analyses of carbamate pesticides by HPLC/UV and LC/MS, respectively. Total time for the HPLC/UV analysis is approximately 13 minutes; the LC/MS analysis is slightly longer, but is less than 20 minutes. Table I (page 9) summarizes conditions for the LC/MS analysis. Including re-equilibration time, the conventional analysis on a 250mm x 4.6mm column takes 40 minutes. Designed especially for carbamates analyses, Ultra Carbamate columns are available in several dimensions in addition to the 50mm x 4.6mm and 100mm x 4.6mm columns used here.

Fast LC Analyses of Vanillin and Vanilla Extract
Figure 4 (page 9) shows fast LC analyses of vanillin/ethyl vanillin and vanilla bean extract on a 50mm x 4.0mm Pinnacle™ DB C18 column. Excellent separations are achieved in less than 5 minutes. Conventional analyses on 150mm x 4.6mm C8 columns take 25 minutes overall with a mobile phase gradient, or more than 40 minutes with an isocratic mobile phase.

In Summary
In diverse applications, fast LC separations enhance laboratory throughput, reduce solvent waste, and improve method sensitivity. In some cases, mobile phase requirements can be simplified, from gradient elution to isocratic elution, when an optimized stationary phase is used, dramatically reducing analysis time.

With these precautions observed, fast LC can be an excellent time- and money-saving tool for many analysts and many applications. If you would like to discuss whether this approach is suitable for your application, please contact our Technical Service Group; they are ready to help you.

Table 1 — Chromatographic conditions for LC/MS analysis of carbamates.

Column:	**Ultra Carbamate**
Catalog #:	9177315
Dimensions:	100 x 4.6mm
Particle Size:	3µm
Pore Size:	100Å
Inj.:	10µL
Sample:	531.1 Carbamate Pesticide Calibration Mix (cat.# 32273)
Conc.:	100µg/mL each
Solvent:	methanol
Mobile Phase:	A: 90:10 water:methanol + 10mM ammonium formate B: 10:90 acetonitrile:methanol + 10mM ammonium formate
Gradient:	90:10 A:B to 10:90 A:B from 0-15 min.
Flow:	1mL/min. (0.75mL/min. to UV, 0.25mL/min. to MS)
Temp.:	ambient

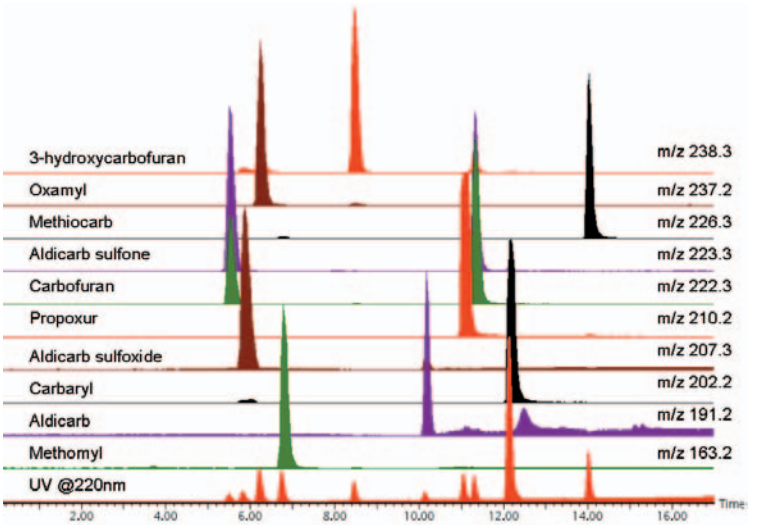
MS Conditions:

Det.:	Micromass ZMD
Mode:	ESI+
Capillary V:	3.50
Extractor:	4.0
Ion Energy:	0.4
Multiplier:	650
Source Temp.:	100°C
Desolv. Temp.:	250°C
Gas Flow:	490 L/hr.

Compound	Ion	Cone V
1. aldicarb sulfone	223.3	25V
2. aldicarb sulfoxide	207.3	18V
3. oxamyl	237.2*	10V
4. methomyl	163.2	15V
5. 3-hydroxycarbofuran	238.3	15V
6. aldicarb	191.2	8V
7. propoxur	210.2	18V
8. carbofuran	222.3	22V
9. carbaryl	202.2	18V
10. methiocarb	226.3	19V

*Ammonium adduct; others [M+H]+ ions.

Figure 3 — LC/MS analysis of carbamates in less than 20 minutes, using an Ultra Carbamate column.



Conditions listed in Table I

Figure 4 — Fast, highly selective analysis of vanillin/ethyl vanillin and vanilla bean extract on a Pinnacle™ DB C18 column.

Peak List:	**Ret. Time (min.)**
1. vanillin	2.00
1. vanillin	2.00
2. ethyl vanillin	4.20

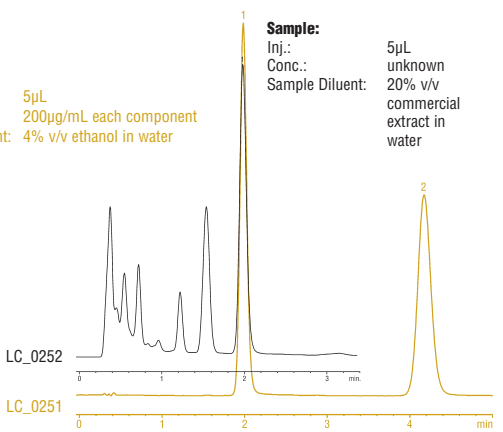
Sample:

Inj.:	5µL
Conc.:	200µg/mL each component
Sample Diluent:	4% v/v ethanol in water

Column:	**Pinnacle™ DB C18**
Catalog #:	9414555
Dimensions:	50 x 4.6mm
Particle Size:	5µm
Pore Size:	140Å
Conditions:	
Mobile Phase:	A: 0.2% v/v conc. phosphoric acid in water B: methanol Isocratic: 80%A: 20%B
Flow:	2mL/min
Temp.:	27°C
Det.:	UV @ 254nm

Sample:

Inj.:	5µL
Conc.:	unknown
Sample Diluent:	20% v/v commercial extract in water



3µm Fast LC Cartridges

Description	Length	2.1mm ID	4.0mm ID
Ultra C18 Fast LC Cartridge	30mm	91743320	91743340
Ultra Aqueous C18 Fast LC Cartridge	30mm	91783320	91783340
Ultra Cyano Fast LC Cartridge	30mm	91063320	91063340
Ultra PFP Fast LC Cartridge	30mm	91763320	91763340

Fast LC Cartridge Holder

Description	qty.	cat.#
Fast LC Cartridge Holder	ea.	25298

Fast LC Development Kits

Four Fast LC Cartridges (Ultra C18, Ultra Aqueous C18, Ultra Cyano, Ultra PFP), Fast LC Cartridge Holder.

Description	qty.	cat.#
Fast LC Development Kit—30 x 2.1mm	kit	25296
Fast LC Development Kit—30 x 4.0mm	kit	25297

Additional HPLC Columns

Description	Dimensions	cat.#
Ultra Carbamate, 3µm	50mm x 4.6mm ID	9177355
Ultra Carbamate, 3µm	100mm x 4.6mm ID	9177315
Pinnacle™ DB C18, 5µm	50mm x 4.6mm ID	9414555

Request our 2003 HPLC minicatalog
Lit. Cat.# 59241A

New Reference Materials

By Katia May, Ph.D., R&D Chemist

Organophosphorus Pesticides Mix

- ✔ High concentration mix.
- ✔ Includes all US EPA Method 8270 organophosphorus pesticide (OPP) target compounds.
- ✔ Enhanced stability—prepared in methanol-free methylene chloride.

This new mix complements our 8270 MegaMix™ (cat.#31686), Appendix IX Mix #1 (cat.#31625), and Appendix IX Mix #2 (cat.#31806), which do not include OPPs.

Organophosphorous Pesticide Mix, 8270/Appendix IX

dimethoate	famphur	sulfotepp
disulfoton	methyl parathion	O,O,O-triethylphosphorothioate
ethyl parathion	phorate	zinophos (thionazine)

2,000µg/mL in methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
32419	32419-510	—
	w/data pack	
32419-500	32419-520	32519

Carbon Number Distribution Marker Standard

For Texas Method 1005 Rev. 03 and Method 1006

- ✔ Includes the minimum required aliphatic markers defining the carbon ranges of interest.
- ✔ Completes set of reference materials for TNRCC methods 1005 and 1006.*

TNRCC Method 1006 is used for determining the total petroleum hydrocarbon (TPH) mass within boiling point ranges of aliphatic fractions (C6-C35).

TNRCC 1006 Retention Time Marker Mix

n-hexane (C6)	*n*-decane (C10)	*n*-heneicosane (C21)
n-heptane (C7)	*n*-dodecane (C12)	*n*-octacosane (C28)
n-octane (C8)	*n*-hexadecane (C16)	*n*-pentatriacontane (C35)

200µg/mL in pentane, 1mL/ampul

Each	5-pk.	10-pk.
32814	32814-510	—
	w/data pack	
32814-500	32814-520	32914

European Pharmacopoeia/ICH Class 1 Mix

- ✔ Concentrations revised to the latest standards for EP Class 1 residual solvents.
- ✔ Dimethylsulfoxide:water solvent for better dispersion during preparation.

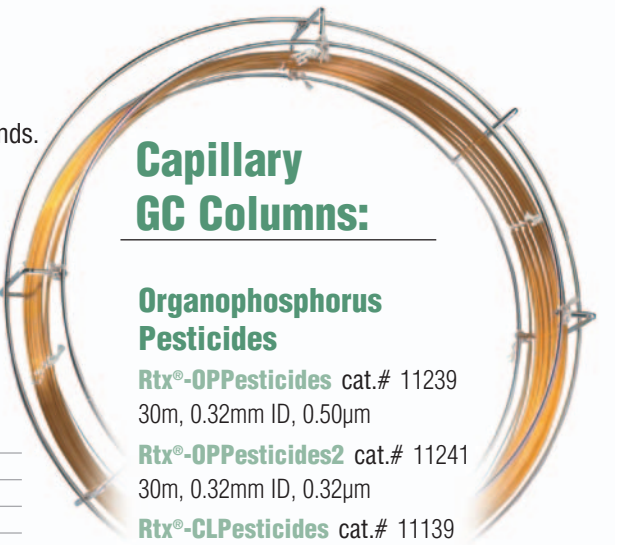
European Pharmacopoeia (EP) monographs now have legal status in 26 member countries. International Conference on Harmonization (ICH) guidelines for residual solvents are an international standard and have been adopted by the United States Pharmacopoeia. The revised concentrations for the components in European Pharmacopoeia/ICH Class 1 Mix meet the latest EP standards. We also offer EP/ICH Class 2 mixes of solvents that pose lesser health hazard (cat.#36229, cat.#36230, cat.#36231). Stabilwax® (cat.#10640) and Rtx®-1301 (G43) (cat.#16085) capillary columns are ideal for analyses of residual solvents.

European Pharmacopoeia/ICH Class 1 Mix (revised)

benzene	2µg/mL	1,1-dichloroethylene	8µg/mL
carbon tetrachloride	4	1,1,1-trichloroethane	10
1,2-dichloroethane	5		

In dimethylsulfoxide:water (90:10), 1mL/ampul

Each	5-pk.	10-pk.
36261	36261-510	—
	w/data pack	
36261-500	36261-520	36361



Capillary GC Columns:

Organophosphorus Pesticides

Rtx®-OPPesticides cat.# 11239
30m, 0.32mm ID, 0.50µm

Rtx®-OPPesticides2 cat.# 11241
30m, 0.32mm ID, 0.32µm

Rtx®-CLPesticides cat.# 11139
30m, 0.32mm ID, 0.50µm

TNRCC 1005 / 1006

Rtx®-5 cat.# 10223
30m, 0.25mm ID, 0.25µm

Rtx®-5 cat.# 10238
30m, 0.25mm ID, 0.50µm

Rtx®-5 cat.# 10253
30m, 0.25mm ID, 1.00µm

Residual Solvents

Rtx®-1301 (G43) cat.# 16085
30m, 0.53mm ID, 3.00µm

Stabilwax® cat.# 10640
30m, 0.53mm ID, 0.50µm

Semivolatiles

Rtx®-5 cat.# 10223
30m, 0.25mm ID, 0.25µm

Rtx®-5Sil MS cat.# 12723
30m, 0.25mm ID, 0.25µm

Rtx®-5MS cat.# 12623
30m, 0.25mm ID, 0.25µm

Volatiles

Rtx®-VMS cat.# 19916
60m, 0.25mm ID, 1.4µm

Rtx®-624 cat.# 10969
60m, 0.25mm ID, 1.40µm

For many more column choices, see our ***2003 chromatography products catalog.*** *If you don't have a copy, just ask!*

* *Additional Restek materials for TNRCC methods 1005 and 1006 include: TPH Locator Mix (cat.#31482), TX TPH Calibration Mix (cat.#31483), TX TPH Matrix Spike Mix (cat.#31484), Alternate Boiling Point/Carbon Number Distribution Marker Stock Standard (cat.#31639).*

Save Preparation and Calibration Time in Skinner List Analyses

With New Restek Reference Mixes

By Ken Herwehe, Analytical Reference Materials Product Marketing Manager

- ✔ Eliminate preparation time:
  - All target volatiles in one mix.
  - All target semivolatiles in one mix.
- ✔ Fast, convenient, single calibrations:
  - *m*- and *p*-xylene at one-half concentration of other volatiles.
  - 3- and 4-methylphenol at one-half concentration of other semivolatiles.
- ✔ Volatiles mix includes methyl *tert*-butyl ether (MTBE).
- ✔ Semivolatiles mix includes optional low concentration polynuclear aromatic hydrocarbons and optional semivolatile organics (indene, dibenzo(a,h)acridine, 1-methylnaphthalene).

In our continual effort to help chromatographers simplify their analyses, we have introduced two new reference mixes for analyses of Skinner List* volatile and semivolatile organic compounds in petroleum refinery waste.

Because *m*-xylene and *p*-xylene coelute, we include each of these isomers at half the concentration of the other compounds in our Skinner List - Volatiles mix. By using this mix, analysts will not have to run an extra calibration to quantify *m*- and *p*-xylene at required limits. The 90% purge & trap methanol and 10% water solvent system we use with this mix protects the keto group in 2-butanone and prevents acetal formation (e.g., in methanol). We include methyl *tert*-butyl ether in the new mix because of its wide use as a gasoline additive.

New Skinner List - SV MegaMix™ reference mix combines the semivolatiles and acid extractables, to greatly shorten preparation time. Because 3-methylphenol and 4-methylphenol coelute, each is included at half the concentration of the other compounds; users of the mix can calibrate at lower levels in order to quantify these compounds at the required limits. The only compound of potential interest excluded from the new mix is benzenethiol (part of the 1985 Skinner List, but an optional constituent in the 1997 revision), because it is unstable and rapidly degrades in methylene chloride. All other compounds, regular and optional, are included.

Detailed information about Skinner List compounds is available from the US EPA. Follow the link:
http://www.epa.gov/req5rcra/r5skin.pdf

**1997 Region 5 Skinner List.*

Skinner List - SV MegaMix™

(33 components, peak list in Figure 1.)

2,000µg/mL each in methylene chloride (3&4 methyl phenol at 1,000µg/mL each), 1mL/ampul

Each	5-pk.	10-pk.
31690	31690-510	—
w/data pack		
31690-500	31690-520	31790

Skinner List - Volatiles

(19 components, peak list in Figure 2.)**

2,000µg/mL each in methanol:water (90:10) (m&p xylene at 1,000µg/mL each), 1mL/ampul

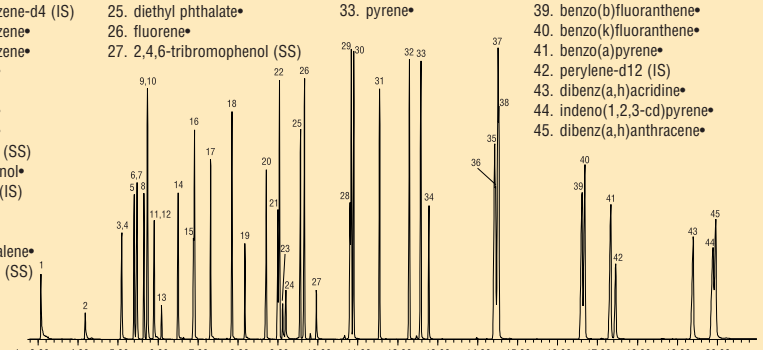
Each	5-pk.	10-pk.
30491	30491-510	—
w/data pack		
30491-500	30491-520	30591

***Benzenethiol, excluded from the mix for stability and an optional compound on the current list, is available by custom order.*

Figure 1 — Calibrate for all Skinner List semivolatiles, including methylphenol isomers, using one Restek reference mix.

Rtx®-5Sil MS 30m, 0.25mm ID, 0.25µm (cat.# 12723)
Sample: Skinner SV MegaMix™ 2,000µg/mL in methylene chloride (cat.# 31690) = MegaMix™ component (•)
B/N Surrogate Mix 1,000µg/mL in methylene chloride (cat.# 31024)
Acid Surrogate Mix 2,000µg/mL in methylene chloride (cat.# 31025)
SV Internal Standard Mix 2,000µg/mL in methylene chloride (cat.# 31206)
Inj.: 1.0µL splitless (hold 0.4 min.), Drilled Uniliner® w/ hole at bottom (cat.# 20756)
Inj. temp.: 275°C
Carrier gas: helium, constant flow
Flow rate: 1mL/min.
Oven temp.: 35°C (hold 1 min.) to 260°C @ 20°C/min. to 300°C @ 6°C/min. (hold 1 min.)
Det: MS
Transfer line temp.: 280°C
Scan range: 35-550amu
Ionization: EI
Mode: scan

1. pyridine•
2. 2-fluorophenol
3. phenol-d6 (SS)
4. phenol•
5. 1,3-dichlorobenzene•
6. 1,4-dichlorobenzene-d4 (IS)
7. 1,4-dichlorobenzene•
8. 1,2-dichlorobenzene•
9. 2-methylphenol•
10. indene•
11. 4-methylphenol•
12. 3-methylphenol•
13. nitrobenzene-d5 (SS)
14. 2,4-dimethylphenol•
15. naphthalene-d8 (IS)
16. naphthalene•
17. quinoline•
18. 1-methylnaphthalene•
19. 2-fluorobiphenyl (SS)
20. dimethylphthalate•
21. acenaphthene-d10 (IS)
22. acenaphthene•
23. 2,4-dinitrophenol•
24. 4-nitrophenol•
25. diethyl phthalate•
26. fluorene•
27. 2,4,6-tribromophenol (SS)
28. phenanthrene-d10 (IS)
29. phenanthrene•
30. anthracene•
31. di-*n*-butylphthalate•
32. fluoranthene•
33. pyrene•
34. *p*-terphenyl-d14 (SS)
35. benz(a)anthracene•
36. chrysene-d12 (IS)
37. chrysene•
38. bis(2-ethylhexyl)phthalate•
39. benzo(b)fluoranthene•
40. benzo(k)fluoranthene•
41. benzo(a)pyrene•
42. perylene-d12 (IS)
43. dibenz(a,h)acridine•
44. indeno(1,2,3-cd)pyrene•
45. dibenz(a,h)anthracene•

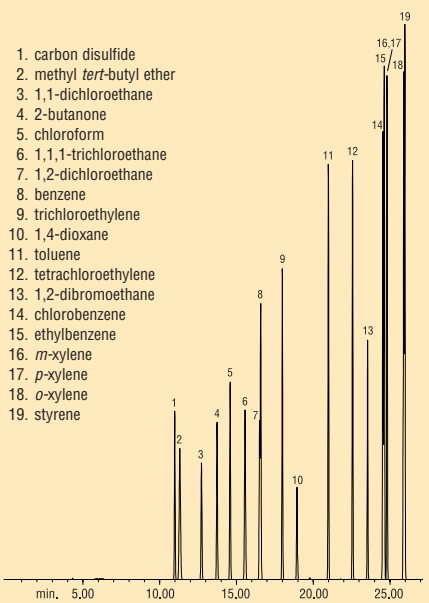


GC_EV00684

Figure 2 — Calibrate for xylene isomers with other Skinner List volatiles, using Restek reference mix.

Rtx®- 502.2, 105m, 0.32 ID, 0.18µm (cat.# 10921)
Sample: Skinner List - Volatiles (cat.# 30491) 2000µg/mL each component, m- & p-xylene at 1000µg/mL
Inj.: 1.0µL split (split ratio 1:20), 4mm split inlet liner with wool (cat.#20782)
Inj. temp.: 200°C
Carrier gas: helium, constant pressure
Column flow: 2.2mL/min.
Oven temp.: 40°C (hold 6 min.) to 240°C @ 6°C/min. (hold 5 min.)
Det.: MSD
Transfer line temp.: 250°C
Scan range: 35–425 amu
Ionization: EI
Mode: TIC

1. carbon disulfide
2. methyl *tert*-butyl ether
3. 1,1-dichloroethane
4. 2-butanone
5. chloroform
6. 1,1,1-trichloroethane
7. 1,2-dichloroethane
8. benzene
9. trichloroethylene
10. 1,4-dioxane
11. toluene
12. tetrachloroethylene
13. 1,2-dibromoethane
14. chlorobenzene
15. ethylbenzene
16. *m*-xylene
17. *p*-xylene
18. *o*-xylene
19. styrene



GC_ST00677

Minimize Adsorption of Active Analytes, Using a Drilled Uniliner® GC Inlet Liner

Now in Two Configurations, to Match Chromatographic Conditions

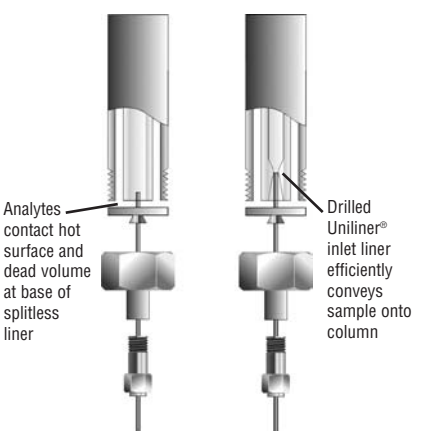
By Gary Stidsen, Innovations Team Manager

- ✔ Eliminate injector active sites and dead volume—minimize adsorption and discrimination.
- ✔ Use one configuration if analytes elute near the solvent peak.
- ✔ Use alternate configuration if analytes elute later than the solvent.

In sample injections into a hot splitless injection port liner, a typical 1µL sample expands to a volume of hundreds of microliters.[1] The sample solvent vapor, and the analytes, fill the entire injector system. Analyte molecules come in contact with hot, active surfaces outside the injection port liner, and occupy the dead volume at the bottom of the injection port, below the inlet end of the column (Figure 1). In splitless injection mode, there is very little carrier gas flow in this area to carry the analytes back up to the column inlet. This situation is most noticeable with active compounds that degrade when they come in contact with active surfaces; recoveries can be significantly reduced. In addition, late-eluting compounds that do not readily vaporize are affected by injection port discrimination.

The innovative geometry of a Drilled Uniliner® inlet liner minimizes active sites in the sample pathway, and reduces injection port discrimination. The analytical column connects to the bottom of a Drilled Uniliner® inlet liner via a Press-Tight® seal (Figure 1), eliminating sample contact with any part of the injector below the column inlet. Recoveries of active analytes are significantly improved.[2] A hole in the side of the liner allows the injector to be operated in traditional split/splitless mode.

Figure 1 — Inlet liner geometry affects analyte recovery.

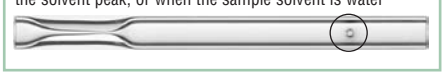


We now offer Drilled Uniliner® inlet liners in two configurations (Figure 2). The liner to use depends on the analysis, and how closely the early-eluting compounds elute to the solvent peak.

Figure 2 — Drilled Uniliner® inlet liners are available in two configurations. The hole allows the injector to be operated in split/splitless mode.

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

Use hole near top configuration if analytes elute later than the solvent peak, or when the sample solvent is water



In flash on-column injection all of the solvent is transferred from the injector to the column, producing a substantial solvent peak tail. Splitless injection eliminates the solvent tail, because the injector goes into the split mode after the compounds of interest are transferred to the column, and all solvent remaining in the injection port is flushed out through the purge vent. The solvent peak ends abruptly, as shown in Figure 3a. Elimination of the solvent peak tail is an advantage to using the splitless injection technique when analyzing compounds that elute close to the solvent.

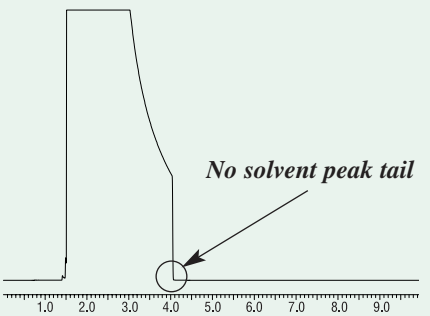
A Drilled Uniliner® inlet liner produces a distinctly different solvent peak shape than the single gooseneck splitless liner, as shown in Figure 3b and Figure 3c. The most noticeable difference is the peak width; the peak is considerably narrower than the peak from the single gooseneck liner. The position of the hole in the Drilled Uniliner® also affects solvent peak shape. A Drilled Uniliner® with the hole near the bottom produces a sharply ending solvent peak, similar to that from a single gooseneck liner (Figure 3b). This liner is a direct replacement for a splitless liner, and should be used when analytes elute closely behind the solvent.

Continued on page 13

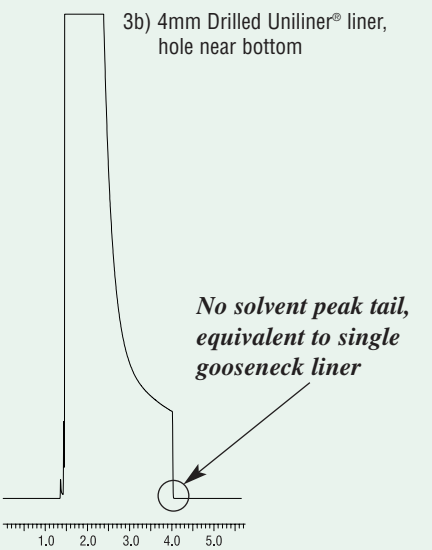
Figure 3 — Solvent peak profiles from Drilled Uniliner® inlet liners and a splitless liner.

The position of the hole in a Drilled Uniliner® inlet liner affects solvent peak shape.

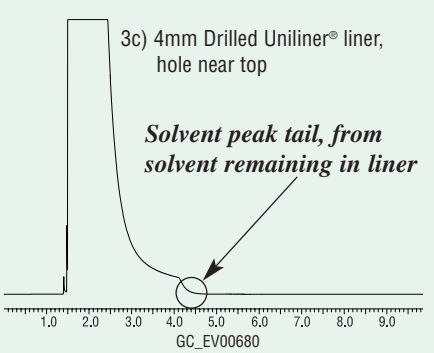
3a) 4mm single gooseneck splitless liner



3b) 4mm Drilled Uniliner® liner, hole near bottom



3c) 4mm Drilled Uniliner® liner, hole near top



Rtx®- 5Sil MS 30m, 0.25 ID, 0.25µm (cat.# 12723)

Sample:	methylene chloride, PR grade
Inj.:	0.5µL, splitless (hold 2.5 min.) 4mm single gooseneck inlet liner (cat.# 20799) 4mm Drilled Uniliner® inlet liner (cat.# 21055) 4mm Drilled Uniliner® inlet liner (cat.# 20756)
Inj. temp.:	260°C
Carrier gas:	helium, constant pressure
Linear velocity:	17cm/sec. @ 50°C
Oven temp.:	50°C, isothermal
Det.:	FID @ 330°C

Minimize Adsorption of Active Analytes...

Continued from page 12

Under the same conditions, a Drilled Uniliner® with the hole near the top produces a solvent peak with a small tail (Figure 3c). This is because solvent remaining within the liner, between the hole and the column entrance, is not swept out of the injection port when the injector goes into the split mode. Consequently, we recommend this liner for analyses in which the analytes would not be affected by a solvent tail, such as chlorinated pesticide analysis. A Drilled Uniliner® with the hole near the top will provide the best sensitivity, and is recommended when sensitivity is paramount. A Drilled Uniliner® with the hole near the top also has exhibited excellent reproducibility for analysis of glycols in water.

For accurate, reproducible, problem-free split/splitless injections, we recommend you use a Drilled Uniliner® inlet liner—and connect it to a Restek capillary GC column.

References

1. *Operating Hints for Using Split/Splitless Injectors* Restek Corporation, Bellefonte, PA, 36pp, 2002. (Reference free on request: cat.# 59880A)
2. *Higher Responses for Chlorinated Pesticides, Using a Drilled Uniliner® GC Inlet Liner and Rtx®-CLPesticides Columns* Restek Corporation, Bellefonte, PA, 4pp, 2003. (Reference free on request: cat.# 59487.)

Drilled Uniliner® Inlet Liners

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

all liners are deactivated

For Agilent 5890 & 6890 GCs (0.25/0.32/0.53mm ID columns)	ID*/OD & Length (mm)	Similar to Agilent	cat.# ea.	cat.# 5-pk.
 Drilled Uniliner® (hole on top)	4.0 ID 6.3 OD x 78.5	G1544-80730	21054	21055
 Siltek™ Drilled Uniliner® (hole on top)	4.0 ID 6.3 OD x 78.5	—	21054-214.1	21055-214.5
 Drilled Uniliner® (hole on bottom)	4.0 ID 6.3 OD x 78.5	G1544-80730	20756	20771
 Double Gooseneck Drilled Uniliner® (hole on top)	4.0 ID 6.3 OD x 78.5	G1544-80700	20508	20509
 Double Gooseneck Drilled Uniliner® (hole on bottom)	4.0 ID 6.3 OD x 78.5	G1544-80700	20954	20989
 Siltek™ 1mm Drilled Uniliner® (hole on top)	1.0 ID 6.3 OD x 78.5	—	21390-214.1	21391-214.5

For PerkinElmer GCs (0.32/0.53mm ID columns)	ID*/OD & Length (mm)	Similar to PE part#	ea.	cat.# 5-pk.
 Auto SYS Drilled Uniliner® (hole on top)	4.0 ID 6.2 OD x 92.1	—	20819	*20822*

**Nominal ID at syringe needle expulsion point.*

Siltek™ Deactivation—The Next Generation of Surface Passivation

- Maximizes the inertness of the sample pathway.
- Minimizes breakdown.
- Low bleed.
- Thermally stable.
- "Clean and green"—manufactured without the use of harmful organic solvents.

Restek offers the next generation of deactivation. The Siltek™ deactivation process (patent pending) produces a highly inert glass surface, which features high temperature stability, extreme durability, and low bleed. Try Siltek™ liners, guard columns, wool, and connectors for better recovery of sample analytes.

For Siltek™ inlet liners, add the corresponding suffix number to the liner catalog number.

qty.	Siltek™		Siltek™ with Siltek™ wool		Siltek™ with CarboFrit™	
each	-214.1	addl. cost	-213.1	addl. cost	-216.1	addl. cost
5-pk.	-214.5	addl. cost	-213.5	addl. cost	-216.5	addl. cost
25-pk.	-214.25	addl. cost	-213.25	addl. cost	-216.25	addl. cost

New / Recent Literature

- ✔ Analysis of Volatile Organics - technical guide (lit. cat.# 59887A)
- ✔ Brominated Flame Retardants - applications note (lit. cat.# 59389B)
- ✔ Environmental Gas Standards - Fast Facts (lit. cat.# 59276A)
- ✔ EZ No-Vent™ GC/MS Connector - new product flyer (lit. cat.# 59498)
- ✔ Foods Flavors & Fragrances In-Review - abstracts of Restek publications (lit. cat.# 59489)
- ✔ High-Resolution Analyses of FAMEs - applications note (lit. cat.# 59584A)
- ✔ HPLC minicatalog (lit. cat.# 59241A)
- ✔ Inlet Supplies - a handy guide to septa, liners, etc. (lit. cat.# 59893B)
- ✔ Pesticides (PCBs) / GC Racer - applications note (lit. cat.# 59457)
- ✔ Pinnacle™ DB HPLC Columns - new product flyer (lit. cat.# 59499)
- ✔ Sample Cylinder Technology - product flyer (lit. cat.# 59618A)
- ✔ STAR Service Rewards - new program: earn credit toward instrument service (lit. cat.# 59522)
- ✔ Vu2 Union™ Column Connector - new product flyer (lit. cat.# 59505)

HOT tech tip

Drilled Uniliner®

Use a Drilled Uniliner® inlet liner with the hole near the bottom if compounds of interest will be affected by a tailing solvent peak. Use a Drilled Uniliner® inlet liner with the hole near the top when compounds of interest elute away from the solvent peak, when sensitivity is critical, or when the sample solvent is water.

Analysis of Complex Semivolatiles Samples

Quantify 126 Semivolatile Compounds, Using an Rtx®-5Sil MS Capillary GC Column

By Gary Stidsen, Innovations Team Manager and Katia May, Ph.D., R&D Chemist

- ✔ Full complement of 126 EPA 8270 semivolatile/Appendix IX compounds in 3 mixes
- ✔ New 8270/Appendix IX Kit includes all 3 mixes
- ✔ New 32 component Appendix IX Mix

Appendix IX is a list of organic and inorganic Hazardous Constituents monitored in groundwater during compliance monitoring and corrective actions at RCRA-regulated hazardous waste treatment, storage, and disposal facilities. The organics usually are evaluated by following US EPA Method 8260 (volatiles), Method 8270 (semivolatiles), or Method 8080 (organochlorine pesticides).

Restek chemists determined the most commonly analyzed Appendix IX compounds, carefully reviewed the latest version of EPA Method 8270, and designed a new reference mix of 32 compounds—Appendix IX Mix #2 —to meet current needs of environmental laboratories.

We formulated the new mix with the goal of preparing a product that is stable as well as useful. Appendix IX constituents include many classes of chemicals: polynuclear aromatic hydrocarbons (PAHs), phenols, chlorinated aromatic hydrocarbons, aldehydes, anilines, benzidines, insecticides. Unstable combinations of these compounds will produce chemical interactions—and flawed calibration data. Methylene chloride is a common solvent for semivolatile organics, but some grades of methylene chloride contain low concentrations of methanol as a stabilizer. Aldehydes (e.g., benzaldehyde) and chlorinated triazines (e.g., atrazine) can react with methanol; chlorinated triazines also react with water. Consequently, we use affirmed methanol-free, water-free methylene chloride in formulating our mixes. We package the new mix in deactivated amber glass ampuls, to prevent reactions catalyzed by light.

Appendix IX Mix #2 is a highly useful complement to our 8270 MegaMix™ (76 compounds, cat.# 31686) and Appendix IX Mix #1 (18 compounds, cat.# 31625). The 126 semivolatiles in these three mixes are separated on an Rtx®-5Sil MS column (cat.# 12723) (Figure 1, page 15). Because the PAHs elute at temperatures over 300ºC, the analysis requires a column that will not bleed at high temperatures. Further, an inert column is important for the active compounds (e.g., pentachlorophenol, dinitrophenols). Rtx®-5Sil MS columns are widely used for analyzing various classes of semivolatiles, because they exhibit versatility, inertness, and low bleed at high temperature. The optimal combination of internal diameter and film thickness of a 30m, 0.25 mm ID, 0.25µm Rtx®-5Sil MS column make this the best choice for analyzing complex mixtures of semivolatiles. The Rtx®-5Sil MS column, in combination with our 8270 MegaMix™ mix, Appendix IX Mix #1, and new Appendix IX Mix #2, and our internal standards and surrogates for EPA Method 8270 and Appendix IX, make Restek the only place you need to look when you want columns and reference materials for semivolatiles analysis.

new!

Appendix IX Mix #2 (32 components)

acetophenone	hexachloropropene
Aramite (2 isomers)	isodrin
atrazine	isosafrole (*cis & trans*)
benzaldehyde	kepone
biphenyl	methyl methanesulfonate
caprolactam (epsilon)	3-methylcholanthrene
chlorobenzilate	1,4-naphthoquinone
1-chloronaphthalene	4-nitroquinoline-N-oxide
diallate	pentachlorobenzene
dibenz(a,h)acridine	pentachloroethane
2,6-dichlorophenol	pentachloronitrobenzene
7,12-dimethylbenz(a)anthracene	phenacetin
1,4-dioxane	pronamide
diphenyl ether	safrole
ethyl methacrylate	1,2,4,5-tetrachlorobenzene
ethyl methanesulfonate	1,3,5-trinitrobenzene

1,000µg/mL each in methylene chloride, 1mL ampul

Each	5-pk.	10-pk.
31806	31806-510	—
	w/data pack	
31806-500	31806-520	31906

8270 MegaMix™ (76 components)

acenaphthene	2,4-dinitrophenol
acenaphthylene	2,4-dinitrotoluene
aniline	2,6-dinitrotoluene
anthracene	di-*n*-butyl phthalate
azobenzene[1]	di-*n*-octyl phthalate
benzo(a)anthracene	diphenylamine[2]
benzo(a)pyrene	fluorene
benzo(b)fluoroanthene	fluoroanthene
benzo(ghi)perylene	hexachlorobenzene
benzo(k)fluoroanthene	hexachlorobutadiene
benzyl alcohol	hexachlorocyclopentadiene
benzyl butyl phthalate	hexachloroethane
bis 2-ethylhexyl adipate	indeno(1,2,3-cd)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
dibenz(a,h)anthracene	4-nitrophenol
dibenzofuran	N-nitrosodimethylamine
1,2-dichlorobenzene	N-nitroso-di-*n*-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:benzene (75:25), 1mL/ampul

Each	5-pk.	10-pk.
31686	31686-510	—
	w/data pack	
31686-500	31686-520	31786

**Concentration is 500µg/mL.*

[1]1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).

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

Ordering Information | Rtx®-5Sil MS column

30m,	0.25 mm ID,	0.25µm	cat.# 12723

Appendix IX Mix #1 (18 components)

2-acetylaminofluorene
4-aminobiphenyl
p-dimethylaminoazobenzene
3,3'-dimethylbenzidine
α,α,-dimethylphenethylamine (free base)
methapyrilene (free base)
1-naphthylamine
2-naphthylamine
5-nitro-*o*-toluidine
N-nitrosodibutylamine
N-nitrosodiethylamine
N-nitrosomethylethylamine
N-nitrosomorpholine
N-nitrosopiperidine
N-nitrosopyrrolidine
1,4-phenylenediamine
2-picoline
o-toluidine

2,000µg/mL each in methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
31625	31625-510	—
	w/data pack	
31625-500	31625-520	31725

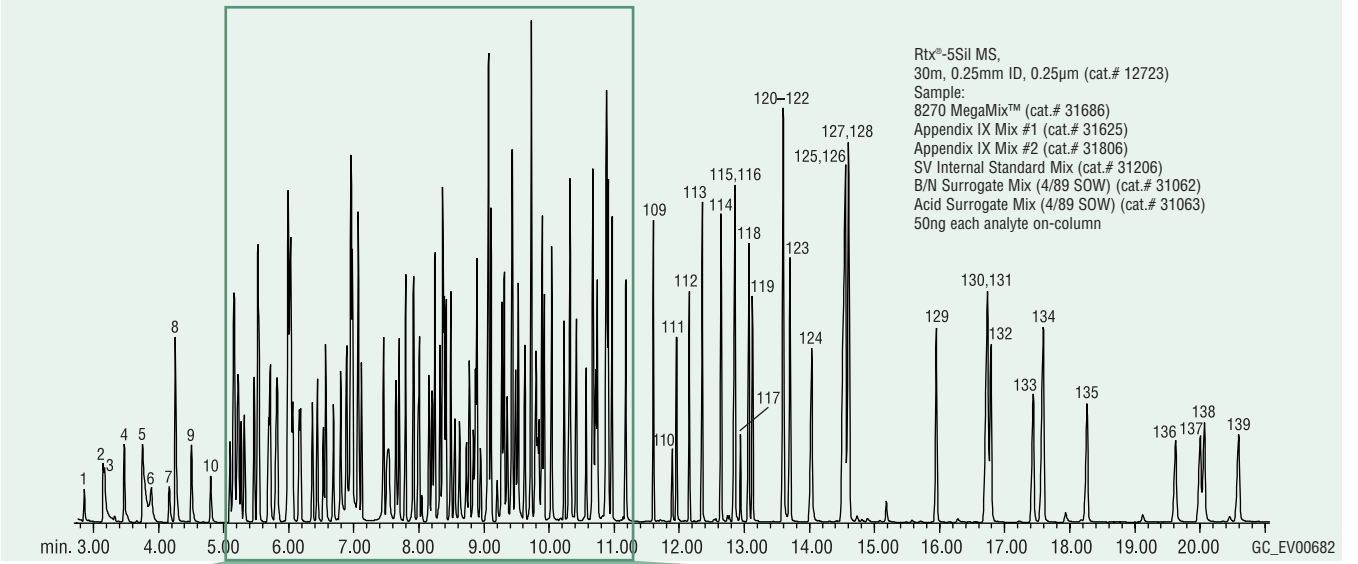
8270/Appendix IX Kit

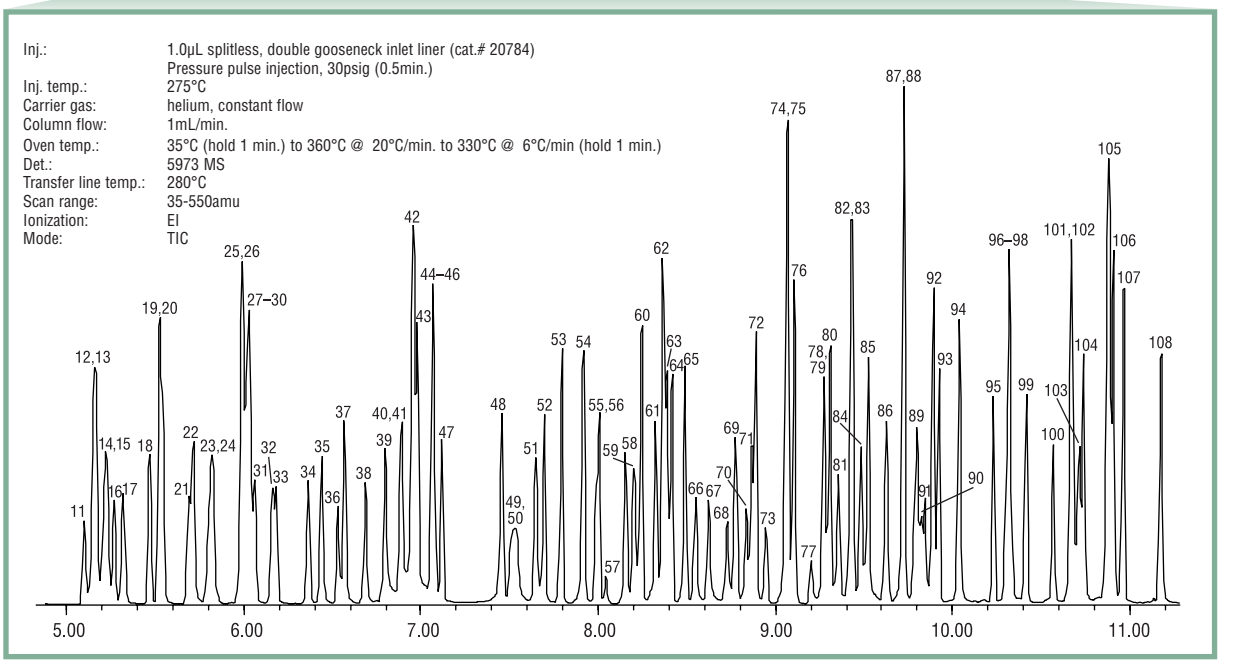
31686: 8270 MegaMix™
31030: Benzidine Mix, EPA 605
31625: Appendix IX Mix #1
31806: Appendix IX Mix #2

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31815	31815-500

Figure 1 — Resolve complex mixtures of semivolatiles with an inert, low-bleed column.





1. 1,4-dioxane
2. pyridine
3. N-nitrosodimethylamine
4. ethyl methacrylate
5. 2-picoline
6. N-nitrosomethylethylamine
7. methyl methanesulfonate
8. 2-fluorophenol (SS)
9. N-nitrosodiethylamine
10. ethyl methanesulfonate
11. benzaldehyde
12. phenol-d6 (SS)
13. phenol
14. aniline
15. pentachloroethane
16. bis(2-chloroethyl)ether
17. 2-chlorophenol
18. 1,3-dichlorobenzene
19. 1,4-dichlorobenzene-d4 (IS)
20. 1,4-dichlorobenzene
21. benzyl alcohol
22. 1,2-dichlorobenzene
23. 2-methylphenol
24. bis(2-chloroisopropyl)ether
25. acetophenone
26. 4-methylphenol/3-methylphenol
27. N-nitroso-di-*n*-propylamine
28. nitrosopyrrolydine
29. *o*-toluidine
30. 4-nitrosomorpholine
31. hexachloroethane
32. nitrobenzene-d5 (SS)
33. nitrobenzene
34. N-nitrosopiperidine
35. isophorone
36. 2-nitrophenol
37. 2,4-dimethylphenol
38. bis(2-chloroethoxy)methane
39. 2,4-dichlorophenol
40. 1,2,4-trichlorobenzene
41. α,α-dimethylphenylamine
42. naphthalene-d8 (IS)
43. naphthalene
44. 2,6-dichlorophenol
45. 4-chloroaniline
46. hexachloropropene
47. hexachlorobutadiene
48. N-nitroso-*n*-butylamine
49. 1,4-phenylenediamine
50. caprolactam
51. 4-chloro-3-methylphenol
52. isosafrole (isomer)
53. 2-methylnaphthalene
54. 1-methylnaphthalene
55. hexachlorocyclopentadiene
56. 1,2,4,5-tetrachlorobenzene
57. isosafrole (isomer)
58. 2,4,6-trichlorophenol
59. 2,4,5-trichlorophenol
60. 2-fluorobiphenyl (SS)
61. safrole
62. biphenyl
63. 2-chloronaphthalene
64. 1-chloronaphthalene
65. diphenyl ether
66. 2-nitroaniline
67. 1,4-naphthoquinone
68. 1,4-dinitrobenzene
69. dimethylphthalate
70. 1,3-dinitrobenzene
71. 2,6-dinitrotoluene
72. acenaphthylene
73. 1,2-dinitrobenzene
74. 3-nitroaniline
75. acenaphthene-d10 (IS)
76. acenaphthene
77. 2,4-dinitrophenol
78. pentachlorobenzene
79. 4-nitrophenol
80. dibenzofuran
81. 2,4-dinitrotoluene
82. 1-naphthalamine
83. 2,3,4,6-tetrachlorophenol
84. 2,3,5,6-tetrachlorophenol
85. 2-naphthylamine
86. diethyl phthalate
87. fluorene
88. 4-chlorophenyl phenyl ether
89. 2-methyl-5-nitroaniline
90. 4-nitroaniline
91. 4,6-dinitro-2-methylphenol
92. diphenylamine
93. azobenzene
94. 2,4,6-tribromophenol (SS)
95. diallate
96. 1,3,5-trinitrobenzene
97. phenacetin
98. 4-bromophenyl phenyl ether
99. hexachlorobenzene
100. atrazine
101. 4-aminobiphenyl
102. pentachlorophenol
103. pentachloronitrobenzene
104. propyzamide
105. phenanthrene-d10 (IS)
106. phenanthrene
107. anthracene
108. carbazole
109. di-*n*-butylphthalate
110. 4-nitroquinoline-1-oxide
111. methapyriline
112. isodrin
113. fluoranthene
114. pyrene
115. Aramite (isomer)
116. *p*-terphenyl-d14 (SS)
117. Aramite (isomer)
118. dimethylaminoazobenzene
119. dichlorobenzilate
120. 3,3-dimethylbenzidine
121. butyl benzyl phthalate
122. kepone
123. bis(2-ethylhexyl)adipate
124. 2-acetylaminofluorene
125. benz(a)anthracene
126. chrysene-d12 (IS)
127. chrysene
128. bis(2-ethylhexyl)phthalate
129. di-*n*-octyl phthalate
130. benzo(b)fluoranthene
131. 7,12-dimethylbenz(a)anthracene
132. benzo(k)fluoranthene
133. benzo(a)pyrene
134. perylene-d12 (IS)
135. 3-methylcholanthrene
136. dibenz(a,j)acridine
137. indeno(1,2,3-cd)pyrene
138. dibenz(a,h)anthracene
139. benzo(ghi)perylene

Peak Performers

all liners are deactivated

By Donna Lidgett, GC Accessories Marketing Manager, and Brad Rightnour, Instrument Innovations Manager

Inlet liners for Varian 1177 GCs

COLUMN INSTALLS THIS END

	Benefits/ Uses:	ID*/OD & Length (mm)	Similar to Varian part #	ea.	cat.# 5-pk.
4mm Split	universal	4.0 ID 6.3 OD x 78.5	39-26119-36	21045	21046
4mm Split w/ FS Wool	universal	4.0 ID 6.3 OD x 78.5	39-26119-37	—	21079
4mm Split Precision™ Liner w/FS Wool new!	dirty samples, trace samples	4.0 ID 6.3 OD x 78.5	—	20759	20762
Laminar Cup Splitter new!	high MW compounds	4.0 ID 6.3 OD x 78.5	—	20765	20768
2mm Splitless w/ FS Wool	trace samples <2µL	2.0 ID 6.5 OD x 78.5	39-26119-38	21045	21077
Gooseneck Splitless (4mm) new!	trace samples <2µL	4.0 ID 6.5 OD x 78.5	39-26119-27	21896	21897
Siltek™ Gooseneck Splitless (4mm) new!	trace samples <2µL	4.0 ID 6.5 OD x 78.5	—	21896-214.1	21897-214.5
Gooseneck Splitless (4mm) w/ FS Wool new!	trace samples <2µL	4.0 ID 6.5 OD x 78.5	39-26119-36	21896-200.1	21897-200.5
Siltek™ Gooseneck Splitless (4mm) w/ Siltek™ Glass Wool new!	trace samples <2µL	4.0 ID 6.5 OD x 78.5	—	21896-213.1	21897-213.5
Double Gooseneck Splitless (4mm) new!	trace, active samples <2µL	4.0 ID 6.5 OD x 78.5	—	21891	21892

Graphite O-Rings for Varian 1177 and Agilent GCs

- Excellent thermal stability at injection port temperature up to 450°C!

Description	Max. temp.	Similar to Agilent part #	Restek cat.# 10-pk.	50-pk.
6.35mm ID Graphite O-rings for split liners	450°C	5180-4168	20296	20297
6.5mm ID Graphite O-rings for splitless liners	450°C	5180-4173	20298	20299

Precision™ Inlet Liners for Many GCs

- Wool maximizes vaporization and helps wipe the needle during injection.
- No guessing where the wool should be placed, easy to change.
- Wool stays in position during pressure pulses in the inlet and during injection.
- 100% polymeric deactivation ensures inertness.
- Similar to FocusLiner™ liners.

COLUMN INSTALLS THIS END

	Benefits/Uses:	ID*/OD & Length (mm)	ea.	cat.# 5-pk.	25-pk.
Agilent 4mm Split Precision™ Liner	dirty samples, trace samples	4.0 ID 6.3 OD x 78.5	21022	21023	20979
PerkinElmer Auto SYS Split Precision™ Liner	dirty samples, trace samples	4.0 ID 6.2 OD x 92.1	21026	21027	—
Shimadzu 17A Split Precision™ Liner	dirty samples, trace samples	3.5 ID 5.0 OD x 95	21020	21021	—
Thermo Finnigan 5mm Split Precision™ Liner	dirty samples, trace samples	5.0 ID 8.0 OD x 105	21028	21029	—
Varian 1075/1077 Split Precision™ Liner	dirty samples, active samples	4.0 ID 6.3 OD x 72	21030	21031	—
Varian 1078/1079 Split Precision™ Liner	dirty samples, trace samples	3.4 ID 5.0 OD x 54	21024	21025	—

Viton® O-Rings For Agilent and PE AutoSys

- Fit split (6.3mm OD) or splitless (6.5mm OD) liners.
- Maximum temperature 350°.

Description	Similar to Agilent part #	qty.	cat.#
Viton® O-rings for Agilent and PE	5180-4182	25-pk.	20377

new!

O-Rings for Apex Liners

Description	qty.	cat.#
Viton® O-rings for APEX liners	25-pk.	22067

new!

Inlet Liner Seals for TRACE™ 2000 GCs: PTV

Description	qty.	cat.#
Inlet Liner Seals	2-pk.	21392

Graphite Sealing Ring and Washer for 8000 and TRACE™ Series GC Inlet Liners

(Similar to Thermo Finnigan part # 290-03406)

Description	qty.	cat.#
Graphite Sealing Ring and Washer	ea.	21898
Graphite Sealing Rings and Washers	2-pk.	21899

new!

Liner Seals for CIS4 and PTV

Description	qty.	cat.#
Liner Seals for CIS4 and PTV	5-pk.	22684

new!

Liner Seals for Varian 1078/1079

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

Replacement Nickel Reaction Tubes

- Pretreated for maximum sensitivity.
- Quality-controlled for reliability.
- Available for various models.

ELCD Model #	To replace these instrument part numbers: Tremetrics	Varian	PerkinElmer	Shimadzu	O.I. Analytical	Order these Restek part numbers: qty.	cat.#
Hall 700A	115439-0003	00-996724-14	0330-2675	—	—	2-pk.	21580
Hall 1000	117459-0003	00-997625-12	N660-1072	220-90435-00	—	2-pk.	21581
O.I. 4420	—	—	—	—	183780	2-pk.	21582

Moisture Control By-Pass Line for Tekmar Instruments

- Increase response for ketones, alcohols, and acetates.
- Silcosteel® tubing for increased inertness.
- Suitable for US EPA Methods 8260, 524.2, and OLM4.1.
- Easily attaches in minutes.

Description	qty.	cat.#
Moisture Control By-Pass Line for Tekmar 3000 Purge & Trap	ea.	21035
Moisture Control By-Pass Line for Tekmar 3100 Purge & Trap	ea.	21109

New!

Replacement FID Jets

Capillary Adaptable Jet for Agilent 5890/6890/6850 GCs (0.011-inch ID tip)

(Similar to Agilent part # 19244-80560.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	20670	3-pk.	20671
High-Performance Silcosteel®	ea.	20672	3-pk.	20673

Capillary Dedicated Jet for Agilent 6890/6850 GCs

(Similar to Agilent part # G1531-80560.)

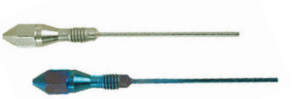
Description	qty.	cat.#	qty.	cat.#
Standard	ea.	21621	3-pk.	21682
High-Performance Silcosteel®	ea.	21620	3-pk.	21683

Capillary Jet for Agilent 5880 GCs

(Similar to Agilent part # 19301-80500.)

Description	qty.	cat.#
Standard	ea.	21637
High-Performance Silcosteel®	ea.	21638

Packed Column Jets for Agilent 5890/6890/6850 GCs



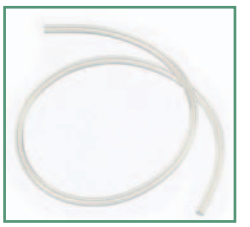
0.018-Inch ID

(Similar to Agilent part # 18710-20119.)	qty.	cat.#	qty.	cat.#
Standard	ea.	21694	3-pk.	21695
High-Performance Silcosteel®	ea.	21696	3-pk.	21697

0.030-Inch ID

(Similar to Agilent part # 18789-80070.)	qty.	cat.#	qty.	cat.#
Standard	ea.	21688	3-pk.	21689
High-Performance Silcosteel®	ea.	21686	3-pk.	21687

Cleaned ELCD Transfer Lines

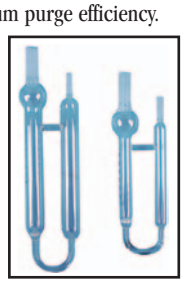


Restek's ELCD Teflon® transfer line tubing is stringently cleaned with an HCl solution to remove any contaminants, then is rinsed with methanol. Conveniently offered in five 6.5-inch precut pieces that directly interface the nickel reaction tube and conductivity cell. Fits Tracor, Tremetrics, O.I., and many other ELCDs.

Description	qty.	cat.#
Teflon® Transfer Lines for ELCDs (five 6.5-inch lines)	5-pk.	20121

Fritted Purge-and-Trap Spargers

- For Tekmar 2000, 3000 and 3100.
- Available in 5 and 25mL sizes.
- Uniform frits ensure maximum purge efficiency.



These spargers provide maximum purge efficiency for water samples.* Manufactured to tight tolerances to ensure a leak-tight seal.

**Not recommended for wastewater samples—frit may become plugged.*

Description	qty.	cat.#
5mL Fritted Sparger, ½-inch mount	ea.	21150
25mL Fritted Sparger, ½-inch mount	ea.	21151

Optimizing the Analysis of Volatile Organic Compounds

Our newly updated technical guide is a concise, thorough overview to analyzing volatile organics in environmental samples:

- ✔ Purge and trap theory - adsorbents and traps - troubleshooting
- ✔ GC system configurations for narrow-bore or wide-bore capillary columns
- ✔ Optimizing detection systems

Many chromatograms show how changes in chromatographic parameters achieve specific goals. Invaluable information for new chromatographers; experienced analysts can find ideas and justification for updating methodology. 72 pages.

Free on request - ask for lit. cat.# 59887A, or visit our website.

Sulfinert™-Treated Sample Cylinders and Valves

Durable, Inert Storage for Active Analytes

By Gary Barone, Metals Passivation Marketing Manager, and Donna Lidgett, Air Products Marketing Manager

- ✔ Inert sample storage containers now available from stock.
- ✔ No adsorption of active compounds at low ppb concentrations.
- ✔ Stable, flexible surface deactivation will not crack or flake.

Whether you monitor hydrocarbons in refinery streams or reactive compounds in chemical reaction vessels, our Sulfinert™-treated sample cylinders and cylinder valves will help you achieve maximum accuracy in your results. These inert containers ensure the stability of sulfur compounds and other active analytes during storage and transport from the field to the laboratory.

Sulfinert™-treated sample cylinders combine the inertness of glass with the strength of stainless steel, making them ideal for analyses of active compounds. Restek's exclusive Sulfinert™ process creates a thin, silica-like layer on the stainless steel surface. Even at trace levels, sulfur compounds and other active molecules can be collected and stored in these cylinders with no significant loss. Effectively incorporated into the steel surface, the Sulfinert™ layer cannot chip, flake, or rinse off - even under stringent sampling, transport, or cleaning conditions.

All sample-contacting surfaces in our Sulfinert™-treated sample cylinder valves are equally inert, making these components ideally compatible with Sulfinert™-treated sample cylinders.

Sulfinert™-treated gas sampling apparatus is ideal for applications that demand only inert surfaces contact the sample, such as sampling natural gas streams or testing beverage-grade carbon dioxide. For other Sulfinert™-treated sample pathway components, refer to our current general catalog, or contact our Technical Service group or your Restek representative.

Sulfinert™-Treated Sample Cylinders

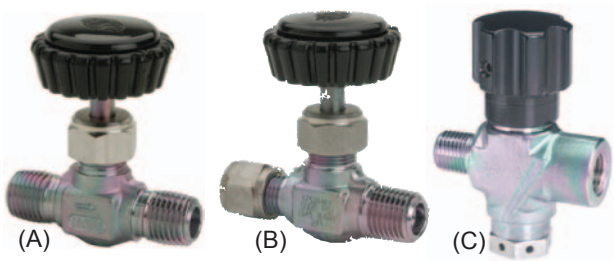
- Sizes from 75cc to 2250cc
- Durable 316 stainless steel
- ¼" female NPT threads on both ends
- D.O.T. rated to 1800psi at room temperature

Sulfinert™-Treated Sample Cylinders

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

Sulfinert™-Treated Sample Cylinder Valves

- Maximum operating pressure: 5000psig
- Temperature range for KEL-F® stem tip: -20ºF to 250ºF (-29ºC to 121ºC)



Sulfinert™ Sample Cylinder Valves

Description	qty.	cat.#
(A) ¼" NPT Exit, KEL-F® Stem Tip	ea.	24127
(B) ¼" Compression Exit, KEL-F® Stem Tip	ea.	24128
(C) ¼" Female NPT Outlet (built-in rupture disc)	ea.	21395

Sulfinert™ Welded 304 Grade Stainless Steel Tubing

ID	OD	cat.#	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
0.011" (0.28mm)	0.022" (0.56mm)	22500				
0.021" (0.53mm)	0.029" (0.74mm)	22501				
0.010" (0.25mm)	1/16" (1.59mm)	22502				
0.020" (0.51mm)	1/16" (1.59mm)	22503				
0.030" (0.76mm)	1/16" (1.59mm)	22504				
0.040" (1.02mm)	1/16" (1.59mm)	22505				
0.085" (2.16mm)	1/8" (3.18mm)*	22506				
0.210" (5.33mm)	1/4" (6.35mm)*	22507				

**0.020" wall thickness*

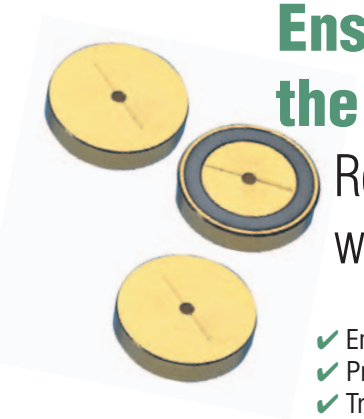
Sulfinert™ Seamless 316 Grade Stainless Steel Tubing

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				

***0.035" wall thickness*

Please note: A charge is applied for cutting Sulfinert™ tubing.
The charge is calculated from the total number of pieces produced from cutting, for each line item.

For Sulfinert™-treated fittings, refer to our general catalog.



Ensure a Leak-Tight Seal the First Time, Every Time

Reliable, Leak-Tight Sealing with Vespel® Ring Inlet Seals•

By Donna Lidgett, GC Accessories Marketing Manager

- ✔ End problems associated with leaks at the inlet seal.
- ✔ Prevent wear or damage to the critical seal.
- ✔ Try Vespel® Ring Inlet Seals, free.

In Agilent split/splitless injection ports, the inlet seal sits at the base of the injector (Figure 1). Dirt, nonvolatile residue, septum fragments, and other debris can accumulate on the surface of the seal and skew the linearity of your analyses. The only way to maintain optimum performance is to change the inlet seal frequently. Each time you change the inlet seal you must check the connection and ensure the seal is leak-tight, to prevent column oxidation and other problems associated with leaks.

Herein is a problem. It can be difficult to make and maintain a good seal with a conventional, metal, inlet disk. The metal-to-metal seal dictates that you apply considerable torque to the reducing nut but, based on our tests, this does not ensure a leak-tight seal. And, even if the seal is good initially, metal-to-metal seals are prone to leak over the course of several oven temperature cycles, allowing oxygen and water vapor to enter the system. This can oxidize the phase in the capillary column, affect chromatogram baselines, and/or create other difficulties, causing expense and downtime. Potentially a worse problem, frequent making and breaking of the metal-to-metal seal ultimately could wear or damage the surface of the critical seal on the bottom of the injector body.

For years, Restek has offered replacement inlet seals for Agilent 5890, 6890, and 6850 split/splitless injection ports. The design and construction of our inlet seals prolong column lifetime - because oxygen is less likely to leak into the carrier gas - and reduce baseline noise from high-sensitivity detectors (e.g., ECDs and MSDs). To lessen breakdown and adsorption of active compounds, we offer inert gold-plated and Silcosteel®-treated inlet seals, as well as stainless steel seals.

Yet, we had not entirely eliminated the problem of leaks. Now, Restek's instrument innovations team has re-engineered the inlet seal to address this issue. The solution, the Vespel® Ring Inlet Seal, is a high-quality stainless steel seal with a soft, easy-sealing Vespel® ring embedded into its face, eliminating the unreliable metal-to-metal seal. While ensuring a leak-tight seal, the Vespel® ring cannot harm the critical seal, and is outside the sample flowpath. Inlet maintenance becomes worry-free!

The Vespel® Ring Inlet Seal is designed to hold its seal, even after repeated temperature cycles, without retightening the reducing nut. To determine the differences between conventional inlet seals and Vespel® Ring Inlet Seals, we compared the leak rate for several inlet seals of each type across a range of torque, using a high sensitivity helium leak detector. Figure 2 shows Vespel® Ring Inlet Seals performed exceptionally at all levels, but especially well at lower torque.

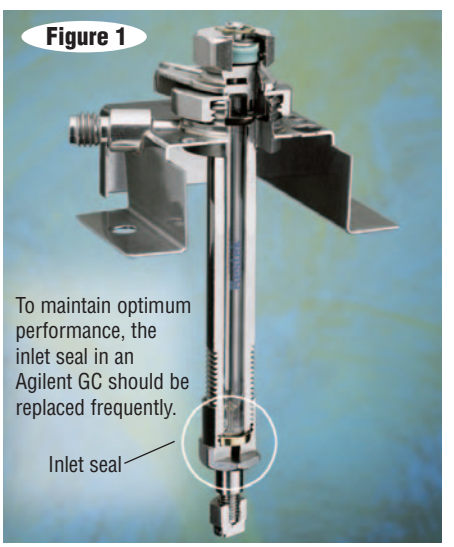
Like our conventional inlet seals, Vespel® Ring Inlet Seals are available in stainless steel, gold-plated, or with Silcosteel® treatment. Use the stainless steel seals for analyses of nonactive compounds. To reduce breakdown and adsorption of active compounds, use gold-plated or Silcosteel®-treated seals. The gold surface offers better inertness than stainless steel; Silcosteel® treatment produces inertness similar to that of fused silica tubing.

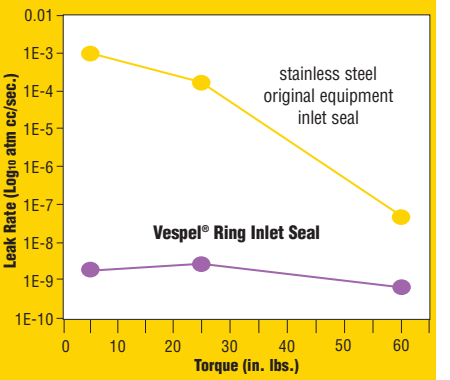
Free Vespel® Ring Inlet Seals!

Are you using conventional inlet seals, but want to try Vespel® Ring Inlet Seals? Purchase a 10-pack of our conventional inlet seals and we'll include a 2-pack of Vespel® Ring Inlet Seals, free. Use the special catalog numbers below.

Hurry-

***June 30* is the last day to take advantage of this offer!**





•Patent pending.

Ordering Information | Inlet Seals Special Offer!

Order a 10-pack of our conventional metal inlet seals, receive a 2-pack of Vespel® Ring Inlet Seals, Free!

Inlet Seal	Qty.	cat.#	Price
0.8mm stainless steel	10 + 2*	21316-425	
0.8mm gold-plated	10 + 2	21318-425	
0.8mm Silcosteel®-treated	10 + 2	21320-425	
1.2mm stainless steel	10 + 2	20391-425	
1.2mm gold-plated	10 + 2	21306-425	
1.2mm Silcosteel®	10 + 2	21308-425	

*10 conventional metal inlet seals, 2 Vespel® Ring Inlet Seals.

Vespel® Ring Inlet Seals

- Low torque, soft seal, reduces wear, prevents damage at critical seal: no unexpected downtime
- Leak-tight connections, for greater sensitivity (less detector noise) and longer column life (no phase oxidation)

FREE
Vespel® Ring Inlet Seals.
See Page 19.

Vespel® Ring Inlet Seals are available in three finishes: gold-plated, Silcosteel®-treated, or stainless steel.

Vespel® Ring Inlet Seals with Washers for Agilent 5890/6890/6850 GCs

0.8mm ID Vespel® Ring Inlet Seal	2-pk.	10-pk.
Gold-Plated	21562	21563
Silcosteel®	21564	21565
Stainless Steel	21560	21561
1.2mm ID Vespel® Ring Inlet Seal	**2-pk.**	**10-pk.**
Gold-Plated	21568	21569
Silcosteel®	21570	21571
Stainless Steel	21566	21567

Note: All seals include washers.

Replacement Inlet Seals for Agilent GCs

- Special grade of stainless steel that is softer and deforms more easily, ensuring a leak-free seal.
- Increases column lifetime because oxygen cannot permeate into the carrier gas.
- Reduced noise benefits high-sensitivity detectors (e.g., ECDs, MSDs).
- Silcosteel® seal offers the inertness of glass.
- All seals include washers.

Single-Column Installation, 0.8mm Opening*		0.25/0.32mm ID Dual-Column Installation, 1.2mm Opening		0.53mm ID Dual-Column Installation, 1/16-inch Opening	
2-pk.	**10-pk.**	**2-pk.**	**10-pk.**	**2-pk.**	**10-pk.**
		Stainless Steel Inlet Seal			
21315	21316	20390	20391	20392	20393
		Gold-Plated Inlet Seal			
21317	21318	21305	21306	—	—
		Silcosteel® Inlet Seal			
21319	21320	21307	21308	—	—

Restek inlet seals feature a ***super-smooth surface*** *for better chromatography!*

**0.8mm ID stainless steel inlet seal is equivalent to Agilent part #18740-20880, 0.8mm ID gold-plated inlet seal is equivalent to Agilent part #18740-20885.*
Note: The 1.2mm inlet seal is recommended for use with Vespel®/graphite ferrules or when installing two columns using a two-hole ferrule. The 0.8mm inlet seal is recommended for use with graphite ferrules and single capillary column installation.

Replacement Inlet Seal Washers

Description	Similar to Agilent part #	qty.	cat.#
Replacement Inlet Seal Washers	5061-5869	15-pk.	21710

ISO 9001 REGISTERED

Lit. Cat. # 59523-INT

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Please direct your comments on this publication to Brett Tyson, Graphic Designer, at btyson@restekcorp.com or call 814-353-1300, ext. 2113.

Resolve Complex Mixtures of Organophosphorus Pesticides,

Using an Rtx®-CLPesticides GC Column and a New Restek Reference Mix

By Katia May, Ph.D., R&D Chemist, and Lydia Nolan, Environmental Innovations Chemist

- ✔ Fast, efficient analyses, using an Rtx®-CLPesticides column and flame photometric detection.
- ✔ New OPP reference mix corresponds to strict European criteria.
- ✔ Concentrations of mix components vary according to FPD response.

Organophosphorus pesticides (OPP) are widely used to protect fruits and vegetables from insects. Unfortunately, these toxic materials can accumulate in human fat tissue, potentially leading to death by respiratory depression.[1] In Europe, OPPs are considered a very serious risk to human health, and analysis of food products for these pesticides is important in quality control. OPPs also are strictly regulated in the United States. Policy in regard to pesticide residues in milk, eggs, meat, or poultry is stated in the Code of Federal Regulations.[2] Multi-residue methods in AOAC Official Methods of Analyses[3] and the FDA Pesticide Analytical Manual[4] are used to determine OPPs and organochlorine pesticides. Analytical procedures for OPPs usually involve GC with a selective detector, such as a flame photometric detector (FPD) or a nitrogen-phosphorous detector (NPD), to detect low ppb levels of target compounds. Use of an FPD in the phosphorous mode minimizes interference by materials that do not contain phosphorus.

Recently, our European customers asked us to develop a stable reference mix of target OPPs, with concentrations appropriate for analysis by GC/FPD. The components in our new European OPP Mix are especially important in quality control of milk, infant formulas, and baby foods. We include these in varied concentrations, according to the responses they elicit from an FPD. One of the OPPs, demeton, is a mixture of O- and S- isomers. We include demeton-S in the mix, and follow US EPA Method 8141B for quantifying this isomer. OPP compounds are photosensitive and are easily degraded during handling, storage, or analysis. When preparing and storing the new mix, we follow stringent measures to ensure prolonged stability.

Analyses of OPPs are challenging and time-consuming. Individual OPPs are difficult to identify because of coelutions and shifting retention times on different capillary phases. OPPs can degrade on reactive sites in the chromatography system, so it is important that the injector be free of surface contaminants. The analysis usually requires high temperatures, and often causes bleed problems. Analysis time typically exceeds 40 minutes. Tributyl phosphate and triphenyl phosphate are recommended surrogates for GC/FPD (EPA Method 8141B).

To meet the challenges of OPPs analysis, Restek chemists use an Rtx®-CLPesticides column. Rtx®-CLPesticides columns have a 330ºC maximum operating temperature, superior inertness, and low bleed. Figure 1 shows excellent resolution of the European OPP Mix, obtained in less than 25 minutes. The 30m, 0.32mm ID, 0.50µm column is well suited to the split/splitless injection and FPD detection: all compounds, including the surrogates, are resolved. The high temperature stability of Rtx®-CLPesticide columns enables us to program the column to 330°C following each analysis, to bake out high molecular weight contaminants common in OPP-containing extracts. The ability to clean an Rtx®-CLPesticides column at high temperatures enables us to make more injections before replacing the column, compared to commonly used cyano phase columns.

Fast, efficient separations are an important goal in any analysis. In analyses of organophosphate pesticides, an Rtx®-CLPesticides column, and our new European OPP Mix, are important parts of attaining this goal.

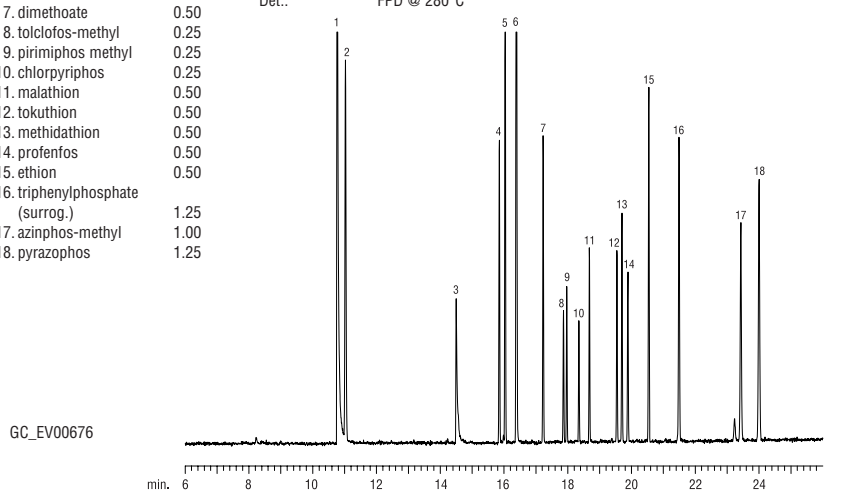
References:

1. *Monitoring exposure of organophosphorus and/or carbamate insecticides* Saskatchewan Labor, Canada.
2. Code of Federal Regulations, 40 CFR sec. 180.6. Office of the Federal Register Archives and Records Administration.
3. Official Methods of Analysis 5th Ed., 1990, Section 970.52. Association of Official Analytical Chemists, AOAC International, Arlington, VA.
4. Pesticide Analytical Manual Vol.1, 3rd Ed., 1994, Section 304-11. U.S. Department of Health and Human Services, Food and Drug Administration.

Figure 1 — Organophosphorus Pesticides Mix, European Formulation on an Rtx®-CLPesticides column.

	Concentration on-column (ng)
1. methamidophos	1.25
2. dichlorvos	1.25
3. acephate	0.50
4. demeton-s-methyl	0.50
5. tributylphosphate (IS)	1.25
6. omethoate	2.50
7. dimethoate	0.50
8. tolclofos-methyl	0.25
9. pirimiphos methyl	0.25
10. chlorpyriphos	0.25
11. malathion	0.50
12. tokuthion	0.50
13. methidathion	0.50
14. profenfos	0.50
15. ethion	0.50
16. triphenylphosphate (surrog.)	1.25
17. azinphos-methyl	1.00
18. pyrazophos	1.25

Rtx®-CLPesticides 30m, 0.32mm ID, 0.50µm (cat.# 11139)
Sample: European OPP Mix (cat.# 32418) 100–1000µg/mL in acetone
Inj.: 0.5µL direct, open-top Siltek™ Drilled Uniliner® inlet liner (cat.# 21055-214.5)
Inj. temp.: 250°C
Carrier gas: helium, constant pressure
Linear velocity: 35cm/sec. @ 80°C
Oven temp.: 80°C (hold 1 min.) to 150°C @ 7°C/min. to 280°C @ 15°C/min. (hold 10 min.)
Det.: FPD @ 280°C



Organophosphorous Pesticide Mix, European Formulation

(16 components)

acephate	200µg/mL
azinphos methyl(guthion)	400
chlorpyrifos	100
demeton-s-methyl	200
dichlorvos (DDVP)	500
dimethoate	200
ethion	200
malathion	200
methamidophos	500
methidathion	200
omethoate	1000
pirimiphos methyl	100
profenfos	200
pyrazophos	500
tokuthion (prothiofos)	200
tolclofos-methyl	100

In methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
32418	32418-510	—
	w/data pack	
32418-500	32418-520	32518

the RESTEK Advantage

Innovators of High Resolution Chromatography Products

Low-Bleed Column, New Reference Mixes for Semivolatile Organic Analytes in Drinking Water

Using Gas Chromatography/Mass Spectrometry (EPA Method 525.2)

By Christopher English, Environmental Innovations Chemist & Katia May, Ph.D., R&D Chemist

Rtx®-5Sil MS column provides:

- ✔ extremely low bleed, for greater sensitivity in GC/MS applications.
- ✔ excellent resolution of 110 target semivolatile compounds in EPA Method 525.2.

EPA 525.2 reference materials are:

- ✔ economical — calibration mixtures at 1000µg/mL concentration, for more analyses per ampul.
- ✔ convenient — 106 compounds in only six mixtures.
- ✔ calibration mixtures formulated by chemical class: semivolatiles, PCB congeners, organochlorine pesticides, nitrogen/phosphorus pesticides.
- ✔ six nitrogen/phosphorus pesticides in a separate mix, for stability.

Gas chromatographic analyses for semivolatile analytes in drinking water require an inert, thermally stable, low-bleed stationary phase. EPA Method 525, a liquid-solid extraction / capillary GC/MS analysis, is applicable for monitoring a wide range of semivolatiles in an aqueous matrix. The new revision, Method 525.2, includes 110 target compounds. Restek provides the materials needed for this analysis: extraction disks, reference materials, and an inert column capable of excellent response for acids and bases, even at single digit nanogram on-column quantities.

Of the EPA GC/MS methods for analyzing semivolatiles, Method 525.2 is the most demanding for column inertness. Method 525.2 target analytes include many active compounds, e.g., endrin, methoxychlor, DDT, pentachlorophenol. Simple adjustments to the injection conditions can yield great improvements in sensitivity, especially for active and high molecular weight compounds. Analytes can degrade in the injection port and exhibit excessive tailing. To prevent this, we use a Drilled Uniliner® inlet liner: a Press-Tight® seal between the fused silica column and the internal surface of the liner eliminates contact between the sample and the hot metal surfaces in the injection port. A pulsed injection (30psi, 0.5 min.) reduces the time the analytes spend in the injection port, and helps to minimize breakdown. Pulsed pressure injection increases the possibility of breaking the seal between the column and the liner. Therefore, head pressure should not exceed 50psi when using the pulsed splitless mode. A starting temperature of 35°C helps ensure excellent peak shapes for early eluting target analytes.

To reduce bleed and activity, Restek continues to explore new synthesis routes for both column deactivation and the stationary phase polymer. Improvements in technology allow our Rtx®-5Sil MS columns to withstand high bake-out temperatures without loss of deactivation. The inset in Figure 1 is an example of superior efficiency and low bleed for a mid-point standard, at 330°C. Peak shape and

in this issue

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2003 vol.3

response are excellent for the very active compounds endrin, DDT, and methoxychlor, peaks 89, 95, and 102, respectively.

Low-bleed Rtx®-5Sil MS columns ensure low detection limits and excellent instrument stability in semivolatiles analysis.

We incorporate 106 Method 525.2 target compounds in six new, stable mixtures. The concentration of each component in these mixes, 1000µg/mL,[1] is considerably higher than in mixes from other sources, and significantly more analyses can be conducted from each ampul of material. We have formulated the new calibration mixes by chemical class: semivolatiles, PCB congeners, organochlorine pesticides, nitrogen/phosphorous pesticides.[2] Pentachlorophenol is included in the semivolatiles mix at a concentration four times higher than the other analytes, as required by the method. For the convenience of our customers we include heptachlor epoxide isomer A, an analyte not on the target list for Method 525.2, in Chlorinated Pesticides Mix #2. Six of the nitrogen/phosphorous pesticide analytes on the target list are unstable in aqueous matrices; we combine these analytes in a separate mix (cat.# 32423). We do not include two compounds, disulfoton sulfoxide and disulfoton sulfone, in either our new mixes or our existing mixes. In water, disulfoton is rapidly oxidized by chemical reaction to the analogous sulfoxide and sulfone. Consequently, the sulfoxide and sulfone cannot be included in the mix with disulfoton. We offer individual solutions of the two analytes — please refer to our catalog or web site.[3]

In addition to calibration mixes for Method 525, we also have all needed quality control standards: internal and surrogate standards, a fortification recovery standard, and a GC/MS performance check standard. New Method 525.2 Surrogate Standards Mix includes optional pyrene-d10. All new mixes are described on page 3.

References

[1] Components in Method 525.2 PCB Congeners Mix (cat.# 32420) are at 200µg/mL each.

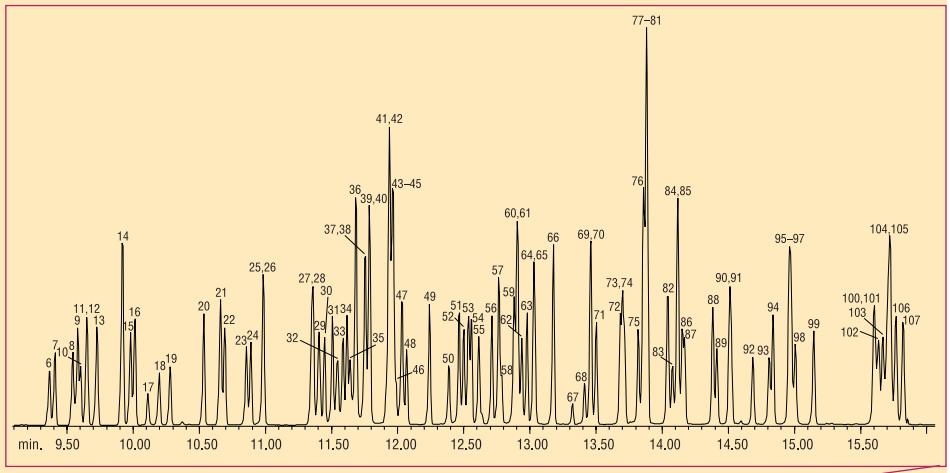
[2] Previously available mixes for Method 525 include Organochlorine Pesticides Mix AB #3 (cat.# 32415), individual Aroclor® PCB solutions (cat.# 32075, 32077, 32079, 32081, 32083, 32085, 32087), and TCLP Toxaphene (cat.# 32015).

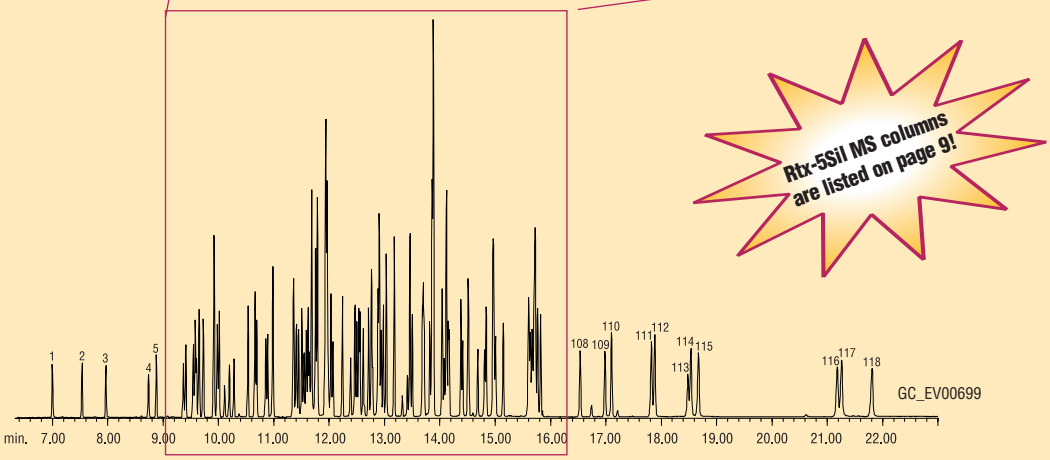
[3] Catalog # MET 652-A and MET 652-B, respectively.

Figure 1 - Low bleed and excellent resolution of complex semivolatiles sample, using an Rtx®-5Sil MS column.

Rtx®-5Sil MS 30m, 0.25mm ID, 0.25µm (cat.# 12723)

Sample:	US EPA Method 525.2 Mix 1µL 10ppm (20ppm IS) Standards used: cat. #s 31824, 32420, 32421, 32422, 32423, 31825, 31826, 31828, 32291
Inj.:	1.0µL 30psi 0.4min. pulsed splitless (hold 0.4 min.), 4mm Drilled Uniliner® (cat.# 21055)
Inj. Temp.:	300°C
Carrier Gas:	helium, constant flow
Flow Rate:	0.8 mL/min.
Oven Temp.:	35°C (hold 1 min.) to 270°C @ 18°C/min. (hold 0 min.) to 330°C @ 5°C/min. (hold 1 min.)
Det:	Agilent 5973 GC/MS
Transfer Line Temp.:	280°C
Scan Range:	40–550 amu
Solvent Delay:	4.0 min.
Tune:	DFTPP
Ionization:	EI





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

Method 525.2 Semivolatile Mix (25 components)

acenaphthylene	1,000µg/mL	2,4-dinitrotoluene	1,000
anthracene	1,000	2,6-dinitrotoluene	1,000
benzo(a)anthracene	1,000	fluorene	1,000
benzo(a)pyrene	1,000	hexachlorobenzene	1,000
benzo(b)fluoranthene	1,000	hexachlorocyclopentadiene	1,000
benzo(ghi)perylene	1,000	indeno(1,2,3-cd)pyrene	1,000
benzo(k)fluoranthene	1,000	isophorone	1,000
benzylbutylphthalate	1,000	pentachlorophenol	**4,000**
bis(2-ethylhexyl)adipate	1,000	phenanthrene	1,000
bis(2-ethylhexyl)phthalate	1,000	pyrene	1,000
chrysene	1,000		
dibenzo(a,h)anthracene	1,000		
diethylphthalate	1,000		
dimethylphthalate	1,000		
di-n-butylphthalate	1,000		

In acetone, 1mL/ampul

Each	5-pk.	10-pk.
31824	31824-510	—
	w/data pack	
31824-500	31824-520	31924

Method 525.2 PCB Congener Mix (8 components)

2-chlorobiphenyl (BZ#1)
2,3-dichlorobiphenyl (BZ#5)
2,4,5-trichlorobiphenyl (BZ#29)
2,2',4,4'-tetrachlorobiphenyl (BZ#47)
2,2',3,4,6-pentachlorobiphenyl (BZ#98)
2,2',4,4',5,6'-hexachlorobiphenyl (BZ#154)
2,2',3,3',4,4',6-heptachlorobiphenyl (BZ#171)
2,2',3,3',4,5',6,6'-octachlorobiphenyl (BZ#200)

200µg/mL each in acetone, 1mL/ampul

Each	5-pk.	10-pk.
32420	32420-510	—
	w/data pack	
32420-500	32420-520	32520

Method 525.2 Chlorinated Pesticide Mix #2 (12 components)

chlorobenzilate
chloroneb
chlorothalonil
chlorpyrifos
cyanazine (Bladex)
DCPA methyl ester (Dacthal®)
etridiazole (Terrazole®)
heptachlor epoxide (isomer A)
trans-nonachlor
cis-permethrin
trans-permethrin
propachlor

1,000µg/mL each in acetone, 1mL/ampul

Each	5-pk.	10-pk.
32421	32421-510	—
	w/data pack	
32421-500	32421-520	32521

Organochlorine Pesticide Mix AB # 3 (20 components)

aldrin
a-BHC
b-BHC
d-BHC
g-BHC (lindane)
a-chlordane
g-chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
dieldrin
endosulfan I
endosulfan II
endosulfan sulfate
endrin
endrin aldehyde
endrin ketone
heptachlor
heptachlor epoxide (isomer B)
methoxychlor

2,000µg/mL each in hexane:toluene (1:1), 1mL/ampul

Each	5-pk.	10-pk.
32415	32415-510	—
	w/data pack	
32415-500	32415-520	32515

Method 525.2 Nitrogen/Phosphorous Pesticide Mix #2 (6 components)

carboxin
diazinon
disulfoton
fenamiphos
merphos (tributylphosphorotrithioite)
terbufos

1,000µg/mL each in acetone, 1mL/ampul

Each	5-pk.	10-pk.
32423	32423-510	—
	w/data pack	
32423-500	32423-520	32523

Method 525.2 Nitrogen/Phosphorous Pesticide Mix #1 (39 components)

alachlor
ametryn
atraton
atrazine
bromacil
butachlor
butylate
chlorpropham
cycloate
dichlorvos (DDVP)
diphenamid
EPTC
ethoprop (ethoprophos)
fenarimol
fluridone (Sonar®)
hexazinone
methyl parathion OA
metolachlor
metribuzin*
mevinphos (phosdrin)
MGK-264
molinate
napropamide
norflurazon
pebulate
prometon
prometryne
pronamide
propazine
simazine
simetryn
stirofos (tetrachlorvinphos)
tebuthiuron
terbacil
terbutryn
triadimefon
tricyclazole
trifluralin
vernolate

1,000µg/mL each in acetone, 1mL/ampul

Each	5-pk.	10-pk.
32422	32422-510	—
	w/data pack	
32422-500	32422-520	32522

**Offered independently; please inquire.*

Method 525.2 Internal Standard Mix

acenaphthene-d10
chrysene-d12
phenanthrene-d10

1,000µg/mL each in acetone, 1mL/ampul

Each	5-pk.	10-pk.
31825	31825-510	—
	w/data pack	
31825-500	31825-520	31925

Method 525.2 GC/MS Performance Check Mix

4,4'-DDT
DFTPP (decafluorotriphenylphosphine)
endrin

1,000µg/mL each in acetone, 1mL/ampul

Each	5-pk.	10-pk.
31827	31827-510	—
	w/data pack	
31827-500	31827-520	31927

Method 525.2 Surrogate Standard Mix

2-nitro-*m*-xylene (1,3-dimethyl-2-nitrobenzene)
perylene-d12
pyrene-d10
triphenylphosphate

1,000µg/mL each in acetone, 1mL/ampul

Each	5-pk.	10-pk.
31826	31826-510	—
	w/data pack	
31826-500	31826-520	31926

Method 525.2 Fortification Recovery Standard

p-terphenyl-d14
1,000µg/mL in acetone, 1mL/ampul

Each	5-pk.	10-pk.
31828	31828-510	—
	w/data pack	
31828-500	31828-520	31928

Resprep™-C18 SPE Disks

- 47mm glass fiber embedded with C18 bonded silica.
- Deep-pore design reduces clogging and allows faster flow rates.

Description	qty.	cat.#
Resprep™-C18–47 Disks	20-pk.	24004

Verify Fruit Juice Quality from Organic Acid Profiles

Using New Allure™ Organic Acids HPLC Column

By Rebecca Wittrig, Ph.D., Senior Innovations Chemist

- ✔ One 30cm Allure™ Organic Acids column replaces two C18 columns in AOAC methodology.
- ✔ Stable and reproducible retention, even with 100% aqueous mobile phases, as in AOAC method 986.13.
- ✔ Facilitates detection of fruit juice adulteration.

Organic acids are difficult to analyze on conventional reversed phase columns. A 100% aqueous mobile phase increases interaction between the acids and the stationary phase, but C18 chains collapse in a totally aqueous environment. The Allure™ Organic Acids column was designed to enhance retention and selectivity for this challenging application. Novel binding chemistry ensures the alkyl groups in Allure™ Organic Acids columns remain extended in 100% aqueous mobile phases; retention is stable and reproducible.

The fruit juice industry in the US alone is worth over $12 billion per year[1] and is many times that worldwide. High-value juices have been replaced or extended through substitution of sugars for juice solids, or by dilution with less expensive juices, such as white grape juice or pear juice. To detect adulteration, investigators examine sugar profiles and sorbitol content; minerals; anthocyanin pigments; phenolics; oligosaccharides; carbon stable isotope ratio for various components; and/or organic acid profiles. Because juices are chemically complex, several complementary analyses should be performed to verify authenticity. The resolving power of high performance liquid chromatography (HPLC) is invaluable for accurately quantifying many of these components.

The organic acids that give fruit products their characteristic tartness vary in combination and in concentrations among different juices, and the organic acid profile can be used to identify a juice or verify its purity. For example, malic acid is a major component of the organic acid content of apple juice. If apple juice has been diluted, e.g., with sugar water, the malic acid content will be low. Cranberry juice contains quinic, malic, and citric acids; grape juice, on the other hand, contains relatively high levels of tartaric acid. A "cranberry juice" that contains measurable amounts of tartaric acid should be suspect.

Typically, organic acids in fruit juices are identified and quantified by using methods such as AOAC method 986.13.[2] In this procedure, reversed phase HPLC is used to separate the acids. Because several of the analytes are extremely difficult to resolve, a 100% aqueous mobile phase is needed to enhance interaction between the acids and the C18 stationary phase, but the C18 chains in conventional columns collapse in a totally aqueous environment, greatly reducing the resolving capability of the column. To compensate, two columns must be used in series.

Now there is a simpler and more reliable approach: a single 30cm Allure™ Organic Acids column effectively resolves key organic acids, under the chromatographic conditions specified in AOAC method 986.13. Figure 1 shows a separation of typical fruit juice organic acids: tartaric, quinic, malic, citric, and fumaric acids. Note the excellent resolution of tartaric and quinic acids! This superior performance makes interpretation of the data more reliable. Similarly, note the distinct organic acid profiles for grape juice and cranberry juice cocktail in Figure 2.

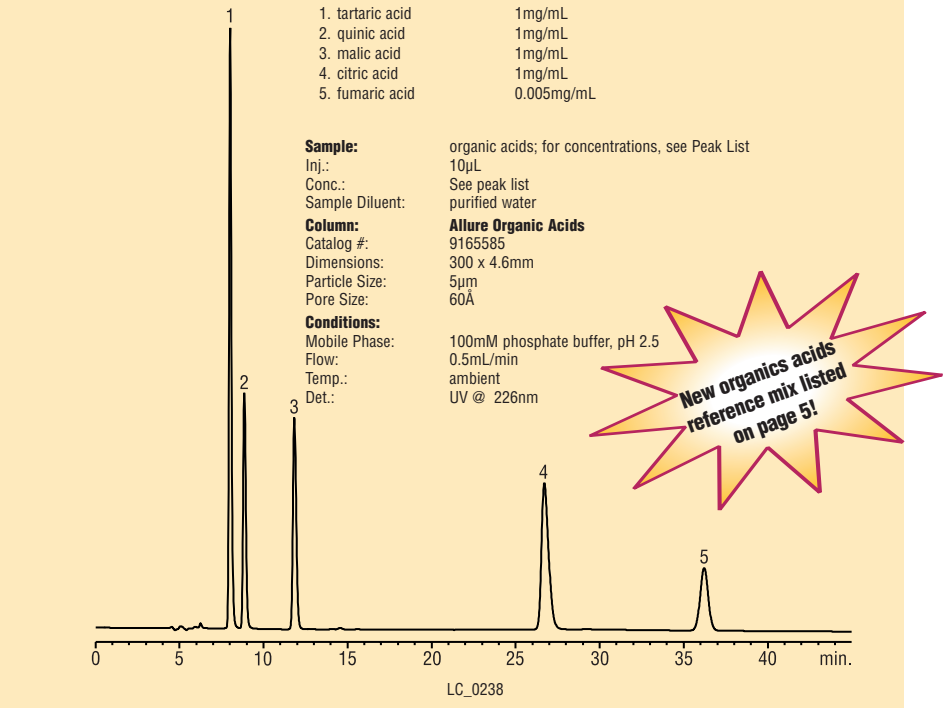
Analysis of polar organic acids is difficult at best on conventional reversed phase columns. In contrast, an Allure™ Organic Acids column provides excellent retention and selectivity for these compounds, allowing the separation to be performed on a single column. Retention is stable and reproducible, even with a 100% aqueous mobile phase as specified in AOAC method 986.13. If you are monitoring fruit juice quality, and want a trouble-free analysis with accurate results, we highly recommend an Allure™ Organic Acids column.

References

[1] *Authenticity of Apple Juice* Technical Bulletin #2 (1996), Analytical Chemical Services of Columbia, Inc.

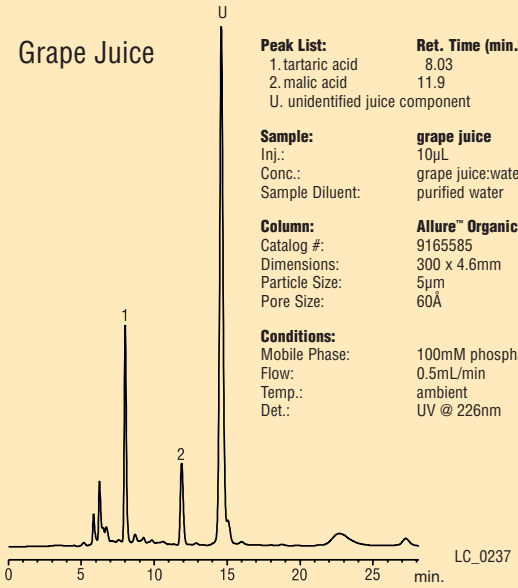
[2] Official Methods of Analysis (2000), AOAC International, 17th edition, method #986.13.

Figure 1 – Excellent separation of organic acids, including tartaric and quinic acids, using one 30cm Allure™ Organic Acids column.



New organics acids reference mix listed on page 5!

Figure 2 – Sharp, easily differentiated organic acid profiles for grape juice and cranberry juice cocktail, provided by an Allure™ Organic Acids column.

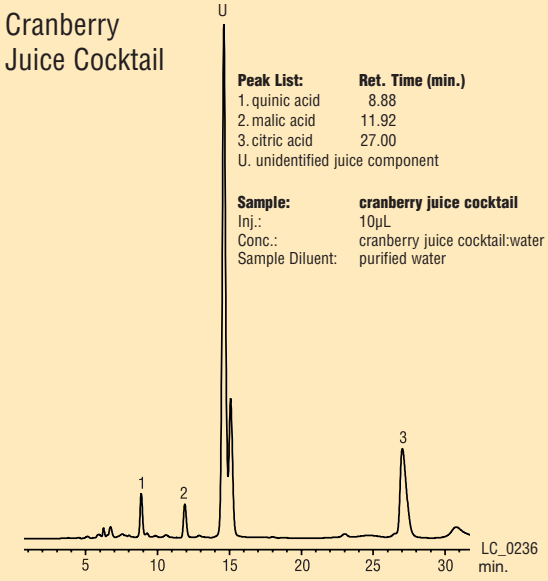


Peak List: **Ret. Time (min.)**
1. tartaric acid 8.03
2. malic acid 11.9
U. unidentified juice component

Sample: **grape juice**
Inj.: 10µL
Conc.: grape juice:water (50:50, v/v)
Sample Diluent: purified water

Column: **Allure™ Organic Acids**
Catalog #: 9165585
Dimensions: 300 x 4.6mm
Particle Size: 5µm
Pore Size: 60Å

Conditions:
Mobile Phase: 100mM phosphate buffer, pH 2.5
Flow: 0.5mL/min
Temp.: ambient
Det.: UV @ 226nm



Peak List: **Ret. Time (min.)**
1. quinic acid 8.88
2. malic acid 11.92
3. citric acid 27.00
U. unidentified juice component

Sample: **cranberry juice cocktail**
Inj.: 10µL
Conc.: cranberry juice cocktail:water (50:50, v/v)
Sample Diluent: purified water

Allure™ Organic Acids Column (5µm silica; 4.6mm ID)

Length	cat.#
150mm	9165565
250mm	9165575
300mm	9165585

Organic Acids Reference Mixture

citric acid	2000µg/mL	quinic acid	2000
fumaric acid	10	tartaric acid	2000
malic acid	2000		

Each	5-pk.	10-pk.
In water, 1mL/ampul		
35080	35080-510	—
35080-500*	35080-520*	35180*
In water, 5mL/ampul		
35081	35081-510	—
35081-500*	35081-520*	35181*

*w/data pack

Baseline Separation of PAHs by HPLC, Using a Pinnacle II™ PAH Column

- ✔ Fast analysis — resolve 16 target PAHs in less than 18 minutes!
- ✔ Excellent resolution — cross-linked C18 phase gives baseline separation.
- ✔ Column-to-column reproducibility — we control the entire manufacturing process: silica production - column manufacture - final testing. You will see the same outstanding performance from every Pinnacle II™ PAH column you use!

Figure 1 - Baseline separation of US EPA Method 610 PAHs in <18 minutes.

Column: **Pinnacle II™ PAH**
Catalog #: 9219563
Dimensions: 150 x 3.2mm
Particle Size: 5µm
Pore Size: 110Å

Conditions:
Mobile Phase: A: purified water, B: acetonitrile

Time (min.)	%B
0	40
7	60
11	100
17.9	100
18	40

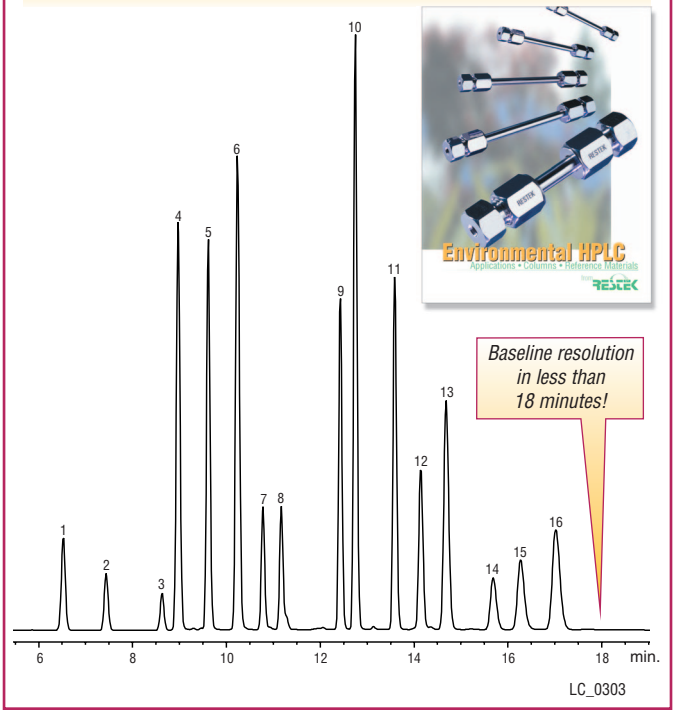
Flow: 1.2 mL/min
Temp.: ambient
Det.: UV @ 254nm

Sample:
Inj.: 5µL PAH standard (cat.#31264)
Conc.: see peak list
Sample Diluent: 1:9 methylene chloride:acetonitrile
Sample Temp.: ambient

Peak List:	Conc. (µg/mL)
1. naphthalene	100
2. acenaphthylene	100
3. acenaphthene	100
4. fluorene	100
5. phenanthrene	50
6. anthracene	100
7. fluoranthene	50
8. pyrene	50
9. benzo(a)anthracene	50
10. chrysene	50
11. benzo(b)fluoranthene	50
12. benzo(k)fluoranthene	50
13. benzo(a)pyrene	50
14. dibenzo(a,h)anthracene	50
15. benzo(ghi)perylene	50
16. indeno(1,2,3-cd)pyrene	50

Other reference mixes available — please see our catalog or website.

An example chromatogram from our new ***Environmental HPLC*** flyer. Request your copy today! lit. cat.#59741



New Volatiles Reference Mix for OLC 03.2 Statement of Work

By Katia May, Ph.D., R&D Chemist and Chris English, Environmental Innovations Chemist

- ✔ All materials needed for analysis of volatiles.
- ✔ New MegaMix™ includes 42 target compounds in a single mixture.
- ✔ Co-eluters *m*- and *p*-xylene at one-half concentration of other volatiles.
- ✔ Deuterated monitoring compound mixes for quality control.
- ✔ Rtx®-VMS column separates OLC 03.2 volatiles in less than 10 minutes.

The latest revision to the US EPA Contract Laboratory Program (CLP) methods, OLC 03.2, adds nine compounds to the target list of volatile organic analytes: cyclohexane, dichlorodifluoromethane, isopropylbenzene, methyl acetate, methyl *tert*-butyl ether, methylcyclohexane, 1,2,3-trichlorobenzene, trichlorofluoromethane, and 1,1,2-trichloro-1,2,2-trifluoroethane (Freon® 113). Further, the method requires adding 14 deuterated monitoring compounds (DMC) to a volatiles sample as a sample-by-sample accuracy indicator. Volatiles and DMC are purged from a sample, trapped, and desorbed into the GC/MS for analysis.

Our new OLC 03.2 VOA MegaMix™ reference mix contains the maximum number of volatile analytes consistent with stable formulation. Co-eluting *m*- and *p*-xylene are included at one-half the concentration of the other 40 compounds, allowing these analytes to be calibrated at lower detection limits and thus eliminating the time and expense of a second calibration. New OLC 03.2 VOA Deuterated Monitoring Compounds Mix contains all 14 DMC specified in OLC 03.2. In combination with the two new mixes, our gas and ketone mixes (cat.# 30042 and cat.# 30006) and L/C VOA Internal Standard Mix (cat.# 30091) make a complete set of reference materials for OLC 03.2 SOW volatiles.

We designed Rtx®-VMS capillary columns specifically for fast analyses of volatile organics. Excellent thermal stability ensures low bleed when analyzing higher-boiling analytes. A 20m, 0.18mm ID, 1.0µm Rtx®-VMS column separates the OLC 03.2 volatiles in less than 10 minutes (Figure 1). These optimized conditions resolve carbon tetrachloride from 1,1,1-trichloroethane (peaks 20 and 21) and prevent ions from these analytes from interfering with quantification. The 45°C initial temperature allows rapid oven cycles without sacrificing resolution of the gases.

OLC 03.2 VOA Deuterated Monitoring Compounds (DMC) (14 components)

Non-Ketones:

benzene-d6
bromoform-d
chloroethane-d5
chloroform-d
1,2-dichlorobenzene-d4
1,2-dichloroethane-d4
1,1-dichloroethene-d2
1,2-dichloropropane-d6
trans-1,3-dichloropropene-d4
1,1,2,2-tetrachloroethane-d2
toluene-d8
vinyl chloride-d3

100µg/mL each in P&T methanol, 1mL/ampul

Ketones:

2-butanone-d5
2-hexanone-d5

200µg/mL each in P&T methanol, 0.5mL/ampul

Each	5-pk.
30493	30493-510

OLC 03.2 VOA MegaMix™ (42 components)

Components listed in Figure 1 (bold)

2,000µg/mL each in P&T methanol (m-& p-xylene at 1,000µg/mL), 1 mL/ampul

Each	5-pk.	10-pk.
30492	30492-510	—
	w/data pack	
30492-500	30492-520	30592

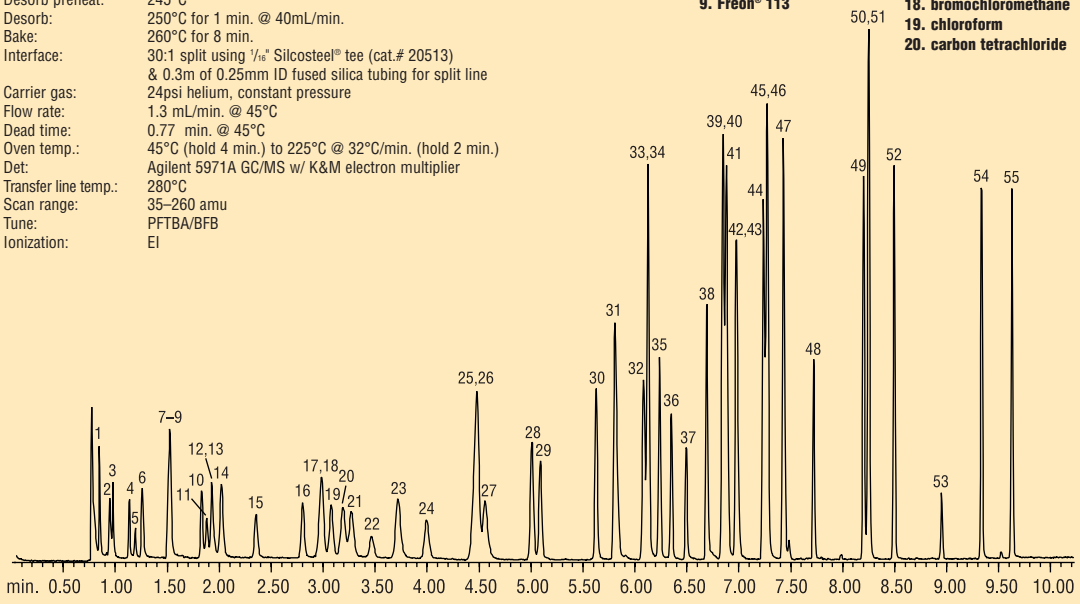
Rtx®-VMS Column (fused silica)

ID	df (µm)	temp. limits	40-Meter
0.18mm	1.00	-40 to 240/260°C	49915

Figure 1 – Ten-minute analysis of 50+ OLC 03.2 volatile compounds, using an Rtx®-VMS column and new Restek reference mixes.

Rtx®-VMS 20m, 0.18mm ID, 1.0µm (cat.# 49915)

Sample:	Components at 10ppb (50 ppb ketones) in 5mL RO water OLC 03.2 VOA MegaMix™, cat.# 30492 502.2 Calibration Mix #1 (gases), cat.# 30042 VOA Calibration Mix #1 (ketones), cat.# 30006 L/C VOA Internal Standard Mix, cat.# 30091
Concentrator:	Tekmar 3100 Purge and Trap
Trap:	Vocarb 3000
Purge:	11 min. @ 40mL/min. @ ambient temperature
Dry purge:	1 min. @ 40mL/min. (MCS bypassed)
Desorb preheat:	245°C
Desorb:	250°C for 1 min. @ 40mL/min.
Bake:	260°C for 8 min.
Interface:	30:1 split using 1/16" Silcosteel® tee (cat.# 20513) & 0.3m of 0.25mm ID fused silica tubing for split line
Carrier gas:	24psi helium, constant pressure
Flow rate:	1.3 mL/min. @ 45°C
Dead time:	0.77 min. @ 45°C
Oven temp.:	45°C (hold 4 min.) to 225°C @ 32°C/min. (hold 2 min.)
Det:	Agilent 5971A GC/MS w/ K&M electron multiplier
Transfer line temp.:	280°C
Scan range:	35–260 amu
Tune:	PFTBA/BFB
Ionization:	EI



Bold type–components of OLC 03.2 VOA MegaMix™ (cat.# 30492)

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. carbon disulfide
9. Freon® 113
10. methylene chloride
11. acetone
12. *trans*-1,2-dichloroethene
13. methyl acetate
14. methyl *tert*-butyl ether
15. 1,1-dichloroethane
16. *cis*-1,2-dichloroethene
17. cyclohexane
18. bromochloromethane
19. chloroform
20. carbon tetrachloride
21. 1,1,1-trichloroethane
22. 2-butanone
23. benzene
24. 1,2-dichloroethane
25. methyl cyclohexane
26. trichloroethene
27. 1,4-difluorobenzene
28. 1,2-dichloropropane
29. bromodichloromethane
30. *cis*-1,3-dichloropropene
31. toluene
32. tetrachloroethene
33. 4-methyl-2-pentanone
34. *trans*-1,3-dichloropropene
35. 1,1,2-trichloroethane
36. dibromochloromethane
37. 1,2-dibromoethane
38. 2-hexanone
39. chlorobenzene-d5
40. chlorobenzene
41. ethylbenzene
42. *m*-xylene
43. *p*-xylene
44. *o*-xylene
45. styrene
46. bromoform
47. isopropylbenzene
48. 1,1,2,2-tetrachloroethane
49. 1,3-dichlorobenzene
50. 1,4-dichlorobenzene-d4
51. 1,4-dichlorobenzene
52. 1,2-dichlorobenzene
53. 1,2-dibromo-3-chloropropane
54. 1,2,4-trichlorobenzene
55. 1,2,3-trichlorobenzene

GC_EV00681

New Semivolatiles Reference Mix for OLC 03.2 Statement of Work

By Katia May, Ph.D., R&D Chemist, and Gary Stidsen, Innovations Team Manager

- ✔ All reference materials needed for analysis of semivolatiles.
- ✔ New MegaMix™ includes 57 target compounds; co-eluters 3- and 4-methylphenol at one-half concentration of other semivolatiles.
- ✔ Fortification mix includes seven semivolatiles that must be calibrated at higher, variable concentrations.
- ✔ Deuterated monitoring compounds mix for quality control.

Revision OLC 03.2 adds six compounds to the target list of CLP semivolatile analytes: acetophenone, atrazine, benzaldehyde, 1,1'-biphenyl, caprolactam, and 1,2,4,5-tetrachlorobenzene. Balancing economy, convenience and long shelf life, Restek chemists have combined 64 target compounds in two mixes: a new semivolatiles MegaMix™ reference mix and a fortification mix. OLC 03.2 SV MegaMix™ contains 57 analytes, at 1000µg/mL each, in methanol-free methylene chloride. We include co-eluting isomers 3- and 4-methylphenol at one-half the concentration of the other compounds, allowing these analytes to be calibrated at lower detection limits (Figure 1). N-Nitrosodiphenylamine decomposes to diphenylamine at the GC inlet. In formulating the new MegaMix™ we include the compound that is analyzed, diphenylamine, rather than the parent compound, N-nitrosodiphenylamine.

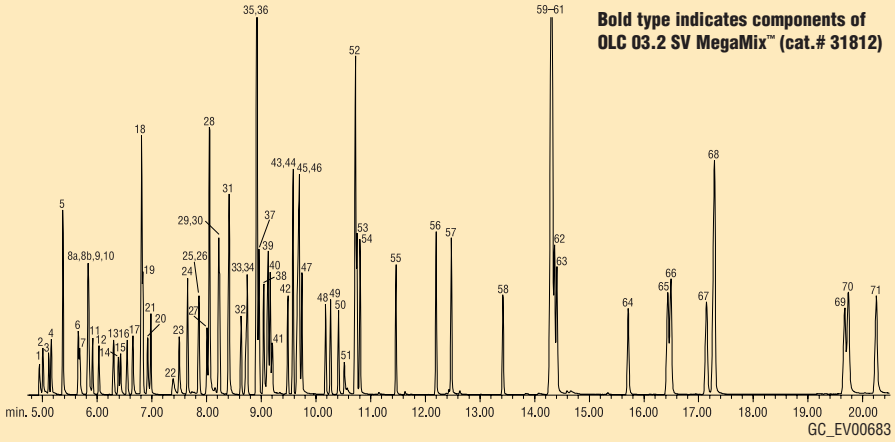
We designed the new Fortification Mix to simplify the analysis — it includes the seven compounds that must be calibrated at higher and variable concentrations, at 2000µg/mL each. To avoid decomposition and reaction issues, we offer 3,3'-dichlorobenzidine (cat.# 31835) and 4-chloroaniline (cat.# 31211) as individual solutions.

Because benzaldehyde reacts with alcohols, we use methanol-free methylene chloride to prepare the OLC 3.2 calibration and single component mixes, to improve the shelf-life of these mixes. In addition, we recommend working dilutions be prepared with methanol-free methylene chloride, to ensure stability. Benzaldehyde also reacts rapidly with 4-chloroaniline or 3,3'-dichlorobenzidine; the analyst should consider generating a separate calibration curve for 4-chloroaniline and 3,3'-dichlorobenzidine, independent of the primary calibration curve.

OLC 03.2 specifies addition of deuterated monitoring compounds to every semivolatiles sample, standard, and blank. Our new OLC 03.2 SVOA Deuterated Monitoring Compounds (DMC) mix meets the requirement. Together with our Semivolatile Internal Standard Mix (cat.# 31206 or 31006), Acid Matrix Spike Mix (cat.# 31005, 31075, or 31085), Base/Neutral Spike Mix (cat.# 31492) and SV Tuning Compound (cat.# 31001), the new formulations make a full complement of reference materials for the semivolatiles listed in OLC 03.2 SOW.

Carefully formulated reference mixes and capillary columns designed specifically for challenging environmental analyses make Restek the perfect source for all of your CLP methods needs.

Figure 1 - Excellent resolution and rapid analysis of OLC 03.2 semivolatile compounds, using an Rtx®-5Sil MS column and new Restek reference mixes.



1. benzaldehyde	**15. 2,4-dimethylphenol**	**30. 2-chloronaphthalene**	45. 4-nitroaniline**
2. phenol	**16. bis(2-chloroethoxy)methane**	31. 2-nitroaniline**	46. 4,6-dinitro-2-methylphenol**
3. bis(2-chloroethyl)ether	**17. 2,4-dichlorophenol**	**32. dimethylphthalate**	**47. diphenylamine**
4. 2-chlorophenol	18. naphthalene-d8 (IS)*	**33. 2,6-dinitrotoluene**	**48. 4-bromophenyl phenyl ether**
5. 1,4-dichlorobenzene-d4 (IS)*	**19. naphthalene**	**34. acenaphthylene**	**49. hexachlorobenzene**
6. 2-methylphenol	20. 4-chloroaniline	35. 3-nitroaniline**	**50. atrazine**
7. bis(2-chloroisopropyl)ether	**21. hexachlorobutadiene**	36. acenaphthene-d10 (IS)	**51. pentachlorophenol**
8a. 4-methylphenol	**22 caprolactam**	**37. acenaphthene**	52. phenanthrene-d10 (IS)*
8b. 3-methylphenol	**23. 4-chloro-3-methylphenol**	38. 2,4-dinitrophenol**	**53. phenanthrene**
9. acetophenone	**24. 2-methylnaphthalene**	39. 4-nitrophenol**	**54. anthracene**
10. N-nitroso-di-*n*-propylamine	**25. hexachlorocyclopentadiene**	**40. dibenzofuran**	**55. di-*n*-butylphthalate**
11. hexachloroethane	**26. 1,2,4,5-tetrachlorobenzene**	**41. 2,4-dinitrotoluene**	**56. fluoranthene**
12. nitrobenzene	**27. 2,4,6-trichlorophenol**	**42. diethyl phthalate**	**57. pyrene**
13. isophorone	28. 2,4,5-trichlorophenol**	**43. fluorene**	**58. butyl benzyl phthalate**
14. 2-nitrophenol	**29. biphenyl**	**44. 4-chlorophenyl phenyl ether**	**59. benzo(a)anthracene**
			60. 3,3'-dichlorobenzidine**
			61. chrysene-d12 (IS)*
			62. chrysene
			63. bis(2-ethylhexyl)phthalate
			64. di-*n*-octyl phthalate
			65. benzo(b)fluoranthene
			66. benzo(k)fluoranthene
			67. benzo(a)pyrene
			68. perylene-d12 (IS)*
			69. indeno(1,2,3-cd)pyrene
			70. dibenzo(a,h)anthracene
			71. benzo(ghi)perylene

*20ppm **50ppm

Rtx®-5Sil MS 30m, 0.25mm ID, 0.25µm (cat.# 12723)

Sample:	1µLof a combination of cat.# 31812 (10µg/mL), cat.# 31813 (50µg/mL), cat.# 31026 (50µg/mL), cat.# 31206 (20µg/mL), cat.# 31211 (10µg/mL). 10ppm each component on-column, except as noted.
Inj.:	splitless (hold 0.4 min.), Drilled Uniliner® (cat.# 20756)
Inj. Temp.:	275°C
Carrier Gas:	helium, constant flow
Flow Rate:	1mL/min.
Oven Temp.:	35°C (hold 1 min.) to 260°C @ 20°C/min. to 330°C @ 6°C/min. (hold 1 min.)
Det:	MS
Transfer Line Temp.:	280°C
Scan Range:	35–550amu
Ionization:	EI
Mode:	scan

Fortification Mix (7 components)

4,6-dinitro-2-methylphenol	4-nitroaniline
2,4-dinitrophenol	4-nitrophenol
2-nitroaniline	2,4,5-trichlorophenol
3-nitroaniline	

2,000µg/mL each in methylene chloride, 1mL/ampul

Each	5-pk.	10-pk.
31813	31813-510	—
	w/data pack	
31813-500	31813-520	31913

OLC 03.2 SVOA Deuterated Monitoring Compounds (DMC) (16 components)

acenaphthylene-d8	4,6-dinitro-methylphenol-d2
anthracene-d10	fluorene-d10
benzo(a)pyrene-d12	4-methylphenol-d8
4-chloroaniline-d4	nitrobenzene-d5
bis-(2-chloroethyl)ether-d8	2-nitrophenol-d4
2-chlorophenol-d4	4-nitrophenol-d4
2,4-dichlorophenol-d3	phenol-d5
dimethylphthalate-d6	pyrene-d10

2,000µg/mL each in methylene chloride, 1mL/ampul

Each	5-pk.
31810	31810-510

OLC 03.2 SV MegaMix™ (57 components)

Components listed in Figure 1 (bold)

1,000µg/mL each in methylene chloride:benzene (75:25) (3-methylphenol & 4-methylphenol at 500µg/mL), 1mL/ampul

Each	5-pk.	10-pk.
31812	31812-510	—
	w/data pack	
31812-500	31812-520	31912

For other reference mixes, refer to our catalog or web site.

Rtx®-5Sil MS Column (fused silica)

ID	df (µm)	temp. limits	30-Meter
0.25mm	0.25	-60 to 330/350°C	12723

New Semivolatiles Reference Mix for Wastewater Analysis

by Gas Chromatography/Mass Spectrometry

By Katia May, Ph.D., R&D Chemist, Chris English, Environmental Innovations Chemist, and John Lidgett, Analytical Reference Materials Manager

- ✔ New MegaMix™ formulation of all 54 target compounds, for fast preparation of working solutions.
- ✔ Mix includes "additional" extractable Method 625 compounds.
- ✔ Inert, low-bleed Rtx®-5Sil MS column ensures fast analysis, reliable data.

US EPA Method 625 is a GC/MS method applicable to analysis of organic compounds in water and soil. The sample is serially extracted with methylene chloride at pH >11, then at pH <2; the extract is dried, concentrated to 1mL, and analyzed.

Method 625 is appropriate for several classes of chemicals: phenols, benzidines, phthalate esters, polyaromatic hydrocarbons, chlorinated pesticides, toxaphene, and Aroclor® PCBs. Our new calibration mix, Semivolatiles MegaMix™, EPA Method 625, combines many of these analytes in a single mix, for faster and more convenient preparation. For completeness, we also include target compounds listed as "additional" in Method 625. The mixture has been formulated carefully, to ensure maximum stability, and two independently prepared lots are available. The components of the mix are listed in Figure 1.

Some of the target compounds in Method 625 are subject to thermal or chemical degradation in the heated GC injection port. The most labile compound, N-nitrosodiphenylamine, totally decomposes to diphenylamine at the GC inlet. In formulating the new MegaMix™ we have taken steps to compensate for the degradation problem. For example, we include the compound that is analyzed, diphenylamine, rather than the parent compound, N-nitrosodiphenylamine, in the mix.

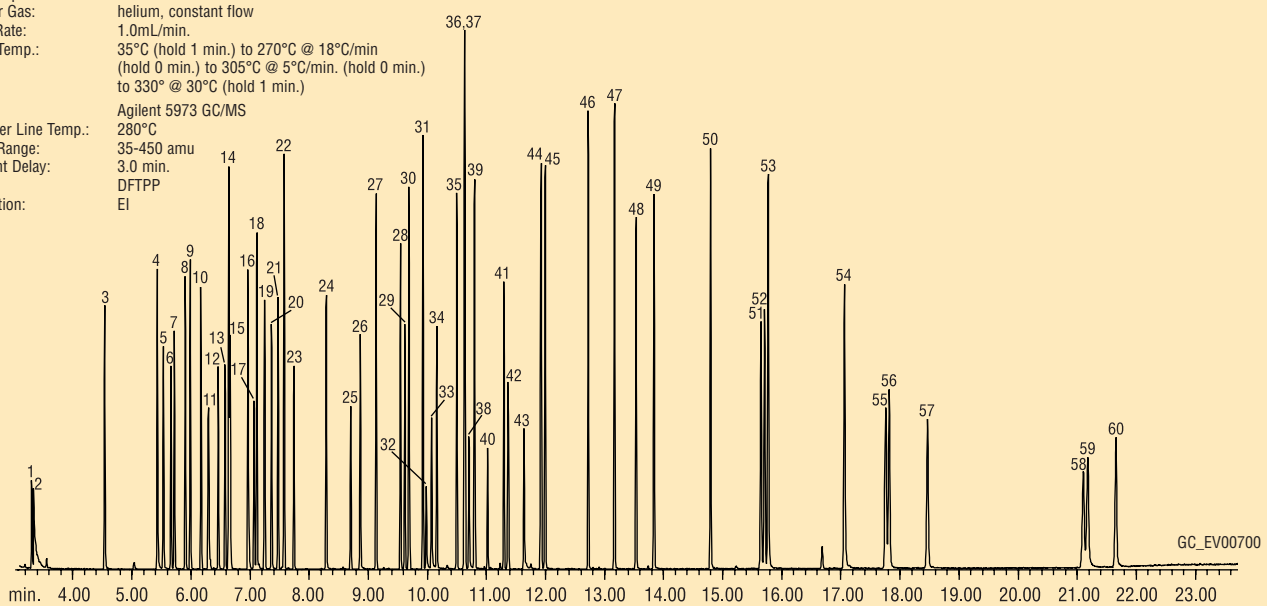
Hexachlorocyclopentadiene, pentachlorophenol, 2,4-dinitrophenol, and other compounds will degrade to varying degrees on contact with active sites in the injection port. To minimize this problem, we use a Drilled Uniliner® inlet liner to prevent the sample from coming into contact with the metal surface below the inlet sleeve. The end of the column seals against the tapered end of the Drilled Uniliner® inlet liner, and the sample is channeled directly from the liner into the column, eliminating the most active portion of the sample flow path. A hole in the side of the liner allows carrier gas to flow normally during split/splitless injections.

Method 625 calls for a column that exhibits low bleed, in addition to providing adequate analyte separation. We recommend a 30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS column (cat.# 12723) — these columns are characterized by effective selectivity for the target analytes, low bleed, excellent inertness, and a high maximum operating temperature. A 30m, 0.25mm ID, 0.25µm column separates critical analyte pairs in less than 22 minutes (Figure 1).

Figure 1 - Method 625 semivolatile analytes resolved for mass spectrometry, using an Rtx®-5Sil MS column.

Rtx®-5Sil MS 30m, 0.25mm ID, 0.25µm (cat.# 12723)

Sample:	US EPA Method 625 Mix 1µL 10ppm (20ppm IS) Standards used: cat. #s 31048, 31044, 31039, 31047, 31046, 31040, 31829
Inj.:	1.0µL 20psi 0.3 min. pulsed splitless (hold 0.3 min.), 4mm Drilled Uniliner® (cat.# 21055)
Inj. Temp.:	300°C
Carrier Gas:	helium, constant flow
Flow Rate:	1.0mL/min.
Oven Temp.:	35°C (hold 1 min.) to 270°C @ 18°C/min (hold 0 min.) to 305°C @ 5°C/min. (hold 0 min.) to 330° @ 30°C (hold 1 min.)
Det:	Agilent 5973 GC/MS
Transfer Line Temp.:	280°C
Scan Range:	35-450 amu
Solvent Delay:	3.0 min.
Tune:	DFTPP
Ionization:	EI



1. N-nitrosodimethylamine
2. pyridine-d5 (SS)
3. 2-fluorophenol (SS)
4. pentafluorophenol (IS)
5. phenol
6. bis(2-chloroethyl)ether
7. 2-chlorophenol
8. 1,3-dichlorobenzene
9. 1,4-dichlorobenzene
10. 1,2-dichlorobenzene
11. bis(2-chloroisopropyl)ether
12. N-nitroso-di-*n*-propylamine
13. hexachloroethane
14. nitrobenzene-d5 (IS)
15. nitrobenzene
16. isophorone
17. 2-nitrophenol
18. 2,4-dimethylphenol
19. bis(2-chloroethoxy)methane
20. 2,4-dichlorophenol
21. 1,2,4-trichlorobenzene
22. naphthalene
23. hexachlorobutadiene
24. 4-chloro-3-methylphenol
25. hexachlorocyclopentadiene
26. 2,4,6-trichlorophenol
27. 2-chloronaphthalene
28. dimethylphthalate
29. 2,6-dinitrotoluene
30. acenaphthylene
31. acenaphthene
32. 2,4-dinitrophenol
33. 4-nitrophenol
34. 2,4-dinitrotoluene
35. diethyl phthalate
36. fluorene
37. 4-chlorophenyl phenyl ether
38. 4,6-dinitro-2-methylphenol
39. diphenylamine
40. 4,4'-dibromooctafluorophenol (SS)
41. 4-bromophenyl phenyl ether
42. hexachlorobenzene
43. pentachlorophenol
44. phenanthrene
45. anthracene
46. di-*n*-butylphthalate
47. 4,4'-dibromobiphenyl (IS)
48. fluoranthene
49. pyrene
50. butyl benzyl phthalate
51. benzo(a)anthracene
52. chrysene
53. bis(2-ethylhexyl)phthalate
54. di-*n*-octyl phthalate
55. benzo(b)fluoranthene
56. benzo(k)fluoranthene
57. benzo(a)pyrene
58. indeno(1,2,3,-cd)pyrene
59. dibenzo(a,h)anthracene
60. benzo(ghi)perylene

Rtx®-5Sil MS columns are especially well suited for Method 625. Extracts of environmental samples analyzed using this method commonly contain high molecular weight contaminants, and high bake-out temperatures are needed to remove these from the column. The Rtx®-5Sil MS stationary phase and the proprietary deactivation used to prepare these columns can reliably withstand sustained temperatures of 330ºC, and will not exhibit the activity that often is caused by subjecting a column to these conditions. Analysts using Rtx®-5Sil MS columns can program their GCs to 350ºC for especially persistent contamination. Figure 1 shows Restek analytical reference materials analyzed on an Rtx®-5Sil MS column programmed to 330ºC. Bleed is negligible, relative to the 10ng on-column concentration of the target analytes, such as benzo(ghi)perylene.

To obtain Figure 1, conditions were optimized to achieve the fastest analysis without sacrificing resolution of analytes that share quantification ions, such as benzo(b)fluoranthene and benzo(k)fluoranthene (peaks 55 & 56). Steps were taken to focus N-nitrosodimethylamine. To minimize tailing, the pulsed-splitless hold-time was reduced to 0.3 minutes to reduce the time the analytes spend in the injection port. This step also sends a higher percentage of solvent to the split vent, allowing resolution of the amine from the solvent. The initial temperature, 35ºC, allows better analyte focus at the column inlet. The pulsed pressure was reduced from 30psi to 20psi to allow even transfer of N-nitrosodimethylamine to the column. Finally, the flow rate was reduced from 1.1mL/min. to 1.0mL/min. Analytes present at higher concentrations will exhibit less tailing at the lower rate.

Semivolatiles MegaMix™, EPA Method 625

(54 components)

acenaphthene	di-n-butylphthalate
acenaphthylene	4,6-dinitro-2-methylphenol
anthracene	2,4-dinitrophenol
benzo(a)anthracene	2,4-dinitrotoluene
benzo(a)pyrene	2,6-dinitrotoluene
benzo(b)fluoranthene	di-n-octylphthalate
benzo(ghi)perylene	diphenylamine*
benzo(k)fluoranthene	fluoranthene
benzyl butyl phthalate	fluorene
bis(2-chloroethoxy)methane	hexachlorobenzene
bis(2-chloroethyl)ether	hexachloro-1,3-butadiene
bis(2-chloroisopropyl)ether	hexachlorocyclopentadiene*
bis(2-ethylhexyl)phthalate	hexachloroethane
4-bromophenyl phenyl ether	indeno(1,2,3-cd)pyrene
4-chloro-3-methylphenol	isophorone
2-chloronaphthalene	naphthalene
4-chlorophenyl phenyl ether	nitrobenzene
2-chlorophenol	2-nitrophenol
chrysene	4-nitrophenol
dibenzo(a,h)anthracene	N-nitrosodimethylamine*
1,2-dichlorobenzene	N-nitroso-di-n-propylamine
1,3-dichlorobenzene	pentachlorophenol
1,4-dichlorobenzene	phenanthrene
2,4-dichlorophenol	phenol
diethylphthalate	pyrene
2,4-dimethylphenol	1,2,4-trichlorobenzene
dimethylphthalate	2,4,6-trichlorophenol

1,000µg/mL each in methylene chloride:benzene (75:25), 1mL/ampul

Each	5-pk.	10-pk.
31829	31829-510	—
	w/data pack	
31829-500	31829-520	31929

**Listed as an "additional" compound in Method 625 (diphenylamine is included in this mix in place of unstable N-nitrosodiphenylamine). The six other "additional" compounds are components in other Restek reference mixes used for Method 625: benzidine is included in cat.# 31030; β-BHC, δ-BHC, endosulfan I, endosulfan II, endrin are in cat.# 32291.*

SV Internal Standard Mix

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

Each	5-pk.	10-pk.
2,000µg/mL each in methylene chloride, 1mL/ampul		
31206	31206-510	—
	w/data pack	
31206-500	31206-520	31306
*4,000µg/mL each in methylene chloride, 1mL/ampul***		
31006	31006-510	—
	w/data pack	
31006-500	31006-520	31106

***Requires special handling (warming and sonication) before use.*

605 Benzidines Calibration Mix

benzidine 3,3'-dichlorobenzidine

2,000µg/mL each in methanol, 1mL/ampul

Each	5-pk.	10-pk.
31030	31030-510	—
	w/data pack	
31030-500	31030-520	31130

Rtx®-5Sil MS Column (fused silica)

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

ID	df (µm)	temp. limits	30-Meter
0.25mm	0.25	-60 to 330/350°C	12723

Let us create the right solution for you!

We can make ***CUSTOM MIXTURES*** to meet your specific compound lists.

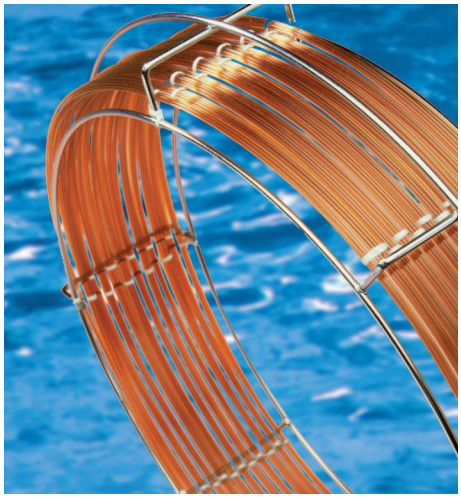
Call us at 800-356-1688 or 814-353-1300 or use the Custom Reference Materials Request Form on our website:

www.restekcorp.com

Rtx®-5Sil MS Columns: Ideal for Semivolatiles Extracts!

- ✔ Excellent low bleed column for semivolatile pollutants, pesticides, PCBs, other environmental applications.
- ✔ Thermally stable to 350ºC.
- ✔ Silarylene phase with polarity similar to 5% diphenyl/95% dimethyl polysiloxane.

The silarylene stationary phase in Rtx®-5Sil MS columns incorporates phenyl rings into the polymer backbone. This improves thermal stability and makes the phase less prone to oxidative degradation, significantly reducing bleed. Rtx®-5Sil MS columns are ideal for use with GC/MS systems, including ion trap systems. The 0.28mm ID columns increase sample capacity, relative to 0.25mm ID columns, without significant loss in resolution. We recommend them for environmental analyses.



Rtx®-5Sil MS Columns (fused silica)

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

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

For Rtx®-5Sil MS columns of other IDs, refer to our chromatography supplies catalog, or visit our website.

Highly Base-Deactivated HPLC Columns from Restek

Pinnacle™ DB Columns: the Performance of Popular BDS Columns, with Plus 1™ Quality and Service

By Greg France, HPLC Products Marketing Manager, and Vernon Bartlett, HPLC Innovations Team Manager

- ✔ Performance-equivalent replacement for Hypersil® BDS columns.
- ✔ In stock, ready to ship.
- ✔ Stock columns in analytical to prep scale.

When we set out to create a new base-deactivated silica, we had five specific goals:

- A material that would match or exceed the performance of any base-deactivated "Type B" silica.
- A material that would provide chromatographic separations equivalent to Hypersil® BDS silica.
- A material with low metals content, for sharp, symmetric peaks for basic analytes.
- A material capable of long lifetime in challenging HPLC environments.
- An effective and efficient manufacturing process, to ensure columns are always available.

Pinnacle™ DB silica meets all of these goals.

Pinnacle™ DB silica performs as well as any base-deactivated Type B silica, or better, and closely matches the desirable physical characteristics of Hypersil® BDS silica. For certain physical parameters we intentionally deviated from Hypersil® BDS material — total metals content, for example. Fewer metal ions on the surface of Pinnacle™ DB silica particles ensure sharper and more symmetric peaks for basic analytes.

Of course, what really matters is how separations on columns made with the two silicas compare. Figure 1 pairs chromatograms for a neutral/base test mix from a C18 bonded phase Pinnacle™ DB column and a Hypersil® BDS column, obtained using the same instrument, mobile phase, and conditions. Retention, peak shape, and efficiency are nearly identical in the two chromatograms.

If physical data and chromatographic comparisons show Pinnacle™ DB and Hypersil® BDS materials are very closely matched, why use Pinnacle™ DB columns? In brief: competitive prices, fast delivery, and unsurpassed Restek service. Why settle for less? But, we want you to be completely comfortable in evaluating our columns. With that in mind, we *guarantee* separations of your samples on Pinnacle™ DB columns will be comparable to separations on Hypersil® BDS columns. If a Pinnacle™ DB column does not meet your satisfaction, simply send us copies of chromatograms for your application on the Pinnacle™ DB column and on a Hypersil® BDS column. We will credit your account for the price of the Pinnacle™ DB column, and you can keep the column - free!

The Pinnacle™ DB line currently includes C18, C8, and cyano bonded phases, and bare silica. Example applications for each of the bonded phase columns, including fast LC analysis, and Pinnacle™ DB / Hypersil® BDS comparisons, are shown in *Pinnacle™ DB HPLC Columns as Replacements for Hypersil® BDS*, Restek lit. cat.# 59742, available on request.

If you need a second source for Hypersil® BDS column performance, if you want a rugged, high quality, base-deactivated material, or if you simply are looking for a reliable supplier who provides columns quickly, Pinnacle™ DB columns are your answer. Of course, as always, you'll get Plus 1™ service and prompt, expert technical help when you deal with Restek.

Figure 1- Pinnacle™ DB columns and Hypersil® BDS columns provide nearly identical retention, peak symmetry, and efficiency.

Pinnacle™ DB C18 Column

Hypersil® BDS C18 Column

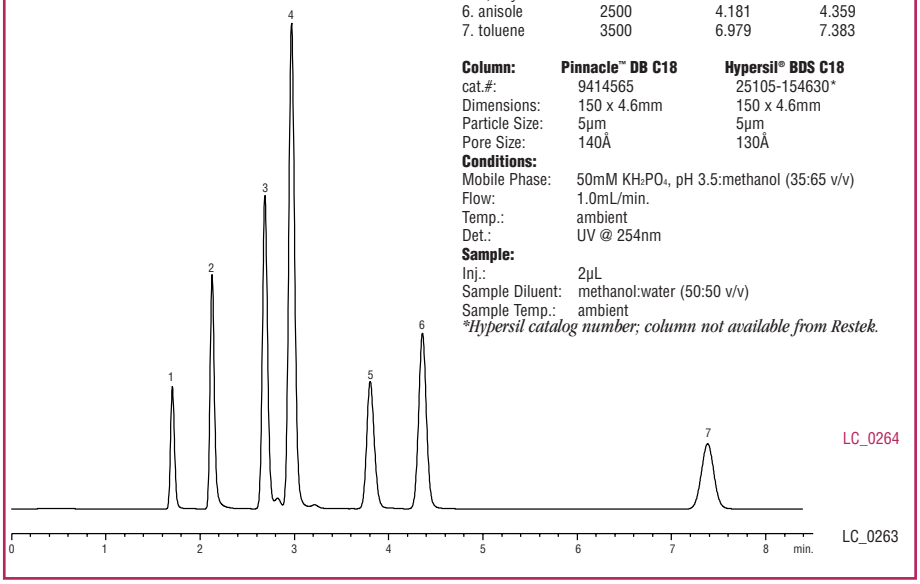
Peak List	Conc. (µg/mL)	Retention Time (min.) Pinnacle™ DB	Hypersil® BDS
1. uracil	50	1.676	1.706
2. aniline	750	2.077	2.127
3. 2-nitroaniline	350	2.618	2.687
4. 2,4-dinitroaniline	350	2.909	2.970
5. 3,5-xylenol	2500	3.668	3.804
6. anisole	2500	4.181	4.359
7. toluene	3500	6.979	7.383

Column:	Pinnacle™ DB C18	Hypersil® BDS C18
cat.#:	9414565	25105-154630*
Dimensions:	150 x 4.6mm	150 x 4.6mm
Particle Size:	5µm	5µm
Pore Size:	140Å	130Å

Conditions:
Mobile Phase: 50mM KH_2PO_4, pH 3.5:methanol (35:65 v/v)
Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 254nm

Sample:
Inj.: 2µL
Sample Diluent: methanol:water (50:50 v/v)
Sample Temp.: ambient

**Hypersil catalog number; column not available from Restek.*



Pinnacle™ DB C18 Column (USP L1)

(5µm silica, 4.6mm ID)

Length	cat.#
150mm	9414565

For the complete selection of Pinnacle™ DB Columns, visit our website, **www.restekcorp.com,** or request **Applications Note 59742**.

More information and additional Pinnacle™ DB / Hypersil® BDS comparisons are included in **Applications Note 59742** *(free on request).*

HPLC Column Selection Guide (lit. cat.# 59454A)

A useful chart to keep with your workbooks, or post on a wall. Quickly scan important characteristics of Restek HPLC columns. Includes brief, practical guidelines for choosing stationary phase, particle size, pore diameter, and column dimensions.

New Reference Materials

Purge and Trap Analysis – Environmental – Forensics

By Katia May, Ph.D., R&D Chemist

Antifoam Agent for Purge & Trap Samples

- ✔ Efficiently controls foam; effective over a wide pH range.
- ✔ Add an antifoaming agent, rather than diluting the sample, to ensure lower detection limits.
- ✔ Effective at less than 0.1% of sample volume.

Methods for monitoring volatile organic compounds (VOC) in waste streams, such as US EPA methods 624 and 8260, involve purge & trap techniques. Often, foam is generated as the purge gas passes through the sample. Foam can enter the analytical trap and potentialy be carried into the GC column. Silica/silicone antifoam agents easily control polyglymes and alkaline detergent-based foams, the most commonly encountered foams in these analyses. Our new silica-containing antifoam agent generally is effective at very low concentration: 1µL per 5mL.[1] By eliminating the need to dilute samples, it allows lower detection limits.

1 Good laboratory practices call for assaying an antifoam blank as a control.

Antifoam Agent for Purge & Trap Samples

Neat, 1mL/ampul

Each	5-pk.
31822	31822-510

Explosives Standard

- ✔ Propylene glycol dinitrate (PGDN), the main component of Otto Fuel II for torpedoes and other weapons.
- ✔ Convenient 1,000µg/mL concentration.

PGDN is very similar to nitroglycerin, but is significantly safer to handle, and can be stored for long periods of time. Nonetheless, the material is an environmental hazard; the National Institute for Occupational Safety and Health (NIOSH) recommended limit of exposure to airborne PGDN is 0.05ppm over a 40-hour work week. Otto Fuel II is in at least 2 National Priorities List sites identified by the US EPA. PGDN enters the environment primarily in wastewater from naval facilities.

Many of our customers have asked us to include PGDN among our stock offerings. We are now able to comply with this request.

PGDN Standard (Propylene Glycol Dinitrate)

1,000µg/mL in methanol, 1mL/ampul

Each	5-pk.	10-pk.
31821	31821-510	—
	w/data pack	
31821-500	31821-520	31921

Bank Dye Standard

- ✔ Qualitative standard of red dye used to thwart bank robberies.

The dye pack or "security pack" used in over 75% of the banks in the United States contains a red dye, 1-N-methylaminoanthraquinone (MAAQ). The dye pack, which is activated when it is taken outside the bank, explodes and releases an aerosol of red smoke, and burns at a temperature of about 400ºF. When events go as planned, the thief discards the bag, the money is recovered, and stained hands and clothes expose the robber. The dye pack has contributed to the recovery of $20 million in stolen money and apprehension of 2500 criminals. Restek offers this new qualitative standard to help investigators in municipal police stations and criminal laboratories fight crime.

Bank Dye Standard (MAAQ)

1-(methylamino)anthraquinone

100µg/mL in methylene chloride, 1mL/ampul

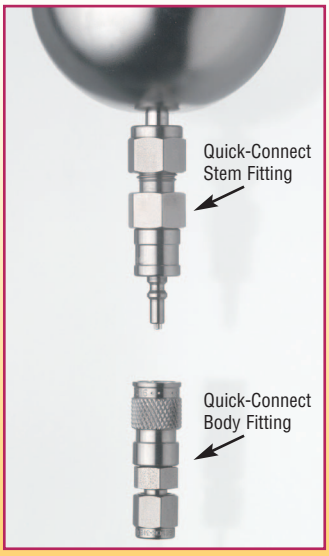
Each	5-pk.	10-pk.
31823	31823-510	31923

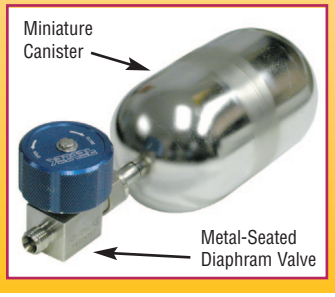
Miniature Air Sampling Canisters

- ✔ 400cc — ideal for indoor air, personal, emergency response, or soil gas sampling.
- ✔ Available with quick-connect fitting that is compatible with sampling and analysis instruments.
- ✔ Also available with non-treated or Sulfinert™-treated valve.

These small canisters are designed for controlled sampling, such as personal air sampling, as an alternative to tube and pump samplers.

Restek offers these products in stainless steel or with Sulfinert™ coating for greatest inertness. We continue to offer passive coating technologies that are unmatched in the air sampling industry—try a Sulfinert™-treated canister and achieve the ultimate in analyte stability.





Miniature Air Sampling Canisters with Quick-Connect Stem Fittings

Description	Volume	qty.	cat.#
Electro-Polished Miniature Canister with Quick-Connect Stem Fitting	400cc	ea.	24188
Sulfinert™-Coated Miniature Canister with Quick-Connect Stem Fitting	400cc	ea.	24189
Sulfinert™-Coated Miniature Canister with Sulfinert™-Treated Quick-Connect Stem Fitting	400cc	ea.	24190

Miniature Air Sampling Canisters with Metal-Seated Diaphragm Valve

Description	Volume	qty.	cat.#
Electro-Polished Miniature Canister with Metal-Seated Diaphragm Valve	400cc	ea.	24191
Sulfinert™-Coated Miniature Canister with Metal-Seated Diaphragm Valve	400cc	ea.	24192
Sulfinert™-Coated Miniature Canister with Sulfinert™-Treated Diaphragm Valve	400cc	ea.	24193

Fittings for Miniature Air Sampling Canisters

Description	qty.	cat.#
Quick-Connect Stem Fitting	ea.	24185
Sulfinert™-Treated Quick-Connect Stem Fitting	ea.	24186
Quick-Connect Body Fitting	ea.	24187

Canisters Optimized for Air Sampling by EPA Methods TO-14 and TO-15

By Donna Lidgett, Air Sampling Products Marketing Manager

- ✔ SUMMA® canister equivalent.
- ✔ Excellent analyte recovery — even after 14 days of storage.

Feature	Benefit
High-purity, ⅔-turn valve with stainless steel diaphragms.	No sample adsorption, for more accurate results; easy to use.
Vacuum/pressure gauge (optional).	Indicates internal conditions.
Variety of sizes.	Meet a range of sampling needs.
Temperature stability to 250°C.	Higher temperature cleaning saves time.

US Environmental Protection Agency (EPA) Compendium of Air Methods TO-14 and TO-15 regulate the collection, storage, and analysis of volatile organic compounds (VOCs) using treated air sampling canisters.

Restek offers a line of TO-Can™ canisters (SUMMA® can equivalent), which are electropolished and extensively cleaned using proprietary processes. This ensures a high-quality, passivated surface to maintain stability of the TO-14/TO-15 compounds during storage. The design of the frame surrounding the electropolished canister eliminates welds on the sphere, thereby eliminating active sites. A Parker Hannifin metal-to-metal diaphragm valve further improves the performance of the canister.

To collect VOCs in ambient air, TO-Can™ canisters should be cleaned and evacuated prior to being sent to the field. In the field, the sample is drawn through a sampling train that regulates the rate and duration of sampling. The TO-Can™ canister is then sent to the analytical laboratory. In the laboratory, a known amount of sample is drawn from the canister and concentrated onto a trap. The sample is analyzed according to Method TO-14/TO-15, typically using a 60m, 0.32mm ID, 1.0µm Rtx®-1 capillary column in a GC/MS system.

To show the inertness of these canisters, and how well they meet the holding time criteria for Methods TO-14/15, a 62-component TO-15 standard (10ppbv) was injected into a TO-Can™ canister and humidified to 70% relative humidity. The standard was analyzed on day 1, day 7, and day 14. The TO-Can™ canister ensured excellent stability for these polar and non-polar compounds, even after 14 days of storage (for data, request lit. cat.# 59189). We also offer sampling kits, sampling bags, thermal desorption tubes, and a range of gas reference standards to meet your environmental gas sampling requirements.

For more information, request our Air Monitoring Products Catalog (lit. cat. #59661A).

For information about environmental gas reference mixes, request lit. cat.#59276A.

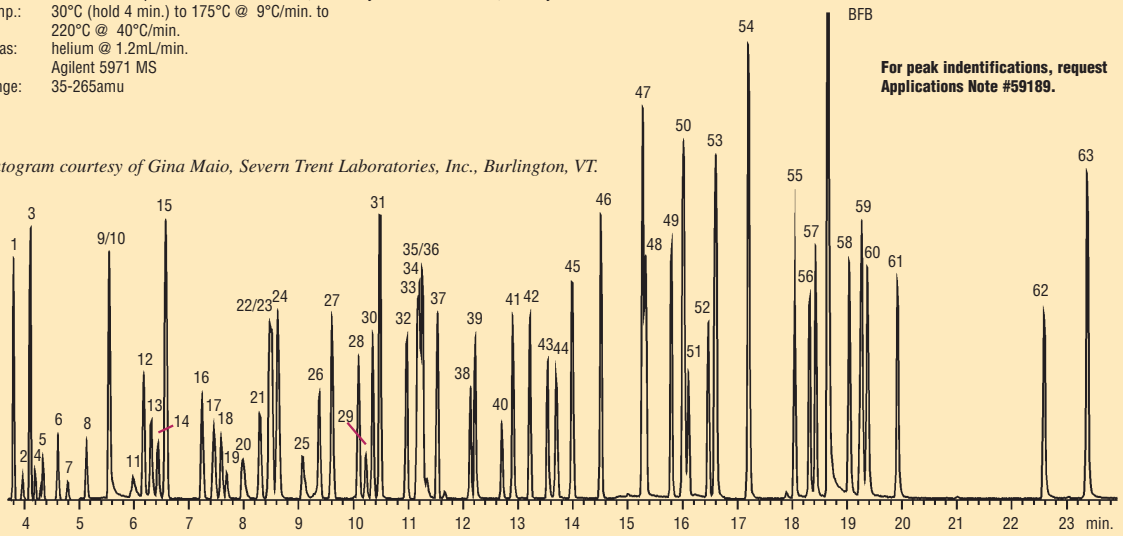
Figure 1 - TO-Can™ canisters ensure excellent recovery for TO-14/TO-15 compounds, even after 14 days of storage.

Rtx®-1 column, 60m, 0.32mm ID, 1.0µm (cat.# 10157).

Sample: 200mL of 10ppbv TO-15 standard (cat.# 34436), injected into TO-Can™ canister and humidified to 70% RH.
Concentrator: Nutech 3550 Preconcentrator
200mL of sample concentrated at 160°C, thermally desorbed at 150°C, and cryofocused at 185°C.
Oven Temp.: 30°C (hold 4 min.) to 175°C @ 9°C/min. to 220°C @ 40°C/min.
Carrier Gas: helium @ 1.2mL/min.
Det.: Agilent 5971 MS
Scan Range: 35-265amu

For peak indentifications, request Applications Note #59189.

Chromatogram courtesy of Gina Maio, Severn Trent Laboratories, Inc., Burlington, VT.



GC_EV00379

TO-Can™ Canisters for EPA Methods TO-14 and TO-15

volume	qty.	cat.#
1L	ea.	24150
3L	ea.	24152
6L	ea.	24153
15L	ea.	24154

TO-Can™ Canisters with Vacuum/Pressure Gauge

volume	qty.	cat.#
1L	ea.	24155
3L	ea.	24156
6L	ea.	24157
15L	ea.	24158

SilcoCan™ Canisters

Ideal for Low-Level (1ppb-20ppb) Reactive Sulfur Compounds

By Donna Lidgett, Air Sampling Products Marketing Manager

- ✔ Stable, long-term storage of sulfur volatile organic compounds.
- ✔ More accurate data than with electropolished canisters.

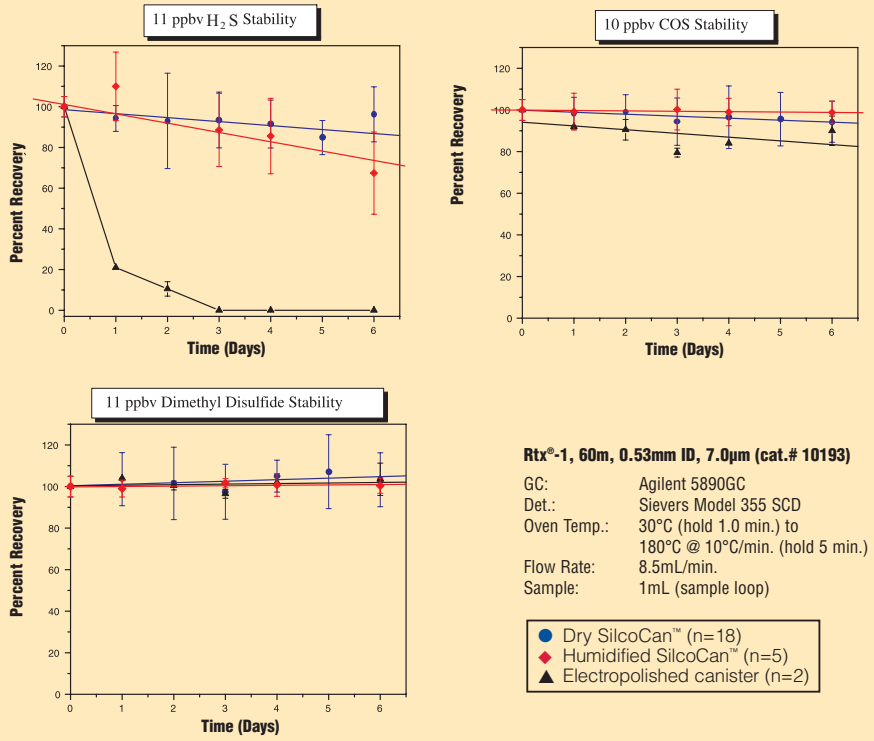
Feature	Benefit
Silcosteel® coated.	Inert surface, ideal for containing low-level sulfur compounds.
High-purity, ⅔-turn valve with stainless steel valve diaphragms.	No sample adsorption, for more accurate results; easy to use.
Vacuum/pressure gauge (optional).	Indicates internal conditions.
Variety of sizes.	Meet extensive range of sampling needs.
Temperature stability to 250°C.	Can be cleaned at higher temperature, producing a cleaner can.
Silcosteel® valve available (add suffix "-650" to cat. #).	Completely passive sample pathway ensures sample stability.

Analysis of low-level sulfur volatile organic compounds (VOCs), such as hydrogen sulfide (H_2S), methyl mercaptan (CH_3SH), ethyl mercaptan (C_2H_5SH), and dimethyl disulfide (CH_3SSCH_3) is important because of health concerns and odor complaints near manufacturing sites and refineries. Collection and measurement of these compounds in the atmosphere is very difficult because of their low concentrations and high reactivity. Sulfur VOCs can react not only with each other, but also with the vessels in which they are collected. This causes low recoveries. SilcoCan™ air monitoring canisters, which feature a Silcosteel®-treated surface, ensure stability of low-level sulfur VOCs.

We evaluated the stability of sulfur VOCs in SilcoCan™ canisters at very low levels (1–20ppbv) for six days.[1] Comparison of dry vs. humidified standards demonstrates the ability of SilcoCan™ canisters to store low-level sulfur VOCs in real-world conditions (Figure 1). Electropolished canisters allowed rapid degradation of hydrogen sulfide, methyl mercaptan, and ethyl mercaptan during a similar study.

When you need to perform sensitive air monitoring analyses for sulfur VOCs, use SilcoCan™ canisters to collect and store your samples.

Figure 1 - SilcoCan™ canisters effectively store low-level organic sulfur compounds under real-world conditions.



Standards: 2mL of 100ppm stock sulfur standard added to each precleaned and evacuated canister, then pressurized to 30psig with ultra-pure nitrogen. The resultant concentrations are listed in Applications Note #59347. Humidified standards made by injecting evacuated canisters with 100µL of deionized water prior to adding 2mL aliquot of stock standard (50% RH).

SilcoCan™ Air Sampling Canisters

volume	qty.	cat.#
1L	ea.	24112
3L	ea.	24113
6L	ea.	24114
15L	ea.	24115

SilcoCan™ Canisters with Vacuum/Pressure Gauge

1L	ea.	24116
3L	ea.	24117
6L	ea.	24118
15L	ea.	24119

Our 20-page technical guide describes the components of a passive sampling train, and presents procedures for sampling, for cleaning, and for certifying the sampling train and canister. **Request lit. cat.# 59977A.**

Reference

[1] *Stability study of Low-Level (1ppb-20ppb) Reactive Sulfurs in SilcoCan™ Canisters.* Restek Corporation, 2001. Available on request: lit. cat.# 59347.

Peak Performers

By Donna Lidgett, GC Accessories Products Marketing Manager

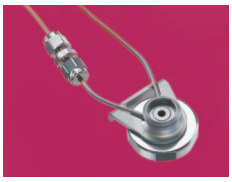
Direct Replacement Split/Splitless Injection Port Weldments for Agilent GCs, for use with Purge and Trap Systems

✔ Easily attach your purge and trap with pre-installed low dead volume fittings.

For Agilent GCs with Tekmar purge and trap systems

Description	qty.	cat.#
Weldment for Agilent 6890 GCs	ea.	22664
Weldment for Agilent 6890 GCs with optional canister filter	ea.	22668
Weldment for Agilent 5890 GCs	ea.	22666

For use with Tekmar purge and trap system

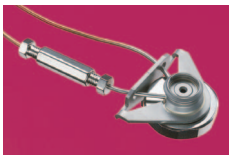


cat.# 22666

For Agilent GCs with OI purge and trap systems

Description	qty.	cat.#
Weldment for Agilent 6890 GCs	ea.	22665
Weldment for Agilent 6890 GCs with optional canister filter	ea.	22669
Weldment for Agilent 5890 GCs	ea.	22667

For use with OI purge and trap system

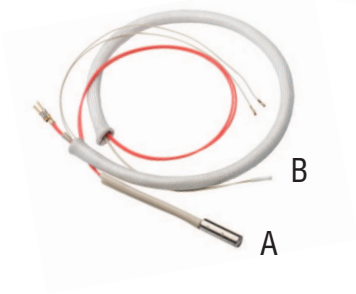


cat.# 22665

Injector/FID Heater & PRT Sensor for Agilent 5890 GCs

✔ Use with 5890 FID and split/splitless weldments.
✔ Meets or exceeds OEM specifications.

Description	Similar to Agilent part #	qty.	cat.#
A+B Injector/FID Heater/PRT Sensor Assembly	05890-61140	ea.	22068
A Injector/FID Heater	19231-60620	ea.	22069
B Injector/FID PRT Sensor	19231-60660	ea.	22070



Heat Sink for Agilent 5890/6890/6850 GC Split/Splitless Injector

✔ Meets or exceeds original equipment manufacturer's specifications.

Description	Similar to Agilent part #	qty.	cat.#
Heat Sink for Agilent 5890/6890/6850 GCs	18740-20940 G1544-20570	ea.	20409

new!

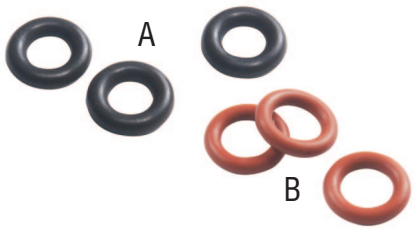
PSS Inlet Liners and O-Ring Liner Seals for PerkinElmer GCs

PSS Liners for PerkinElmer GCs	Benefits/Uses:	ID*/OD & Length (mm)	Similar to PE part #	cat.# ea.	cat.# 5-pk.
PSS Split/Splitless (1mm ID)	trace samples	1.0 ID 4.0 OD x 86.2	N612-1006	20738	20741
Auto SYS® XL PSS Split/Splitless w/ FS Wool	most common analyses	2.0 ID 4.0 OD x 86.2	N612-1004	21717	21718

all liners are deactivated!

**Nominal ID at syringe needle expulsion point.*

Description	Similar to PE part #	Qty.	cat.#
A O-Ring Liner Seals for PerkinElmer PSS	N6101747	10-pk.	20366
B O-Rings Liner Seals for PerkinElmer Auto SYS® GCs	N6101374	10-pk.	20262



For more information: Request our current catalog (lit. cat.# 59473) for a complete listing of our consumables for Agilent, Varian, Shimadzu, PerkinElmer, and Thermo instruments.

cool tools

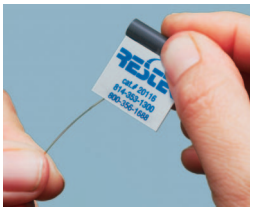
For Easier GC Maintenance Try These Tools from Restek

by Brad Rightnour and Michael Goss, Instrument Innovations Team

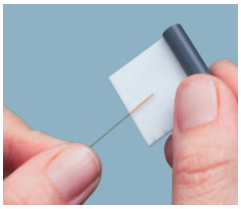
Ceramic Scoring Wafers—Clean, square cuts for better connections

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

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



Hold the scoring wafer at a 45° angle to the tubing. Use gentle pressure and a smooth, perpendicular stroke.



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

Rethreading Tool—Save the cost of replacing expensive injectors

- ✔ Achieve a better seal.
- ✔ Repair worn or damaged threads.
- ✔ Built-in guide, to prevent cross-threading.

Description	qty.	cat.#
Rethreading Tool for 1/16" compression fitting	ea.	23016
Rethreading Tool for 1/8" compression fitting	ea.	23017
Rethreading Tool for 1/4" compression fitting for Agilent split/splitless injection ports	ea.	23018
Rethreading Tool for 7/16" compression fitting for Varian injection ports	ea.	23019
Rethreading Tool for 1/4" Varian-style capillary column fittings new!	ea.	21893

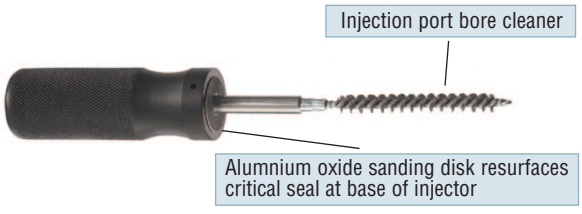
Due to constant installation, removal, and exposure to extreme temperature changes, threads on GC parts become worn and damaged. This can cause a poor seal, and oxygen can enter the system, compromising analytical results and possibly destroying expensive analytical columns.

Screw the rethreading tool completely onto the injection port in a clockwise direction.

Unscrew the rethreading tool and inspect the threads. Repeat as necessary.

Injection Port Repair Tool—Remove contaminants, achieve a better seal

- ✔ Resurfaces critical inlet seal areas.
- ✔ For Agilent split/splitless injection ports.*



Description	qty.	cat.#
Injection Port Repair Tool	ea.	21393
Replacement Sanding Disks (5 fine & 5 medium)	10-pk.	22689

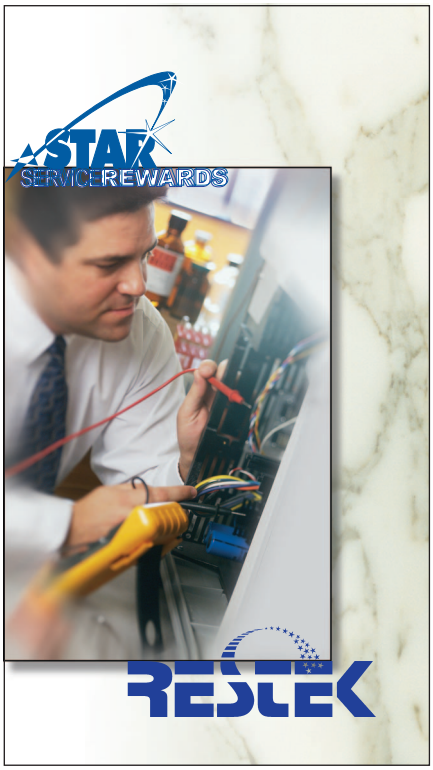
Should not be used on Siltek™-treated injection ports.

The inlet seal at the base of a split/splitless injector forms a seal between the injection port and the inlet liner. This inlet seal wears over time and may become scratched or pitted, which compromises the sealing ability of the injector. Use the Restek injection port repair tool to easily resurface the inlet seal and remove contaminants; it saves time and money by preventing leaks.

Try Our Exclusive

SILTEK™-Treated Injection Ports

For more information, request newly updated *Genuine Restek Replacement Parts for Agilent GCs* (lit. cat.#59627D)



STAR™ Service Rewards Program

Register Your Lab, Start Saving on Service

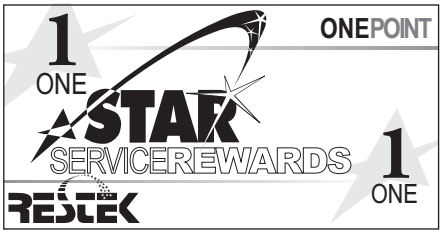
Restek Corporation has formed an alliance with some of the premiere independent instrument service providers in the United States, with a goal of bringing you the finest chromatography operating supplies, equipment service, and applications support available.

We are pleased to introduce a new program that pays you for using Restek products, by reducing your costs for quality instrument service: STAR Service Rewards. Similar to our popular Restek Wizard Dollar program, STAR Service Rewards pays you one STAR Point for every $50 of Restek products you purchase. Redeem STAR Points with participating STAR member service providers for selected service, equipment, and training products. You get the finest chromatography operating supplies from Restek and high quality instrument service from your preferred service provider. STAR Service Rewards is one more example of why Restek is the company chromatographers trust for complete chromatography support.

STAR, the **Service and Technology Alliance by Restek**, is an affiliation of independent instrument service providers, original equipment manufacturers, and instrument remanufacturers, working with Restek to provide chromatographers with the most complete level of support available in the industry.

For more information about the STAR Service Rewards Program, to view the list of member service providers, or to register your lab:

- Call Restek Customer Service at **800-356-1688 ext.3,** or
- Go to the STAR page on the Restek web site—**http://www.restekcorp.com/star**
- Request our STAR flyer, lit. cat.# 59522.



New / Recent Literature

Brochures

Environmental HPLC Applications-Columns-Reference Materials (lit. cat.# 59741)
Gas Purification Essentials (lit. cat.# 59216D)
Miniature Air Sampling Canisters (lit. cat.# 59491)
Passive Air Sampling Kits (lit. cat.# 59290A)

Minicatalog

Genuine Restek Replacement Parts for Agilent GCs (lit. cat.# 59627D)

Technical Guide

Cleaning and Personal Care Products - Gas and Liquid Chromatography (lit. cat.# 59738)

Applications Notes

Single-Column Method for HPLC of Organic Acids in Fruit Juices (lit. cat.# 59530)
Pinnacle™ DB HPLC Columns as Replacements for Hypersil® BDS (lit. cat.# 59742)

Fast Facts

ASTM 2887-01 Simulated Distillation (lit. cat.# 59383A)
Ethanol Reference Materials (lit. cat.# 59382A)
Explosives Reference Materials (lit. cat.# 59381A)
PAHs in Diesel Fuel (lit. cat.# 59384A)
US EPA 8260B Reference Mixes (lit. cat.# 59332A)
UST Monitoring: Alaska (lit. cat.# 59503)
UST Monitoring: California (lit. cat.# 59433)
UST Monitoring: Iowa (lit. cat.# 59504)
Rtx®-1 Capillary GC Columns (lit. cat.# 59308)
ShinCarbon ST Micropacked Columns (lit. cat.# 59519A)

Wall Charts

HPLC Column Selection Guide (lit. cat.# 59454A)
HPLC Technical Tips (lit. cat.# 59894A)

ISO 9001 REGISTERED

Lit. Cat. # 59852

the RESTEK Advantage
Innovators of High Resolution Chromatography Products

High Resolution GC/MS Separations of Dioxin and Furan Congeners

Using Restek's New Rtx®-Dioxin2 Capillary GC Column

Restek Exclusive!

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

- ✔ Resolves 2,3,7,8-substituted congeners from each other and from non-toxic congeners.
- ✔ Resolves furan congeners from chlorodiphenyl ethers.
- ✔ Stable to 320ºC, for longer column life.

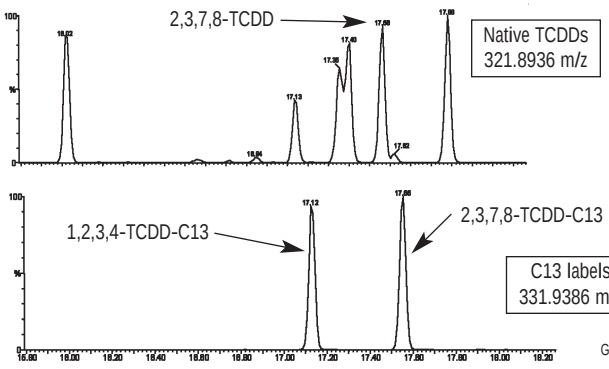
An accurate GC analysis of dioxin and furan congeners is a challenge. Separation of the toxic congeners (configurations with substitutions at the 2, 3, 7, and 8 positions) from the non-toxic congeners is difficult on almost any stationary phase. Most laboratories perform an initial analysis using a 5% diphenyl / 95% dimethyl polysiloxane column (e.g., Rtx®-5) to obtain reasonable estimates of concentrations for the 2,3,7,8-substituted congeners. For some of the target congeners, this quantitation is biased toward high values, due to coelution with non-toxic congeners. As many as five non-toxic TCDFs can coelute with 2,3,7,8-tetrachlorodibenzofuran, for example, in an analysis on a 5% diphenyl / 95% dimethyl polysiloxane (5-type) column.

Because of this coelution issue, many laboratories use confirmation columns, most commonly high cyanopropyl (225-type) stationary phases (e.g., Rtx®-225, Rtx®-2330), in order to more accurately quantify the toxic congeners. Unfortunately, cyanopropyl columns exhibit poor thermal stability, and therefore column lifetimes are short.

Since most methods for analysis of dioxins and furans include extensive sample extract cleanup, and high-resolution mass spectrometry, a primary requirement of the ideal analytical column is complete separation of the toxic dioxin and furan congeners from each other. Additionally, it is desirable for the column to have high thermal stability and long lifetime.

With these characteristics in mind, Restek chemists developed Rtx®-Dioxin2 capillary GC columns. These new columns completely resolve the 2,3,7,8- substituted congeners from each other, and from the non-toxic congeners as well. Figure 1 shows the separation of the tetrachlorodibenzodioxins on a 60m x 0.25mm ID x 0.25µm Rtx®-Dioxin2 column. 2,3,7,8-TCDD is well resolved from the other congeners in this group and can be quantified accurately. The column also is available in an alternative format commonly used for this analysis: 40m x 0.18mm ID x 0.18µm. Either column is stable to 320ºC.

Figure 1—2,3,7,8-Tetrachlorodibenzodioxin can be resolved from other TCDD congeners by using an Rtx®-Dioxin2 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

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

in this issue

RESTEK

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2003 vol. 4

Quantitation of the hexachlorodibenzofurans on Rtx®-5 or equivalent columns, like quantitation of the dioxins, can be made difficult by coelutions of toxic and non-toxic congeners. The new column resolves furan congeners as effectively as dioxins. Figure 2 is a chromatogram for the HCDF congener group in reference material WMS-01; the congeners are very well resolved (reference material courtesy of Wellington Laboratories, Guelph, Ontario, Canada).

Table 1 lists values for 1,2,3,4,7,8-hexachlorodibenzofuran in several reference materials. In analyses on 5-type stationary phases, a number of non-toxic hexafuran congeners can coelute with the toxic 1,2,3,4,7,8-HCDF congener, producing inflated values for 1,2,3,4,7,8-HCDF. In fact, it is generally assumed that in fly ash the actual value for 1,2,3,4,7,8-HCDF is approximately 3-fold less than the value obtained when using a 5-type column. This is one of the reasons confirmation on a high-cyano phase is necessary. As shown in Table 1, the value for 1,2,3,4,7,8-HCDF on an Rtx®-Dioxin2 column is approximately 3-fold less than what was quantified using a 5-type column. The difference is explained by the excellent separation in Figure 2: the lower, more accurate value is due to elimination of coelutions with non-toxic congeners. Values for other congeners compare equally well.

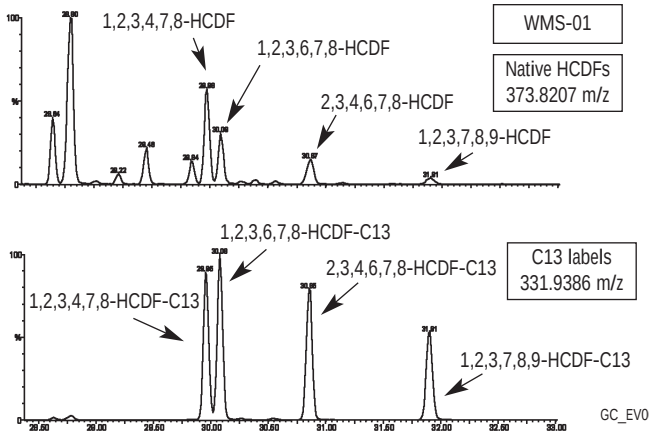
Table 1—An Rtx®-Dioxin2 column gives more accurate quantitation than 5-type columns for 1,2,3,4,7,8-hexachlorodibenzofuran in common matrices (all values pg/g).

	5-type column	Rtx®-Dioxin2 column	Certified Value
Biota-1	nd	nd	
Biota-2	nd	nd	
Sediment	290	210	
Fly ash	570	200	
EC-2 (DX-1)	780	630	714 ±276
NST 1974	nd	nd	

Table 2—Values for 2,3,7,8-tetrachlorodibenzofuran in biota reflect the Rtx®-Dioxin2 column's ability to resolve target compounds from potentially interfering chlorodiphenylethers (all values pg/g; provisional (non-2,3,7,8-TCDF confirmed)).

	Column			
	5-type	225-type	Rtx®-Dioxin2	Certified Value
Biota-1	1	1.3	0.8	
Biota-2	4.3	4.3	2.2	
Sediment	37	19	19	
Fly ash	240	38	32	
EC-2 (DX-1)	88	n/a	37	89 ±44
NST 1974	4.7	n/a	3.3	

Figure 2— Hexachlorodibenzofuran congeners resolved by an Rtx®-Dioxin2 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

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

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

Additional columns for dioxins analysis

Rtx®-Dioxin, 60m, 0.25mm ID, 0.15µm, cat.# 10755, $715
Rtx®-Dioxin, 40m, 0.18mm ID, 0.11µm, cat.# 10756, $655
Rtx®-5, 60m, 0.25mm ID, 0.25µm, cat.# 10226, $705
Rtx®-5MS, 60m, 0.25mm ID, 0.25µm, cat.# 12626, $710

An additional advantage of the Rtx®-Dioxin2 column is its ability to separate the chlorodiphenylethers, commonly found in biota extracts, from the furans. Coelution of these materials is a common problem on both 5% diphenyl / 95% dimethyl stationary phases and cyanopropyl stationary phases, but chromatographic separation is necessary for accurate quantification of the chlorofurans: the chlorodiphenylethers form chlorofurans in the ion source of the mass spectrometer, and therefore cannot be separated spectrally from the target compounds. Table 2 summarizes results from analyzing several matrices for 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF). The values for the biota extracts demonstrate the importance of the furan/chlorodiphenylether separation. Because neither the 5% diphenyl / 95% dimethyl-type column nor the cyanopropyl-type column solves the coelution problem, quantified values for 2,3,7,8-TCDF in biota are high for both columns. The Rtx®-Dioxin2 column separates these compounds, and the quantified values for 2,3,7,8-TCDF, approximately one-half of the values obtained on the other stationary phases for these particular samples, are more accurate values.

If you are involved in analyzing dioxins and furans, and would like detailed information about Rtx®-Dioxin2 columns, we can provide elution orders for all of the commonly analyzed congeners, and chromatograms for each congener group in the WMS-01 reference material. Please contact our Technical Service chemists at 800-356-1688 or 814-353-1300, ext. 4, or contact your Restek representative.

Genuine Restek Replacement Parts for Agilent GCs

(lit. cat.# 59627D) Restek chromatography supplies and accessories—designed by chromatographers, for chromatographers. This 50-page reference manual lists the consumer-replaceable items, supplies, and accessories you need to keep your Agilent GC running at top performance: injector and inlet supplies, detector parts and supplies, gas system components, tools, vials, syringes, and much more. Many items have been designed to save you time or improve your results, and are exclusive to Restek. Many other items are manufactured specifically to duplicate or exceed the performance of the instrument manufacturer's parts.

Analyze Underivatized Chlorophenoxyacid Herbicides by HPLC

Using an Ultra Aqueous C18 Column and New Reference Mixtures

By Rebecca Wittrig, Ph.D., Senior Innovations Chemist, and Katia May, Ph.D., Senior R&D Chemist

- ✔ HPLC eliminates time-consuming derivatizations (required for GC).
- ✔ Ultra Aqueous C18 column has excellent selectivity for chlorophenoxyacid herbicides.
- ✔ New reference mixes for the most widely performed analyses.

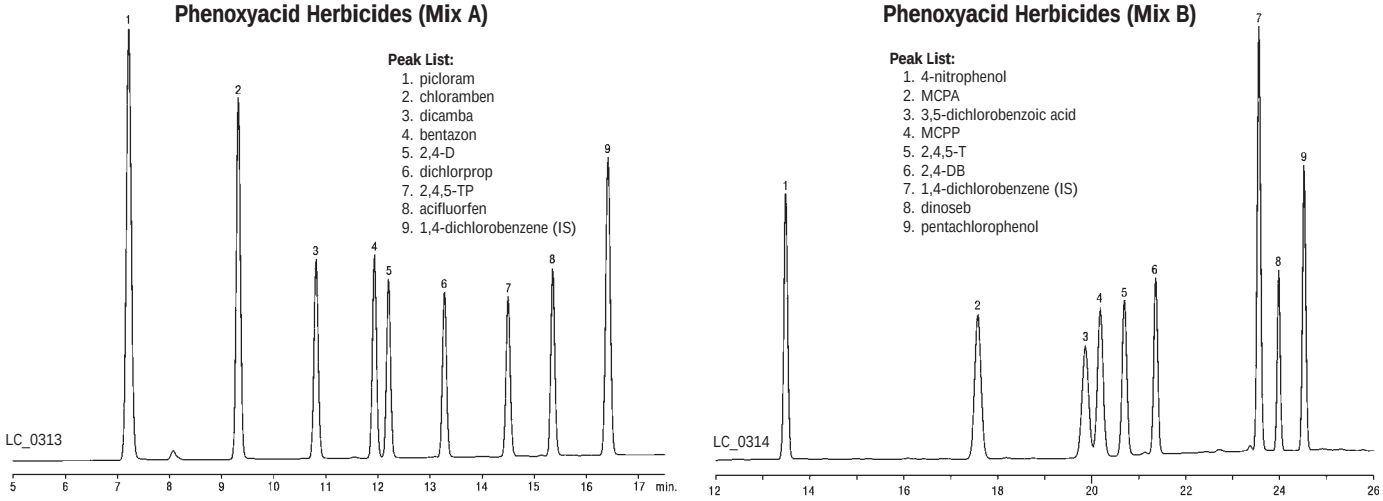
Chlorophenoxyacid herbicides - 2,4-D, dicamba, picloram, Silvex (2,4,5-TP), and others - are used to control agricultural and aquatic weeds. While not considered highly toxic, chlorophenoxyacid herbicides are monitored in agricultural monitoring wells and drinking water sources. They are encountered in the acid form, or as the salts or esters. Traditionally, these compounds have been analyzed by gas chromatography, according to US EPA Method 8151 or other methods. To make them amenable to GC, the acids must be converted to methyl esters, using a derivatizing agent such as diazomethane. High performance liquid chromatography is an attractive option to this lengthy, hazardous procedure. Unlike in the GC procedures, derivatization is not necessary; the analytes can be separated and detected in the free acid form. Comparatively large injection volumes, relative to GC, also make HPLC attractive.

US EPA Method 555 was developed for analysis of chlorophenoxyacid herbicides, in the acid form, in drinking water. To minimize coelutions, the herbicides are divided into two sets. Figure 1 includes a chromatogram for each set, analyzed on an Ultra Aqueous C18 column, using gradient conditions optimized for each analysis. Note that this column has excellent selectivity for resolving these structurally similar compounds. The gradient procedure is useful when analyzing a range of these herbicides; an isocratic mobile phase saves analysis and reequilibration time if samples contain only 2,4-D and Silvex.[1] EPA Method 8321, a general LC/MS or LC/UV method for semivolatile compounds, also includes a discussion of these herbicides.

Restek chemists have formulated a full complement of reference materials for Method 555. New chlorinated acids mixes A and B include all target compounds except 5-hydroxydicamba, a product of dicamba oxidation. Dicamba is stable under normal chromatographic conditions, but strong oxidizers in a sample could convert it to 5-hydroxydicamba and make identification difficult. To avoid this problem, we offer 5-hydroxydicamba in a single, separate solution. We designed these mixes with special consideration for stability, which is a concern because these herbicides, especially in the acid form, are light sensitive and readily degrade in the presence of alkaline substances.

(continued on pg. 5)

Figure 1—Chlorophenoxyacid herbicides are resolved well by an Ultra Aqueous C18 column. HPLC eliminates time-consuming, hazardous derivatizations.



Conditions for Mix A:
Mobile Phase A: 0.05% H_3PO_4
Mobile Phase B: acetonitrile

Time	%B
0	20
15	80
20	80
21	20

Flow: 1.0mL/min
Temp.: ambient
Det.: UV @ 225nm

Conditions for Mix B:
Mobile Phase A: 0.05% H_3PO_4
Mobile Phase B: acetonitrile

Time	%B
0	10
10	45
16	45
22	90
24	90
25	10

Flow: 1.0mL/min
Temp.: ambient
Det.: UV @ 225nm

Column and Sample for both chromatograms:

Column:	Ultra Aqueous C18
Cat. #:	9178565
Dimensions:	150 x 4.6mm
Particle Size:	5µm
Pore Size:	100Å
Sample:	
Inj.:	10µL
Conc.:	10 ppm each herbicide
Sample Diluent:	acetonitrile

Chlorinated Acids by HPLC, Mix A

acifluorfen (Blazer®)	dicamba
bentazon	dichlorprop
chloramben	picloram
2,4-D	2,4,5-TP (Silvex)

1,000µg/mL each in acetonitrile, 1mL/ampul

Each	5-pk.	10-pk.
32431	32431-510	
	w/data pack	
32431-500	32431-520	32531

Chlorinated Acids by HPLC, Mix B

2,4-DB	MCPP (mecoprop)
3,5-dichlorobenzoic acid	4-nitrophenol
dinoseb	pentachlorophenol
MCPA	2,4,5-T

1,000µg/mL each in acetonitrile, 1mL/ampul

Each	5-pk.	10-pk.
32430	32430-510	
	w/data pack	
32430-500	32430-520	32530

HPLC columns and additional reference materials listed on pg. 5.

[1]Isocratic mobile phase: 0.05% phosphoric acid:acetonitrile, 60:40. For an example chromatogram of the isocratic analysis, request *Environmental HPLC: Applications-Columns-Reference Materials* (lit. cat.# 59741).

A Good Word

"After the disaster of 9-11, Diazald, a highly explosive compound used in Herbicide analysis, was immediately controlled by the U.S. government which made shipment impossible. Restek was instrumental in helping me to develop an isocratic HPLC method that did not require the use of Diazald. This method is not only safer, but it saves us time and money. Thanks, Restek!"

Chris Domaradzki, Organics manager, Environmental Testing Laboratories

Extracolumn Volume and its Effects in Gradient HPLC

To Maintain Efficiency and Resolution, Use Short Lengths of Narrow-Bore Tubing

By Randy Romesberg, HPLC Applications Chemist

- ✔ *Amount* and *location* of extracolumn volume affect efficiency and resolution.
- ✔ Extracolumn volume *after* the column has greater effect than extracolumn volume *before* the column.
- ✔ 150µL of extra volume can cut efficiency by almost 50%.

Effects of extracolumn volume on band broadening, and the resulting chromatography, have been well studied and documented. These investigations, however, have primarily explored effects in isocratic separations. In this investigation, we have taken a practical look at extracolumn volume in gradient analyses, and studied the effects on actual separations. The data we have obtained show that the location of extracolumn volume in the sample flow path, as well as the amount of extracolumn volume, has a negative effect on theoretical plates (efficiency) and resolution. These extracolumn effects, in combination with the variables of column dimension and analyte retention, play important roles in the resulting chromatography.

To establish baseline chromatographic performance, we analyzed a homologous series of compounds consisting of toluene, ethylbenzene, propylbenzene, butylbenzene, and pentylbenzene on an optimized Agilent series 1100 chromatograph, using a 150 x 4.6mm Pinnacle II™ C18 column (5µm packing) and a methanol gradient (80-100% in 10 min.) or a 50 x 4.6mm Pinnacle II™ C18 column and methanol gradient (80-100% in 3.3 min.). After establishing performance baselines, we added PEEK® tubing of a known internal volume to the sample flow path, ahead of the column or after the column, and repeated the analysis.

Effect of Extracolumn Volume and Location: 15cm Column

Figures 1a and 1b show the effect on efficiency (plates/meter, N/m) and resolution caused by increased extracolumn volume when using a 150 x 4.6mm C18 column under gradient conditions. Chromatographic performance deteriorates, as expected. Unlike observations from isocratic separations, however, extracolumn volume in the portion of the sample path between the column and the detector has a more significant effect than extracolumn volume in the tubing, connections, guard column, etc. located before the column inlet. In fact, for the later-eluting compounds in the test mix, 150µL of extra volume after the column had the same effect as 500µL of extra volume before the column.

Effect of Extracolumn Volume and Location: 5cm Column

Figures 1c and 1d show the effect on efficiency and resolution caused by adding extracolumn volume when using a 50 x 4.6mm C18 column. The effects are, overall, equivalent to those observed with the 150 x 4.6mm column. Since the peak volume is much smaller for this shorter column, however, equal amounts of extracolumn volume have greater effect than on a 150mm column. In this system, 150µL of extra volume before the column reduced efficiency by 46%, whereas with the 150mm column the loss in efficiency was only 20%.

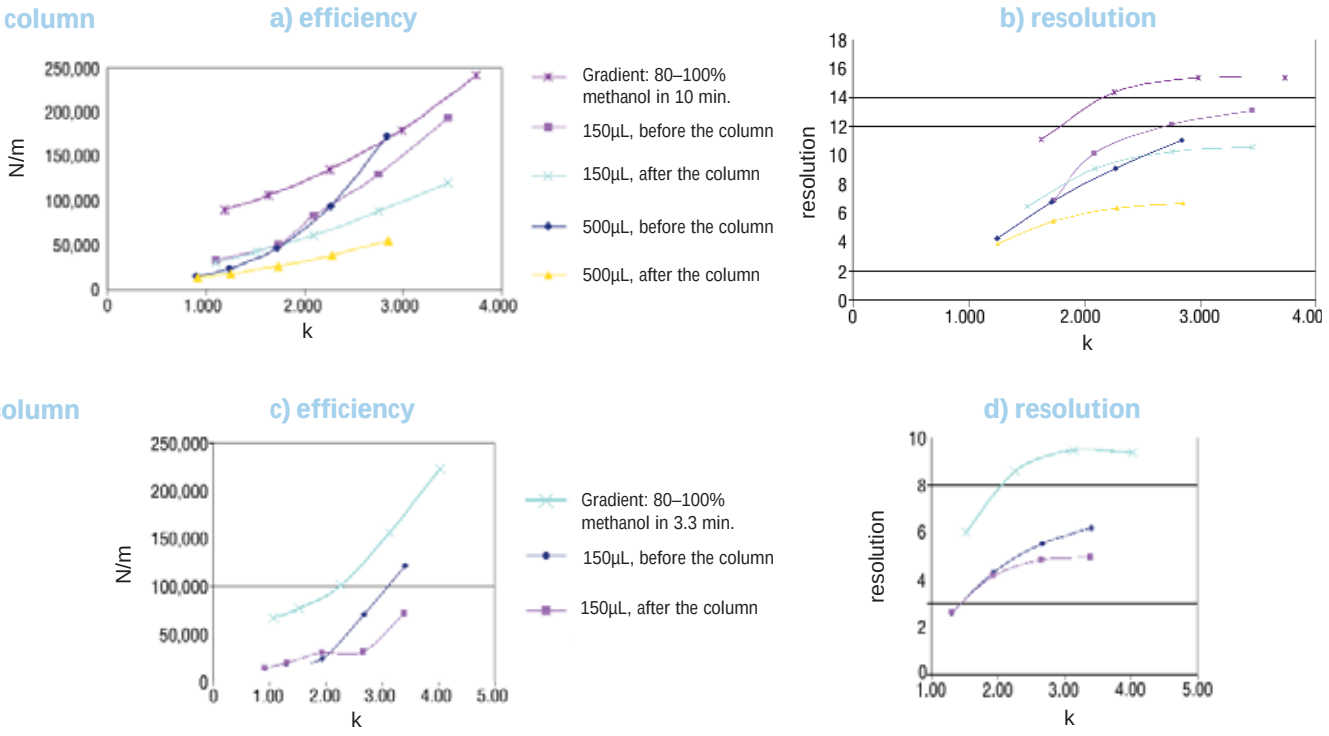
Conclusions

In a gradient analysis, the location of extracolumn volume in the sample flow path can be equally important to the amount of extracolumn volume in its effects on chromatographic performance. In particular, extra volume after the column should be reduced. This is especially important for fast analyses on short columns.

For any HPLC separation, it is best to keep tubing as short and the ID as narrow as practical. Additionally it is wise to use precut stainless steel tubing, or PEEK® tubing cut with a guillotine-style cutter, to ensure square, burr-free ends for minimal dead volume at connections.

For an extensive selection of tubing, low-volume fittings, and related tools, request our HPLC catalog (lit. cat.# 59241A).

Figure 1— Extracolumn volume after the column has greatest negative impact on efficiency and resolution in gradient analysis, as shown by consistently lowest values for plates/meter and resolution versus k.



Inert PEEK® Tubing

- Replaces stainless steel, titanium, Teflon® and Tefzel® tubing.
- Use to 7,000psi.

Description	Color Code	qty.	cat.#	price
PEEK® Tubing, 1/16" OD x 0.0025" ID x 1m	natural	3-pk.	25320	
PEEK® Tubing, 1/16" OD x 0.005" ID x 3m	red stripe	ea.	25065	
PEEK® Tubing, 1/16" OD x 0.007" ID x 3m	yellow stripe	ea.	25066	
PEEK® Tubing, 1/16" OD x 0.010" ID x 3m	blue stripe	ea.	25067	
PEEK® Tubing, 1/16" OD x 0.020" ID x 3m	orange stripe	ea.	25068	

HPLC Stainless Steel Capillary Tubing

- 316 grade stainless steel.
- Precise pre-cut lengths.

Length	ID	OD	qty.	cat.#	price
5cm	0.005"	1/16"	3-pk.	25240	
10cm	0.005"	1/16"	3-pk.	25241	
20cm	0.005"	1/16"	3-pk.	25242	
30cm	0.005"	1/16"	3-pk.	25243	
5cm	0.007"	1/16"	3-pk.	25244	
10cm	0.007"	1/16"	3-pk.	25245	
20cm	0.007"	1/16"	3-pk.	25246	
30cm	0.007"	1/16"	3-pk.	25247	
5cm	0.010"	1/16"	3-pk.	25248	
10cm	0.010"	1/16"	3-pk.	25249	
20cm	0.010"	1/16"	3-pk.	25250	
30cm	0.010"	1/16"	3-pk.	25251	
5cm	0.020"	1/16"	3-pk.	25252	
10cm	0.020"	1/16"	3-pk.	25253	
20cm	0.020"	1/16"	3-pk.	25254	
30cm	0.020"	1/16"	3-pk.	25255	

Clean-Cut™ Tubing Cutter

- Burr-free, perpendicular cuts that will not distort tubing OD.
- Use on PEEK®, Teflon®, Tefzel®, other polymeric tubing.

Description	qty.	cat.#	price
Clean-Cut™ Tubing Cutter	ea.	25069	
Replacement Blade for Clean-Cut™ Cutter	ea.	25070	

Environmental HPLC: Applications, Columns, Reference Materials

(lit. cat.# 59741) Restek HPLC columns support environmental HPLC applications with rapid analysis times and effective resolution of target analytes. Sample turn-around can be 50% faster, or more, than with alternative columns. Applications in this 8-page publication include polyaromatic hydrocarbons, carbamates, phenoxyacid herbicides, explosives, and carbonyls. Analytical reference materials and solid phase extraction sample clean-up products also are listed.

Analyze Underivitized Chlorophenoxyacid Herbicides by HPLC (cont. from pg. 3)

Historically, 2,4-D and Silvex mixtures have comprised many herbicide formulations, and we developed a reference mix for laboratories that analyze only these two compounds. Similarly, we offer an individual solution of dalapon, a herbicide not listed in Method 555, but included in more general Method 8321A. We offer 1,4-dichlorobenzene as an internal standard for this assay. All of our new herbicides mixes are prepared in acetonitrile, as appropriate for HPLC applications, at a convenient concentration of 1000µg/mL.

If your analyses include monitoring chlorophenoxyacid herbicides, an Ultra Aqueous C18 column and our new reference mixes will help you obtain the most accurate data.

Ultra Aqueous C18 HPLC Columns (USP L1)

particle size: 3 or 5µm, spherical; not end-capped; pore size: 100Å; pH range: 2.5 to 7.5; temperature limit: 80°C

	1.0mm ID		2.1mm ID		3.2mm ID		4.6mm ID	
Length	cat.#	price	cat.#	price	cat.#	price	cat.#	price
3µm Columns								
30mm	9178331		9178332		9178333		9178335	
50mm	9178351		9178352		9178353		9178355	
100mm	9178311		9178312		9178313		9178315	
5µm Columns								
30mm	9178531		9178532		9178533		9178535	
50mm	9178551		9178552		9178553		9178555	
100mm	9178511		9178512		9178513		9178515	
150mm	9178561		9178562		9178563		9178565	
200mm	9178521		9178522		9178523		9178525	
250mm	9178571		9178572		9178573		9178575	

Chlorinated Acid Herbicide Mix

2,4-dichlorophenoxyacetic acid
2,4,5-TP (Silvex)
1,000µg/mL each in acetonitrile, 1mL/ampul

Each	5-pk.	10-pk.
32429	32429-510	—
	w/data pack	
32429-500	32429-520	32529

Dalapon

dalapon
1,000µg/mL in acetonitrile, 1mL/ampul

Each	5-pk.	10-pk.
32432	32432-510	—
	w/data pack	
32432-500	32432-520	32532

1,4-Dichlorobenzene

1,4-dichlorobenzene
1,000µg/mL in acetonitrile, 1mL/ampul

Each	5-pk.	10-pk.
30498	30498-510	—
	w/data pack	
30498-500	30498-520	30598

5-Hydroxydicamba

5-hydroxydicamba
100µg/mL in acetone:water (90:10), 5mL/ampul

Each
MET-346A

Restek Replacement Parts for HPLC Systems

By Greg France, HPLC Products Marketing Manager

- ✔ Designed to meet or exceed original equipment performance.
- ✔ Simplify ordering—single source for parts for most popular equipment.
- ✔ Renowned Restek Plus 1™ service.

The column may be the heart of an HPLC system but, just like the human body, the system can only perform as well as the weakest part. Detector lamps, check valves,* pump piston seals, and other components wear out or become contaminated over time. Working with defective parts means poor chromatography and, possibly, shortened column lifetimes.

To keep your system running smoothly and your chromatography looking sharp, we carry a wide range of replacement parts for Agilent, Shimadzu, and Waters instruments. All components meet or exceed the performance of original equipment manufacturer (OEM) parts. If you don't see the part you need on these pages, call us—we regularly add to our inventory.

Replacement Parts for Agilent HPLC Systems

	Description	Model #	Similar to Agilent part #	qty.	cat.#	price
PUMP	Preventive Maintenance Kit	1050	01078-68721	kit	25259	
PUMP	Autosampler Preventive Maintenance Kit	1100	G1313-68709	kit	25271	
PUMP	Pump Maintenance Kit	1050 & 1100	G1311-68710	kit	25270	
PUMP	Outlet Ball Valve, Binary Pump	1100	G1312-60012	ea.	25267	
PUMP	Outlet Ball Valve	1050 & 1100	G1311-60012	ea.	25276	
PUMP	Sieves for Outlet Valve	1050 & 1100	5063-6505	10-pk.	25266	
PUMP	Piston Seals	1050 & 1100	5063-6589	2-pk.	25274	
PUMP	Seal Wash Kit, Binary Pump (4 seals, 4 gaskets)	1100	—	kit	25268	
PUMP	Seal Wash Kit (2 seals, 2 gaskets)	1100	—	kit	25269	
PUMP	Wash Seal	1050 & 1100	0905-1175	ea.	25277	
PUMP	Piston (Sapphire)	1050 & 1100	5063-6586	ea.	25273	
PUMP	Pump Piston Rod (Sapphire)	1050, 1100	—	ea.	25197	
PUMP	Pump Piston Rod (Sapphire)	1090	—	ea.	25198	
AUTOSAMPLER	Needle Seat	1050	79846-67101	ea.	25258	
AUTOSAMPLER	Needle Seat Assembly	1100	G1313-87101	ea.	25265	
AUTOSAMPLER	Needle Assembly	1100	G1313-87201	ea.	25278	
AUTOSAMPLER	Rotor Seal (not for use with 7125)	1050	0101-0626	ea.	25272	
AUTOSAMPLER	Rotor Seal	1100	0100-1853	ea.	25275	
DETECTOR	Detector Lamp, 1090 DA, 1050 VW/DA/MWD	1090, 1050	79883-60002	ea.	25260	
DETECTOR	Lamp, DAD G1315A, G1365A	1100	2140-0590	ea.	25261	
DETECTOR	Lamp, VWD G1314A	1100	G1314-60100	ea.	25262	
DETECTOR	8453 Deuterium Lamp	—	2140-0605	ea.	25263	
DETECTOR	G1321 Fluorescence Detector Flash Lamp	—	2140-0600	ea.	25264	

Replacement Parts for Shimadzu HPLC Systems

	Description	Model #	Similar to Shimadzu part #	qty.	cat.#	price
PUMP	Inlet Check Valve	LC-6A, LC-10AS	228-12353-91	ea.	25287	
PUMP	Inlet Check Valve	LC-600, LC-9A, LC-10AD	228-18522-91	ea.	25295	
PUMP	Outlet Check Valve	LC-6A, LC-10AS	228-09054-93	ea.	25288	
PUMP	Check Valve Rebuild Kit	LC-6A, LC-10AS	228-11200-91	2-pk.	25289	
PUMP	Outlet Check Valve	LC-600, LC-9A, LC-10AD	228-18522-92	ea.	25282	
PUMP	Plunger Seal	LC-6A	228-11999-00	ea.	25285	
PUMP	Plunger Seal	LC-10AS	228-21975-00	ea.	25290	
PUMP	Plunger Seal	LC-600, LC-9A, LC-10AD	228-18745-00	ea.	25293	
PUMP	Plunger Rinse Seal	LC-10AS	228-28499-00	ea.	25292	
PUMP	Plunger	LC-6A	228-12904-93	ea.	25286	
PUMP	Plunger (Sapphire)	LC-10AS	228-17019-93	ea.	25291	
PUMP	Plunger (Sapphire)	LC-600, LC-9A, LC-10AD	228-18523-91	ea.	25294	
PUMP	Deuterium Lamp	SPD-6A	062-65056-02	ea.	25283	
DETECTOR	Deuterium Lamp	SPD-10A, 10AV	228-34016-02	ea.	25284	

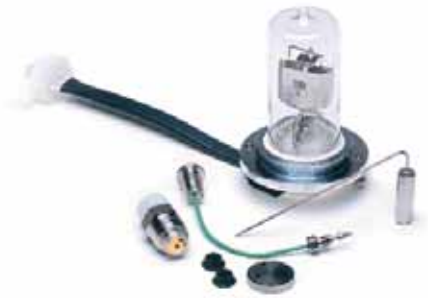
Rheodyne® Style Replacement Parts for Waters™ HPLC Systems

Description	Similar to Rheodyne® part #	qty.	cat.#	price
7010 Vespel® Rotor Seal	7010-039	ea.	25279	
7125 Vespel® Rotor Seal	7125-047	ea.	25280	
Isolation Seal, 7010	7010-015	ea.	25281	

Replacement parts for Shimadzu HPLC systems

Restek also offers replacement parts for PerkinElmer HPLC systems. Please contact us for more information.

Pump piston rods for Waters™ HPLC systems



Replacement parts for Agilent HPLC systems

Replacement parts for Waters™ HPLC systems

*Check valves listed separately - please request our HPLC catalog (lit. cat.# 59241A).

Replacement Parts for Waters™ HPLC Systems

Description	Model #	Similar to Waters™ part #	qty.	cat.#	price
Inlet Check Valve Assembly	M6KA, 501, 510, 515, 590, 600E	33679, 25214	ea.	25360	
Inlet Check Valve Housing	M6KA, 501, 510, 515, 590, 600E	25203	ea.	25361	
Inlet Check Valve Rebuild Kit	M6KA, 501, 510, 515, 590, 600E	60495	2-pk.	25362	
Outlet Check Valve Assembly (Actuator Style)	M6KA, 501, 510, 515, 590, 600E	25030	ea.	25363	
Outlet Check Valve Housing (Actuator Style)	M6KA, 501, 510, 515, 590, 600E	25212	ea.	25364	
Outlet Check Valve Rebuild Kit (Actuator Style)	M6KA, 501, 510, 515, 590, 600E	26016	2-pk.	25365	
Outlet Check Valve Assembly (Ball & Seat Style)	M6KA, 501, 510, 515, 590, 600E	25216	ea.	25366	
Outlet Check Valve Housing (Ball & Seat Style)	M6KA, 501, 510, 515, 590, 600E	25207	ea.	25367	
Outlet Check Valve Rebuild Kit (Ball & Seat Style)	M6KA, 501, 510, 515, 590, 600E	26014	2-pk.	25368	
Inlet Check Valve Assembly, 225µL (Extended Flow)	M6KA, 501, 510, 515, 590, 600E	60307	ea.	25369	
PerformancePLUS™ Check Valve Cartridge	M6KA, 501, 510, 515, 590, 600E	700000254	2-pk.	25370	
Check Valve Rebuild Kit (Extended Flow)	M6KA, 501, 510, 515, 590, 600E	88223	2-pk.	25371	
PerformancePLUS™ Check Valve Housing	M6KA, 501, 510, 515, 590, 600E	—	ea.	25372	
Round Pump Head w/ Actuator Outlets	M6KA, 510, 590, 600	60058	ea.	25413	
Round Pump Head, Ball & Seat Check Valves	M6KA, M45, 501	—	ea.	25414	
Round Pump Head w/o Check Valves (Actuator Style)	M6KA	—	ea.	25415	
Round Pump Head w/o Check Valves (Ball & Seat Style)	M45, 501	—	ea.	25416	
Check Valve Cartridges	Alliance™	WAT270941	2-pk.	25373	
Super Seal™ for Analytical Heads	M6KA, 501, 510, 515, 590, 600E	22946, 22934	ea.	25374	
Plunger Seal, Gold (Analytical Heads)	M6KA, 501, 510, 515, 590, 600E	22934	ea.	25375	
Plunger Seal, Tan	M6KA, 501, 510, 515, 590, 600E	25384	ea.	25376	
Plunger Seal, Red	M6KA, 501, 510, 515, 590, 600E	25638	ea.	25377	
Plunger Seal, Black	M6KA, 501, 510, 515, 590, 600E	26613	ea.	25378	
Plunger Seal, Black (EF Heads)	510, 590, 600E	26644	ea.	25379	
Plunger Seal, Gold (EF Heads)	510, 590, 600E	26644	ea.	25380	
Seal Wash Plunger Seal	Alliance™	WAT271018	2-pk.	25386	
Head Plunger Seal Kit	Alliance™	WAT270938	2-pk.	25387	
Head Plunger Seal Kit (Black)	Alliance™	WAT271066	2-pk.	25388	
Insert Seal Parts Kit	M6KA, 501, 510, 515, 590, 600E	60012	kit	25389	
Plunger (Sapphire)	M6KA, 510, 590, 600	25656	ea.	25381	
Plunger (Sapphire Extended Flow)	510, 590, 600E	60304	ea.	25382	
Plunger (Sapphire)	M45, M501	26524	ea.	25383	
Plunger (Sapphire)	M515	WAT207069	ea.	25384	
Plunger (Sapphire)	616, 625, 626	31788	ea.	25420	
Plunger (Sapphire)	Alliance™	WAT270959	ea.	25385	
Pump Piston Rod (Sapphire)	616, 625, 626	—	ea.	25195	
Pump Piston Rod (Sapphire)	Alliance™ 2690	—	ea.	25196	
Single Solvent Inlet Manifold	600E	60034, 60042	ea.	25390	
Pressure Transducer	M6KA, 501, 510, 515, 590, 600E	60328	ea.	25391	
Draw-Off Tube Assembly	M6KA, 501, 510, 515, 590, 600E	25470	ea.	25392	
1/16" Stainless Steel TEE	M6KA, 501, 510, 515, 590, 600E	75215	ea.	25411	
Inlet Manifold Kit	M45, 501, 510, 590, 600E	60448	kit	25412	
Ferrule, Stainless Steel	515	22330	ea.	25417	
Gradient Proportioning Valve, 9 Volt	600E	34423	ea.	25418	
Gradient Proportioning Valve, 12 Volt	600E	62037	ea.	25419	
Wash Face Seal	Alliance™ 2690	WAT271017	ea.	25428	
Wash Tube Seal Kit	Alliance™ 2690	WAT270940	4-pk.	25429	
Proportioning Valve	Alliance™ 2690	WAT270927	ea.	25430	

Replacement Parts for Waters™ Detectors

Description	Model #	Similar to Waters™ part #	qty.	cat.#	price
LED	410 Refractometer	70162	ea.	25402	
Solenoid Valve	410 Refractometer	70376	ea.	25421	
Quartz Flow Cell	410, 401	48414, 70239	ea.	25422	
Window Gasket	484, 486, 490	80335	ea.	25423	
Lamp Side Gasket	484, 486, 490	80336	ea.	25424	
Quartz Cell Window	484, 486, 490	97091	ea.	25425	
Quartz Lens	486	80687	ea.	25427	
Xenon Lamp (w/o holder or mirror)	470	—	ea.	25404	
Xenon Lamp	474	—	ea.	25405	
Deuterium Lamp (UV/Vis)	480, 481	99499	ea.	25403	
Deuterium Lamp (UV/Vis)	484	80357	ea.	25406	
Deuterium Lamp (UV/Vis)	486	80678	ea.	25407	
Deuterium Lamp	996, 2996	WAT052586	ea.	25408	
Deuterium Lamp	2487	WAS081142	ea.	25409	
Deuterium Lamp, long life (2000 hours)	—	—	ea.	25410	

Restek is your technical literature source!

Visit our website to request free application notes, technical guides, product catalogs, and more!

HPLC Mobile Phase Accessories

(lit. cat.# 59728A)

Items in this 2-page note include: Trident™ guard system components; in-line and reservoir filters and spargers; PEEK® tubing and connectors, Teflon® tubing; PEEK®/Teflon® tubing cutter; tubing clips and elbows; ValvTool wrench; our HPLC Survival Kit of tubing, connectors, filters, and tools. To see our latest HPLC columns and accessories innovations, visit our website.

HPLC Tech Tips Wall Chart

(lit. cat.# 59894A)

Almost everything you need to remember about HPLC, condensed into 3 feet by 2 feet: mobile phase basics, buffers (types, pKas, pH ranges, formula masses, more), miscibility and solubility chart (invaluable!), system setup and optimization, detector tips, pressure conversion factors, most-used chromatographic equations, column storage essentials. Post near your instrument to save time; perhaps save a column.

Faster GC Analysis of Volatile Organics

Using an Rtx®-624 Capillary GC Column and a New MegaMix™ Reference Mix

By Christopher English, Environmental Innovations Chemist, and Katia May, Ph.D., Senior R&D Chemist

- ✔ New MegaMix™ completes a line of reference materials for volatile organics in wastewater.
- ✔ High concentration mixes—more analyses per ampul.
- ✔ Rtx®-624 column: fast analysis, excellent resolution of early-eluting gases.

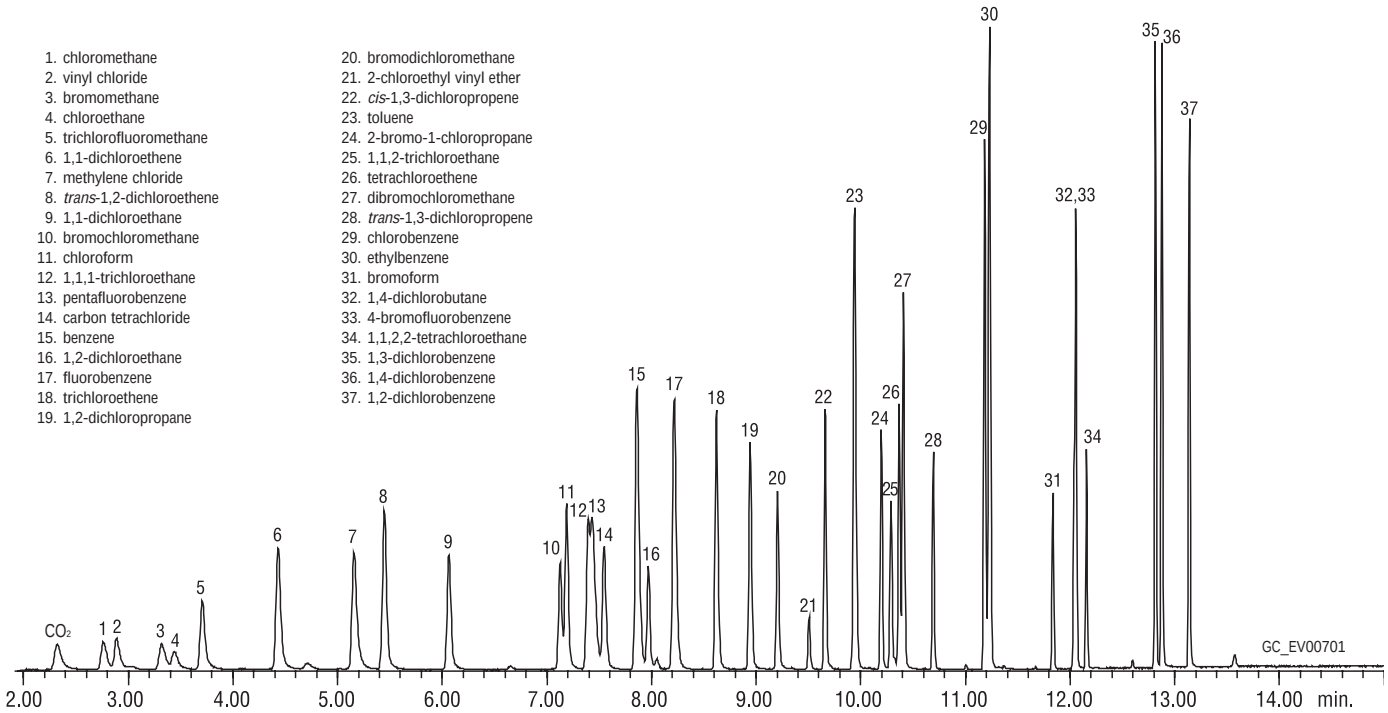
In the US Environmental Protection Agency method for determining 31 volatile organic pollutants in wastewater, EPA Method 624, the target volatile organics are analyzed using purge and trap gas chromatography/mass spectrometry. The purge and trap system efficiently transfers the volatile analytes from the aqueous phase to the vapor phase, from which they are adsorbed to the packing material in a sorbent trap. The concentrated sample is transferred to the chromatographic column by heating the trap under carrier gas flow.

Our new *Volatiles MegaMix™ EPA Method 624* includes the 26 target compounds in Method 624 that are not gases at ambient temperature and pressure. The 5 target gases in Method 624—bromomethane, chloromethane, chloroethane, trichlorofluoromethane, and vinyl chloride—are components in our 624 Calibration Mix #1 (cat.# 30020). We prepare the new MegaMix™, and the gas mix, at a high concentration of 2000µg/mL each component, to enable you to conduct several analyses from each ampul of material.

One of the target compounds in Method 624, 2-chloroethyl vinyl ether, is stable in solution by itself, but hydrolyzes to the enol form in weakly acidic media, then converts to the aldehyde and ketone forms. Because the halocarbon target analytes in Method 624 are slightly acidic compounds, 2-chloroethyl vinyl ether usually is offered separately from the other analytes. We studied the stability of 2-chloroethyl vinyl ether and decided we could include it in our MegaMix™ solution of Method 624 volatiles if we also included a small amount of preservation agent. 2-Chloroethyl vinyl ether is stable in the MegaMix™ mix, but analysts should monitor the stability of this compound in working solutions, regardless of what reference material is the source of the compound. To meet user preference, we also offer 2-chloroethyl vinyl ether in individual solution (cat.# 30265).

Method 624 calls for spiking all samples with surrogate standards, to monitor laboratory performance.

Figure 1—Rapid analysis of Method 624 analytes, with excellent chromatography for early-eluting gases, using a short, narrow bore Rtx®-624 column.



Rtx®-624, 40m, 0.18mm ID, 1.0µm (cat.# 40925)

Sample:	624 Internal Standard Mix (cat.# 30023) 624 Surrogate Standard Mix (cat.# 30243) Volatiles MegaMix™ EPA Method 624 (cat.# 30497)

Purge and trap conditions:

Concentrator:	Tekmar LSC-3100 Purge and Trap
Trap:	Vocarb 3000 (type K) compounds at 50 ppb (IS @ 40ppb) in 5mL of RO water
Purge:	11 min. @ 40 mL/min. @ ambient temperature
Dry purge:	1 min. @ 40mL/min. (MCS bypassed using Silcosteel® tubing)
Desorb preheat:	245°C
Desorb:	250°C for 2 min., flow 10mL/min.
Bake:	260°C for 8 min.
Interface:	Silcosteel® transfer line, 1: 40 split at injection port. 1mm ID liner

Chromatography:

Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1.1 mL/min.
Dead time:	2.06 minutes @ 50°C
Oven temp.:	50°C (hold 4 min.) to 100°C @ 12°C/min. (no hold) to 330°C @ 27°C/min. (hold 2 min.).
Det.:	Agilent 5971A GC/MS
Transfer line temp.:	280°C
Scan range:	35-260 amu
Tune	PFTBA/BFB
Ionization:	EI

Our reference materials for the method include the recommended surrogates (cat.# 30243). The method also requires 3 or more internal standards that are similar in analytical behavior to the target compounds. Our internal standard mix (cat.# 30023) includes recommended compounds and satisfies this requirement.

Based on a cyanopropylphenyl/dimethyl polysiloxane phase, our Rtx®-624 column has unique polarity, and provides excellent separation of the target compounds. By using a short, narrow bore column (40m x 0.18mm ID, 1.0µm phase) we reduced analyses time and improved resolution. This particular column configuration also eliminates non-target interference with target compounds. Figure 1 shows an analyses of all Method 624 target compounds on a 40m x 0.18mm ID Rtx®-624 column. Total cycle time is 17 minutes, which matches the cycle time for the purge and trap concentrator.

By using a 40m x 0.18mm ID, 1.0µm Rtx®-624 column and Restek's convenient, carefully formulated reference materials, you can obtain rapid analyses and accurate data. These practical advantages make Restek the best source for materials for EPA Method 624, and for wastewater volatiles analyses in general.

Rtx®-624 Columns (fused silica)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits	20-Meter	40-Meter
0.18mm	1.00	-20 to 240°C	40924	40925

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	-20 to 240°C	10968	10969		
0.32mm	1.80	-20 to 240°C	10970	10972		
0.45mm	2.55	-20 to 240°C			10982	
0.53mm	3.00	-20 to 240°C	10971	10973	10974	10975

624 Calibration Mix #1

bromomethane
chloroethane
chloromethane
trichlorofluoromethane
vinyl chloride

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30020	30020-510	—
	w/data pack	
30020-500	30020-520	30120

624 Surrogate Standard Mix

4-bromofluorobenzene
fluorobenzene
pentafluorobenzene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30243	30243-510	—
	w/data pack	
30243-500	30243-520	30343

Volatiles MegaMix™, EPA Method 624 (26 components)

benzene
bromodichloromethane
bromoform
carbon tetrachloride
chlorobenzene
2-chloroethyl vinyl ether
chloroform
dibromochloromethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethylene
trans-1,2-dichloroethylene
1,2-dichloropropane
cis-1,3-dichloropropylene
trans-1,3-dichloropropylene
ethylbenzene
methylene chloride (dichloromethane)
1,1,2,2-tetrachloroethane
tetrachloroethylene
toluene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethylene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30497	30497-510	—
	w/data pack	
30497-500	30497-520	30597

HOT Tech Tip!

Stability of 2-Chloroethyl vinyl ether

2-Chloroethyl vinyl ether is stable in neutral pH and in slightly basic solutions. If the solution is slightly acidic, the analyte will rapidly decompose. Restek specially prepares stable individual solutions of 2-chloroethyl vinyl ether in neutral purge and trap methanol. These solutions are very stable and can be diluted without problems, using pure, neutral, P&T grade methanol. Be careful when combining these solutions with other calibration materials - some solutions can contain trace acidic impurities that will cause rapid decomposition of 2-chloroethyl vinyl ether. Be especially cautious of calibration mixtures that contain high concentrations or a large number of chlorinated target compounds; these often will contain sufficient trace HCl to cause stability problems with 2-chloroethyl vinyl ether.

624 Internal Standard Mix

bromochloromethane
2-bromo-1-chloropropane
1,4-dichlorobutane

1,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30023	30023-510	—
	w/data pack	
30023-500	30023-520	30123

mixitup! Restek's MegaMix™ Standards

MegaMix™ Reference Mixes

For US EPA Methods
Volatiles: 524.2, 624, 8260, OLC 03.2, OLM 04.2, 502.2
Semivolatiles: 525.2, 625, 8270, OLC 03.2, OLM 04.2

- ✔ Fewest mixes needed for calibration or matrix spike
- ✔ Maximum stability
- ✔ Most commonly analyzed compounds:
  - 8260—76 volatile compounds in 1 mix
    gases and ketones in separate mixes, for maximum stability
  - 8270—76 semivolatile compounds in 1 mix
  - Appendix IX—59 semivolatile compounds in 3 mixes
  - 524—73 volatile compounds in 1 mix
  - 525—90 semivolatile compounds in 5 mixes
  - 625—54 semivolatile compounds in 1 mix
  - CLP 03.2 (volatiles and semivolatiles)
  - CLP 04.2 (volatiles and semivolatiles)

For details, visit our website at www.restekcorp.com

New Analytical Reference Materials

For Forensic and Environmental Analyses

By Katia May, Ph.D., Senior R&D Chemist

Forensic Reference Materials

Forensic Ethanol Standards

- ✔ 0.08g/dL standard supports new federal blood alcohol limit.
- ✔ 0.4g/dL standard supports autopsy of alcohol-related deaths.
- ✔ 0.05g/dL standard supports limits for long-haul truck drivers.
- ✔ Many other concentrations available.

The United States' blood alcohol limit has been reduced to 0.08g/dL. Consistent with our commitment to support police and crime laboratories, we are introducing three new reference mixes to meet current needs. Restek Forensic Ethanol Standards are NIST traceable. Data packs included.

Forensic ethanol solutions w/data pack	5-pk. 1mL/ampul	10-pk. 1mL/ampul	ea. 5mL/ampul	ea. 20mL/ampul
0.05g/dL forensic ethanol solution	36257	36259	36258	36260
0.08g/dL forensic ethanol solution	36262	36264	36263	36265
0.4g/dL forensic ethanol solution	36266	36268	36267	36269

Blood Alcohol Mix Resolution Control Standard

acetaldehyde
acetone
acetonitrile
ethanol (NIST certified value)
ethyl acetate
isopropanol
methanol
methyl ethyl ketone

0.100g/dL each in water, 1mL/ampul

Each w/data pack
36256

Environmental Reference Materials

Carbazole

- ✔ No interference with OLC 03.2 target compounds.

Many laboratories following US EPA Contract Laboratory Program OLC 03.2 Statement of Work also analyze for carbazole. Most carbazole reference solutions are in methanol, but certain target compounds in OLC 03.2 SOW react with methanol (e.g., benzaldehyde, atrazine). We prepare our new reference standard in methanol-free methylene chloride, to prevent reactions when it is added to OLC 03.2 Semivolatile MegaMix™ (cat.# 31812).

Carbazole

carbazole

1,000µg/mL in methylene chloride (methanol free), 1mL/ampul

Each	5-pk.	10-pk.
31836	31836-510	—
	w/data pack	
31836-500	31836-520	31936

ε-Caprolactam

A precursor in the synthesis of nylon-6, ε-caprolactam is one of the most heavily and widely used chemical intermediates—more than 9.5 billion pounds each year, worldwide. Environmental contamination should be anticipated, and caprolactam has toxic effects. This solution is suitable for monitoring ε-caprolactam.

ε-Caprolactam

ε-caprolactam

2,000µg/mL in methylene chloride (methanol free), 1mL/ampul

Each	5-pk.	10-pk.
31833	31833-510	—
	w/data pack	
31833-500	31833-520	31933

Glyphosate and AMPA (glyphosate metabolite)

- ✔ Glyphosate packaged in two volumes, to meet varied requirements.
- ✔ Glyphosate at 1000µg/mL concentration, for more analyses per ampul.

Glyphosate (N-phosphonomethyl glycine) is a broad-spectrum post-emergence herbicide used in agriculture and forestry and for aquatic weed control. A weak organic acid, glyphosate usually is formulated as the isopropylamine salt to increase solubility. Our new mix is suitable for EPA Method 547, for identifying and measuring glyphosate in drinking water (HPLC with fluorescence detection and post-column derivatization).

Aminomethylphosphonic acid—AMPA—is the primary degradation product of glyphosate in plants, soil, and water. The chemical structures of the two compounds are very similar, and they have similar toxicological profiles. The health base value for glyphosate also applies to AMPA, and to glyphosate and AMPA in combination.

Glyphosate

glyphosate

Each	5-pk.	10-pk.
1,000µg/mL in DI water, 1mL/ampul		
32426	32426-510	—
	w/ data pack	
32426-500	32426-520	32526
1,000µg/mL in DI water, 5mL/ampul		
32427	32427-510	—
	w/ data pack	
32427-500	32427-520	32527

AMPA (glyphosate metabolite)

aminomethyl phosphonic acid (AMPA)

100µg/mL in DI water, 1mL/ampul

Each	5-pk.	10-pk.
32428	32428-510	—
	w/data pack	
32428-500	32428-520	32528

PCB-Free Transformer Oil

- ✔ Convenient 5mL and 50mL packaging.

Use and disposal of all mineral oil-filled transformers have been subject to federal regulation since 1978. Traditionally, transformer oil is tested for polychlorinated biphenyls (PCBs) contamination by GC.

PCB-Free Transformer Oil

PCB-Free transformer oil

Neat

5mL	50mL
32424	32425

Acrolein & Acrylonitrile

- ✔ High concentrations:
  acrolein: 10,000µg/mL
  acrylonitrile: 2,000µg/mL
  acrolein/acrylonitrile: 2,000µg/mL.

Acrolein and acrylonitrile are monomers used in manufacturing polyacrylamide and other acrylic polymers. These new mixes are suitable for use with US EPA Method 603, or other methods for testing water samples for acrylonitrile and acrolein by purge & trap GC. They have a shelf life of 2-3 months.

Acrolein

acrolein

10,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30499	30499-510	—
	w/data pack	
30499-500	30499-520	30599

Acrylonitrile

acrylonitrile

2,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30246	30246-510	—
	w/data pack	
30246-500	30246-520	30346

Acrolein/Acrylonitrile

acrolein
acrylonitrile

2,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30600	30600-510	—
	w/data pack	
30600-500	30600-520	30700

Bulk HPLC Silica and Bonded Phase Packings

For Scale-Up or Other Applications

By Greg France, HPLC Product Marketing Manager

- ✔Consistent, high-quality porous spherical silicas for
  - neutral to slightly acidic compounds
  - basic compounds.
- ✔ Silica and bonded materials rigorously tested; ISO 9001:2000 registered facility.

Restek is one of the few HPLC column suppliers manufacturing chromatography grade silica. We now offer our high-quality silicas and bonded phase materials in bulk, for packing analytical-scale columns, for preparative and process purification, or as raw materials for other LC analytical platforms.

Pinnacle II™ is a Type A spherical silica with a mean pore size of 110Å and a surface area of 180m2/g. It matches Hypersil® silica in many respects, but the metals content in Pinnacle II™ is almost an order of magnitude less than that in Hypersil® silica.[1] Practically, this is expressed as a less active surface and a more durable particle. Packings based on Pinnacle II™ silica are excellent for analyzing or purifying neutral to slightly acidic compounds (Figure 1). Standard particle sizes for Pinnacle II™ materials are 3µm and 5µm; 10µm and 15µm particles are available on request.

Highly base-deactivated Pinnacle™ DB silica has mechanical strength and durability similar to Pinnacle II™ silica, but Pinnacle™ DB silica has larger pores (140 Å). The total metals content is less than 250ppm, and no single metal exceeds 100ppm.[2] Packings based on Pinnacle™ DB silica are suitable for a wide range of analytes; basic analytes often can be analyzed with little or no mobile phase modifier (Figure 2). Performance is very similar to Hypersil® BDS material.

Our extensive QC program ensures the quality and reproducibility of Pinnacle II™ and Pinnacle™ DB silicas: each lot of material must meet specifications for mean particle size, particle size distribution, pore diameter, surface area, and total metals content. You can use these materials with confidence.

Figure 1—Morphine sulfate resolved from manufacturing solvent, using a Pinnacle II™ C18 column (USP 25 Resolution Solution).

Peak List:	**Conc.** (µg/mL)
U. unknown	unknown
1. phenol	0.15
2. morphine sulfate	0.24

Column:	**Pinnacle II™ C18**
Cat.#:	9214575-700
Dimensions:	250 x 4.6mm
Particle Size:	5µm
Pore Size:	110Å
Conditions:	
Mobile Phase:	A: 0.73g 1-heptane sulfonic acid and 10mL glacial acetic acid diluted to 720mL with water, pH=2.33 B: methanol 72A:28B, v/v
Flow:	1.00mL/min
Temp.:	26.5°C
Det.:	UV @ 284nm
Sample:	
Inj.:	20.0µL
Solvent:	mobile phase

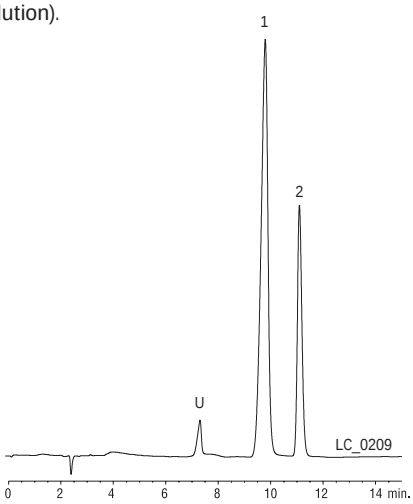
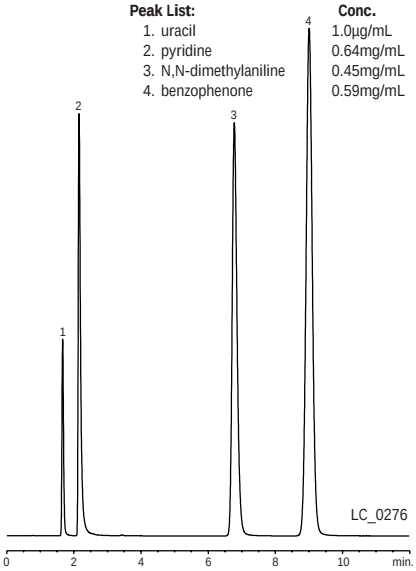


Figure 2—Sharp, near-symmetric peak for a basic analyte (pyridine) without a mobile phase modifier, using a Pinnacle™ DB column.

Peak List:	**Conc.**
1. uracil	1.0µg/mL
2. pyridine	0.64mg/mL
3. N,N-dimethylaniline	0.45mg/mL
4. benzophenone	0.59mg/mL



Column:	**Pinnacle™ DB C18**
Cat. #:	9414565
Dimensions:	150 x 4.6mm
Particle Size:	5µm
Pore Size:	140Å
Conditions:	
Mobile Phase:	water:methanol (40:60 v/v)
Flow:	1.0mL/min.
Temp.:	ambient
Det.:	UV @ 254nm
Sample:	
Inj.:	5µL
Sample Diluent:	methanol:water (50:50 v/v)

3µm Pinnacle II™ Bulk Packing Materials

Description	min. qty.	cat.#	price per gram
Pinnacle II™ C8 Bulk Packing	5g	92133	
Pinnacle II™ C18 Bulk Packing	5g	92143	
Pinnacle II™ Cyano Bulk Packing	5g	92163	
Pinnacle II™ Phenyl Bulk Packing	5g	92153	
Pinnacle II™ Silica Bulk Packing	5g	92103	

5µm Pinnacle II™ Bulk Packing Materials

Description	min. qty.	cat.#	price per gram
Pinnacle II™ C8 Bulk Packing	5g	92135	
Pinnacle II™ C18 Bulk Packing	5g	92145	
Pinnacle II™ Cyano Bulk Packing	5g	92165	
Pinnacle II™ Phenyl Bulk Packing	5g	92155	
Pinnacle II™ Silica Bulk Packing	5g	92105	

5µm Pinnacle™ DB Bulk Packing Materials

Description	min. qty.	cat.#	price per gram
Pinnacle™ DB C18 Bulk Packing	5g	94145	
Pinnacle™ DB C8 Bulk Packing	5g	94135	
Pinnacle™ DB Cyano Bulk Packing	5g	94165	
Pinnacle™ DB Silica Bulk Packing	5g	94105	

HOT Tech Tip!

How important is metals content?

Metal ions on a silica particle weaken the particle and negatively affect chromatography, particularly for basic analytes. These problems can be overcome—temporarily—by annealing the metals into the framework of the particles. As the particles age, the metals are re-exposed. Base deactivation is lost and the particles' stability in highly aqueous mobile phases is further eroded.

Pinnacle II™ and Pinnacle™ DB silicas do not require annealing; they provide more consistent peak shapes for bases as the column ages, and a potentially longer column lifetime.

[1]Request publication 59517.
[2]Request Applications Note 59742.

Peak Performers

Avoid Septum Problems

By Donna Lidgett, GC Accessories Marketing Manager

- ✔ Handle septa carefully, to prevent contamination.
- ✔ Minimize bleed—use preconditioned, low-bleed septa.

Septum Handling

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

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

Septum Bleed

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

Because most GCs are equipped with a septum purge, septum bleed generally will disappear within 30 minutes after installing a new septum and exposing it to normal injector temperatures. All Restek septa eliminate this conditioning period because they are preconditioned and can be used without delay.

Why are Low-Bleed Septa Important?

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

Why Does Septum Puncturability Matter?

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

What Septum Configurations are Available, and for Which GCs?

Restek has fashioned septa for all major brands of gas chromatographs and injectors. Use the septum size chart to determine the septum diameter for your instrument, or measure an old septum against the template if your model is not listed.

Which Septa Should I Use?

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

InfraRed™ septa are low bleed septa with a 325ºC maximum temperature. Even at maximum temperature, InfraRed™ septa provide long lifetime with little coring.

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

Thermolite® Septa

- Usable to 340°C inlet temperature.
- Each batch tested with FIDs, ECDs, and MSDs to ensure lowest bleed.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.

Septum Diameter	25-pk./price	50-pk./price	100-pk./price
5mm (3/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	— —
9mm	20354	20358	20362
9.5mm (3/8")	20359	20360	20361
10mm	20378	20379	20380
11mm (7/16")	20363	20364	20365
11.5mm	22385	22386	22387
12.5mm (1/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374

InfraRed™ Septa

- Usable to 325°C inlet temperature.
- Preconditioned and ready to use.
- Excellent puncturability.
- Do not adhere to hot metal surfaces.
- Low bleed.
- Packaged in non-contaminating glass jars.

Septum Diameter	25-pk./price	50-pk./price	100-pk./price
9mm	21417	21418	21419
9.5mm (3/8")	21421	21422	21423
10mm	21424	21425	21426
11mm (7/16")	21427	21428	21429
11.5mm	21430	21431	21432
12.5mm (1/2")	21433	21434	21435
17mm	21436	21437	21438
Shimadzu Plug	21439	21440	21441

IceBlue™ Septa

- Usable to 250°C inlet temperature.
- General-purpose septa.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.
- Ideal for SPME.

Septum Diameter	50-pk./price	100-pk./price
9mm	22381	22382
9.5mm (3/8")	22388	22389
10mm	22390	22391
11mm (7/16")	22392	22393
11.5mm	22383	22384
12.5mm (1/2")	22394	22395
17mm	22396	22397
Shimadzu Plug	22398	22399

Measure your old septum here (size in mm)

Septum Puller

- Keep several on hand in your laboratory—can be used in many different ways.
- Hooked end can remove septa and O-rings; pointed end works well for removing stuck ferrule fragments.

Remove septa, o-rings, and ferrules without damaging fittings.

Description	qty.	cat.#	price
Septum Puller	ea.	20117	

Leak Detective™ II Leak Detector

Compact, sensitive, affordable.

- Affordable thermal conductivity leak detector—every analyst should have one.
- Compact, ergonomic design is easy to hold and operate.
- Sensitive—detects helium, hydrogen, or nitrogen at $1x10^{-4}$cc/sec. (absolute concentration as low as 100ppm.)*
- Fast results—responds in less than 2 seconds to trace leaks of gases with thermal conductivities different from air.
- Auto zeroing with the touch of a button.
- Battery-operated for increased portability (requires one 9-volt battery; two Ni-MH rechargeable batteries and charger included for your convenience).

Description	qty.	cat.#	price
Leak Detective™ II Leak Detector with 110Volt Battery Charger	ea.	20413	
Leak Detective™ II Leak Detector with 220Volt European Battery Charger	ea.	20413-EUR	
Leak Detective™ II Leak Detector with 220Volt UK Battery Charger	ea.	20413-UK	

*Never use liquid leak detectors on a capillary system because liquids can be drawn into the column.
Caution: NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possible hazardous conditions.

Merlin Microseal™ Septa for Agilent GCs

- High-pressure capability allows operation from 2 to 100psi.
- Top wiper rib improves resistance to particulate contamination and can be taken apart for cleaning.
- High resistance to wear greatly reduces shedding of septum particles into the injection port liner, eliminating a major source of septum bleed and ghost peaks.
- Longer life reduces the risk of septum leaks during extended automated runs.

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

handy septum size chart

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

cool tools

Super-Clean™ Gas Filters

By Donna Lidgett, GC Accessories Marketing Manager

- ✔ MS-quality output: 99.9999% pure gas.
- ✔ "Quick connect" fittings for fast, easy, leak-free cartridge changes.
- ✔ Glass inside prevents diffusion, plastic outside for safety.

Super-Clean™ system: fast, simple cartridge changes

Cartridge-style gas purification systems make changing gas filters quick and easy, and the Super-Clean™ gas filter system is the latest improvement to cartridge-style technology. A baseplate in the Super-Clean™ system allows cartridges to be exchanged without introducing atmospheric oxygen and water vapor: spring-loaded check valves seal when a cartridge is removed and open only when a new cartridge has been locked in place. You no longer need to loosen and tighten fittings every time you change a cartridge, and your system cannot become contaminated during the changing process.

Use a 2- or 3-position baseplate to purify multiple GC gas streams at one location—Figure 1 shows some possible filter cartridge combinations. Many combinations are possible because any Super-Clean™ filter cartridge can be used with any baseplate.

Figure 1—Filter cartridges can be configured for different applications.

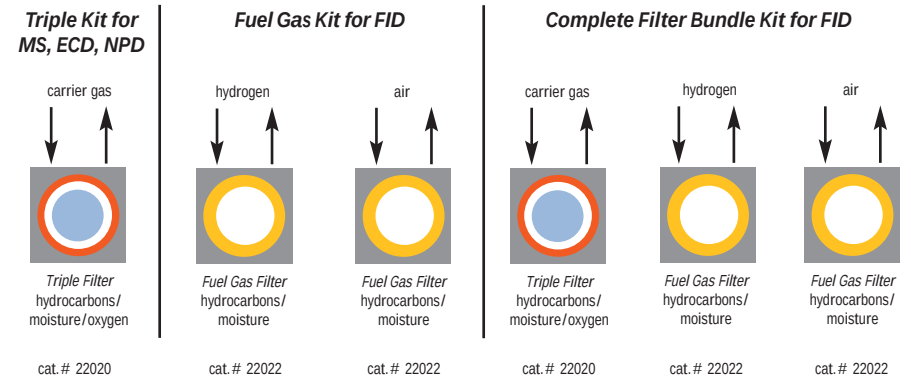


Table I—Each Super-Clean™ filter provides high-purity outlet gas.

Type of Filter	Outlet Gas Quality (%)	Max. Pressure	Use for:	Indicator Color Change	H_20 (g)	Capacity O_2 (mL)	Hydrocarb.	Estimated Lifetime (years)
Moisture cat.# 22028	>99.9999	11 bar 159psi	Inert carrier gas Air Hydrogen	Yellow to Clear	7.2	—	—	>2
Oxygen cat.# 22029	>99.9999	11 bar 159psi	Inert carrier gas	Green to Grey	NA	1000	—	>2
Hydrocarbons cat.# 22030	>99.9999	11 bar 159psi	Inert carrier gas Air Hydrogen	No Indicator	NA	—	—	>2
Fuel Gas Filter cat.# 22022	>99.9999	11 bar 159psi	Inert carrier gas Air Hydrogen	Yellow to Clear	3.6	—	—	>1.5
Triple (Moist., O_2, Hydroc.) cat.# 22020	>99.9999	11 bar 159psi	Inert carrier gas	Yellow to Clear Green to Grey	1.8	500	—	>1
Helium cat.# 21982	>99.9999	11 bar 159psi	Helium	Yellow to Clear Green to Grey	1.8	500	—	>1

High-purity output improves sensitivity

The Triple Filter cartridge (cat.# 22020) is ideal for purifying carrier gas (Figure 1). It combines oxygen, moisture, and hydrocarbon removers in one cartridge. Purity of carrier gas leaving a Triple Filter is better than "six nines" (99.9999%), which is ideal for noise-free baselines from sensitive mass spectrometry or electron capture detection equipment, and for protecting your analytical columns against damage from contaminants.

The Fuel Gas Filter cartridge (cat.# 22022) is perfect for purifying flame ionization detector fuel gases, removing both moisture and hydrocarbons. Use Fuel Gas Filters in a 2-position baseplate for FID hydrogen and air (Figure 1), to produce a stable baseline and improve overall sensitivity and reproducibility. The new Helium Specific Carrier Gas Cleaning Kit (cat.# 21983) is designed specifically for purifying helium used in GC/MS systems. The cartridge is prepared and conditioned using high-purity helium, to minimize conditioning time in your system.

All Super-Clean™ filter cartridges except the hydrocarbon cartridge feature **easy-to-read indicators.** The indicator code is shown on every trap so there is no confusion about when to replace it.

Refer to the **Purus™ Gas Systems** section of the Restek catalog for all your gas system needs:

- Many additional gas purifiers.
- Gas generators: convenient, safe, economical alternatives to gas cylinders.
- Pressure regulators.
- Tubing, tubing tools, fittings and valves.
- Leak detectors.
- Much more.

Super-Clean™ Filter and Baseplate Kits

- High-purity output ensures 99.9999% pure gas.
- "Quick connect" fittings for easy, leak-free cartridge changes.
- Glass inside to prevent diffusion; plastic outside for safety.

Description	qty.	cat.#	price
Carrier Gas Cleaning Kit (includes mounting baseplate, 1/8" inlet/outlet fittings, and oxygen/moisture/hydrocarbon Triple Filter)	kit	22019	
Fuel Gas Purification Kit (includes mounting baseplate, 1/8" inlet/outlet fittings, and hydrocarbon/moisture filter)	kit	22021	

All traps measure: 10 5/8" x 1 3/4"
Each baseplate unit measures: 4" x 4" x 1 7/8"

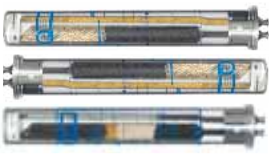
Replacement Filters

Description	qty.	cat.#	price
Replacement Triple Filter (removes oxygen, moisture and hydrocarbons)	ea.	22020	
Replacement Fuel Gas Filter (removes moisture and hydrocarbons)	ea.	22022	

Filter Bundle Kit

Kit includes two Fuel Gas Filters for FID fuel gases and one Triple Filter for carrier gas. Ideal for use in combination with 3-position baseplate—purchase separately.

Description	qty.	cat.#	price
Filter Bundle Kit	kit	22031	



Helium-Specific Super-Clean™ Filter and Kit

new

- Specifically designed for purification of helium in GC/MS Systems.
- Traps are packed and conditioned using helium.
- Uses standard single-position baseplate.

Description	qty.	cat.#	price
Helium-Specific Carrier Gas Cleaning Kit (includes mounting baseplate, 1/8" inlet/outlet fittings, and helium-conditioned oxygen/moisture/hydrocarbon filter)	kit	21983	
Helium-Specific Replacement Filter (removes oxygen, moisture and hydrocarbons)	ea.	21982	

Super-Clean™ Ultra-High Capacity Filters

Description	qty.	cat.#	price
Ultra-High Capacity Hydrocarbon Filter	ea.	22030	
Ultra-High Capacity Moisture Filter	ea.	22028	
Ultra-High Capacity Oxygen Filter	ea.	22029	

Baseplates

All baseplate fittings are 1/8". To adapt to 1/4", order 1/8" to 1/4" tube-end union listed below.

Description	qty.	cat.#	price
Single-Position Baseplate	ea.	22025	
2-Position Baseplate	ea.	22026	
3-Position Baseplate	ea.	22027	

Replacement O-Rings

Pack includes 10 large O-rings and 10 small O-rings.

Description	qty.	cat.#	price
Replacement O-Rings for Cartridge Baseplates	20-pk.	22023	

1/8-Inch to 1/4-Inch Tube-End Unions

All Super-Clean™ baseplate fittings are 1/8". To adapt to 1/4", use a 1/8" to 1/4" tube-end union.

Description	qty.	cat.#	price
1/8" to 1/4" Tube-End Unions	5-pk.	21833	

Wall Mounting Bracket

new

Baseplates may be mounted by using screws and the mounting holes on the baseplate or by using this optional wall mounting bracket.

Description	qty.	cat.#	price
Wall Mounting Bracket for Super-Clean™ Baseplates	ea.	21984	

Service Rewards Program Distributes More Than 50,000 STAR™ Points

Labs Already Cutting Service Costs

By Doug Elliott, STAR Service Rewards Coordinator

✔ Order high quality products, obtain credits toward instrument service and repair.

The STAR™ Service Reward Program was initiated in April 2002 in a test-market mode on the US west coast. The program was launched nationally in May of 2003. Since its inception the program has distributed more than 50,000 STAR™ Points to chromatography laboratories who are, in turn, using the points to lower their service and repair parts costs. One west coast lab already has redeemed 600 STAR™ Points with their preferred service provider, another lowered their service costs by redeeming over 500 STAR™ Points.

If you're not participating in this program, it's not too late! Register your lab by calling the Restek Customer Service Team at 1-800-356-1688, ext. 3. Just provide your company account number and your ship-to address—that's it! After your lab is registered, you will begin to receive STAR™ Points in your product packages, just like Wizard Dollars—1 STAR™ Point for every $50 increment of Restek products you purchase.

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- ferrules
- FID jets
- filters for GC & HPLC
- gas generators
- gas leak detectors
- gas purifiers
- Hamilton & SGE syringes
- HPLC system parts
- inlet liners
- inlet seals
- instrument repair parts
- mobile phase accessories
- PID lamps
- plumbing supplies
- pressure regulators
- Press-Tight® column-connectors
- septa
- fittings and tubing, GC & HPLC
- tools

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- Helpful Technical Information
- New GC and HPLC Columns
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- New Analytical Reference Materials
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If you're having difficulty locating parts for your Agilent 5890, 6890, or 7673A/B, call Restek. We now offer vial turrets, motors, belts, and more. Performance will equal or exceed that of OEM parts. For descriptions of current parts, see the revolving Feature Product box on the home page of our web site: **www.restekcorp.com**

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Please direct your comments on this publication to Carrie Sprout, Graphic Designer, at carrie@restekcorp.com or call 814-353-1300, ext. 2151.

RESTEK

Restek Corporation

THE RESTEK ADVANTAGE

Turning Visions into Reality™

2005 vol. 2

Rtx®-PCB: Unique Selectivity for PCBs

110 of 158 target PCB Congeners Elute Individually, Using GC/ECD

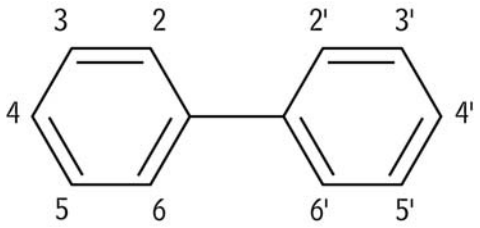
by Gary Stidsen, GC Columns Marketing Manager

- NEW low polarity, inert polymer phase provides distinct separations of PCB congeners.
- Unmatched selectivity and low bleed—a column of choice for trace analysis.
- Thermally stable to 340ºC.

Rtx®-PCB columns show unique selectivity for polychlorinated biphenyl (PCB) congeners. In previous publications (lit.# 59925 and Advantage 2005v1, lit.# 59077, page 13), we discussed the excellent performance of this column and showed each of the European PCB congener indicator compounds - PCBs 28, 52, 101, 118, 138, 153, and 180 - can be resolved from other, interfering PCB congeners and quantified, using GC/MS.

"Weathering" of Aroclor® mixes that have been in the environment for more than 30 years, and changes in Aroclor® patterns in tissue samples, due to bioaccumulation, have dictated that PCBs now be reported as congeners, rather than as Aroclor® mixes. Consequently, many laboratories are analyzing longer lists of PCB congeners, using the data in determining specific congener patterns, in compiling congener results to obtain an accurate total PCB concentration, and in other ways.

Figure 1 Biphenyl structure supports 209 PCB congeners, many with very similar retention characteristics.



The structure of the biphenyl molecule is shown in Figure 1. Identification and quantification of PCB congeners is chromatographically challenging because there are 209 possible combinations in which chlorine atoms can be added to the biphenyl structure, ranging from addition of a single chlorine (monochlorobiphenyls) to addition at every available carbon atom (decachlorobiphenyl). In 1996, George Frame published work he performed in order to determine which PCB congeners are present in Aroclor® mixes.[1] Using this work as a guide, the list of 209 possible PCB congeners can be reduced to a target list of 158 congeners. This final target list includes PCB congeners found in Aroclor® mixes above 0.01%wt/wt, and a few compounds not detected in Aroclor® mixes, but detected in tissue due to

in this issue

Restek Goes West!

Roy Lautamo gliding over Kings Canyon in the southern Sierra.

Restek celebrates continued growth in 2005 with the opening of Restek West, our new R&D facility in Shingle Springs, California. Roy Lautamo, Director of Innovative Research Chemistries, will manage the facility, focusing on R&D for our chromatography column product lines. Roy has an extensive range of experience in chromatography, acquired over more than a quarter of a century of research.

We welcome Roy and his staff into the Restek family!

Correction

In *Advantage* 2005v1: *Fast GC/MS Analysis of Semivolatile Organic Compounds*, Figure 1 (page 14) the splitless hold time and pressure pulse time are reversed. The splitless hold time should be 0.15 min. and the pressure pulse time should be 0.20 min. We apologize for any inconvenience caused by this error.

bioaccumulation (e.g., PCB#169). The list encompasses the seven European indicator compounds and the 12 most toxic congeners, according to the World Health Organization (WHO) list.[2]

Using a 40m x 0.18mm ID, 0.18µm film Rtx®-PCB column and the conditions listed in Figure 2, we evaluated a sample composed of Aroclor® PCB mixes 1242, 1254, and 1262. The 158 target PCB congeners were identified in the sample, eluted as 135 chromatographic peaks. Of the 158 congeners, 110 eluted singly and 48 were unresolved.

Produced through one of our newest polymer technologies, Rtx®-PCB columns undergo rigorous quality assurance measures, to ensure every column meets exacting standards and to give you highly reproducible performance, from column to column. If you are analyzing PCBs—as congeners, as Aroclor® mixtures, or as other mixtures (e.g., Kaneclor, Clophen, or Phenoclor mixes)—we highly recommend using these new columns.

References

1. Frame, G., J. Cochran, and S., Bowadt, *Complete PCB Congener Distributions for 17 Aroclor Mixtures Determined by 3 HRGC Systems Optimized for Comprehensive, Quantitative, Congener-Specific Analysis* J. High Res. Chromatogr. 19, Dec. 1996, pp. 657-668.
2. Executive Summary, *Assessment of the Health Risk of Dioxins: Re-evaluation of the Tolerable Daily Intake (TDI)*, WHO Consultation, May 25-29, 1998, Geneva Switzerland. See: http://www.who.int/ipcs/publications/en/exe-sum-final.pdf

PCB Congener Standard #2

2,4,4' (BZ #28)	2,2',3,4,4',5' (BZ #138)
2,2',5,5' (BZ #52)	2,2',4,4',5,5' (BZ #153)
2,2',4,5,5' (BZ #101)	2,2',3,4,4',5,5' (BZ #180)
2,3',4,4',5 (BZ #118)	

10µg/mL each in isooctane, 1mL/ampul
cat. # 32294 (ea.)

For additional PCB congener mixes, and Aroclor® reference materials, please see our current catalog, or visit our website.

Rtx®-PCB (fused silica)

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

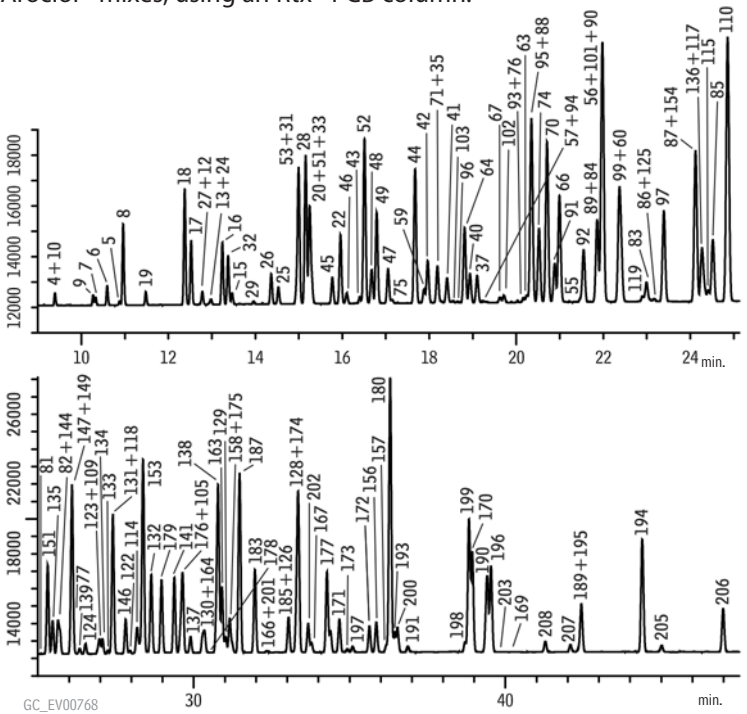
The maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Table I 110 of 158 target PCB congeners in Aroclor® mixes are eluted singly from an Rtx®-PCB column.

PCB#	s/m*	PCB#	s/m*	PCB#	s/m*	PCB#	s/m*	PCB#	s/m*
1	s	35	s	82	s	126	s	173	s
2	s	37	s	83	s	128	m	174	m
3	s	40	s	84	m	129	s	175	s
4	m	41	s	85	s	130	s	176	m
5	s	42	s	86	m	131	m	177	s
6	s	43	s	87	m	132	s	178	s
7	s	44	s	88	m	133	s	179	s
8	s	45	s	89	m	134	s	180	s
9	s	46	s	90	m	135	s	183	s
10	m	47	s	91	s	136	m	185	s
11	m	48	s	92	s	137	s	187	s
12	m	49	s	93	m	138	s	189	s
13	m	51	m	94	m	139	s	190	m
15	s	52	s	95	m	141	s	191	s
16	s	53	m	96	s	144	s	193	s
17	m	54	m	97	s	146	s	194	s
18	s	55	s	99	m	147	m	195	s
19	s	56	m	101	m	149	m	196	m
20	m	57	m	102	s	151	s	197	s
21	m	59	s	103	s	153	s	198	s
22	s	60	m	105	m	154	m	199	s
23	m	63	s	109	m	156	s	200	s
24	m	64	s	110	s	157	s	201	s
25	s	66	s	114	s	158	s	202	s
26	s	67	s	115	s	163	s	203	s
27	m	70	s	117	m	164	s	205	s
28	s	71	s	118	m	166	s	206	s
29	s	74	s	119	s	167	s	207	s
31	m	75	s	122	s	169	s	208	s
32	s	76	m	123	m	170	s	209	s
33	m	77	m	124	s	171	s		
34	s	81	s	125	m	172	s		

*s - compound eluted singly; m - compound eluted with one or more other congeners.

Figure 2 Excellent separation and peak shape for PCBs in three Aroclor® mixes, using an Rtx®-PCB column.



Column:	Rtx®-PCB 40m, 0.18mm ID, 0.18µm (cat.# 41303)
Sample:	300ng/mL Aroclor® 1242/1254/1262 in hexane: Aroclor® 1242 (cat.# 32009), Aroclor® 1254 (cat.# 32011), Aroclor® 1262 (cat.# 32409)
Inj.:	1.0µL splitless (hold 0.75 min.), 4mm single gooseneck inlet liner (cat.# 20983)
Inj. temp.:	230°C
Carrier gas:	hydrogen, constant pressure
Linear velocity:	40cm/sec. @ 100°C
Oven temp.:	100°C (hold 1 min.) to 200°C @ 30°C/min., to 320°C @ 2°C/min. (hold 1 min.)
Det.:	ECD @ 330°C

Accurately Monitor Mercury-Sulfur-Nitrogen Compounds

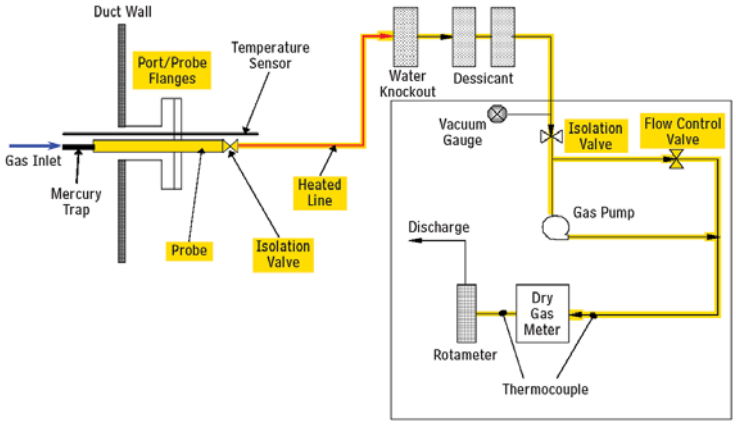
Siltek®/Sulfinert® Treatment Prevents Adsorption of Mercury, Sulfur Oxides, or Nitrous Oxides in Emission Monitoring Equipment

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

- Improved analytical reliability and sensitivity for mercury, SO_x, or NO_x compounds.
- Protection from corrosion—longer component lifetime.
- Apply to new or existing equipment.

The United States Environmental Protection Agency (US EPA) is actively developing regulations, limits, and control measures for monitoring and controlling mercury emissions from coal-fired power generators—one of the major sources of mercury emissions into the environment.[1] As these regulations and guidelines are developed and implemented, proper equipment will be needed for accurate sampling and analysis. Testing costs for mercury can be substantial (Table 1)[2], so inaccurate analyses can have financial as well as environmental repercussions.

Figure 1 Highlighted components of a mercury sampling train,[4] and all tubing in the system, can be Siltek®/Sulfinert® treated.



In flue streams from coal-fired power generators, mercury exists in three forms: elemental, the +2 oxidation state (Hg^{++}), and attached to particulate matter. Hg^{++} often reacts with sulfur compounds, nitrogen, chlorine, and/or oxygen, to produce sulfurous, nitrous, chloride, and oxide mercury species. Elemental and oxidized mercury can easily be lost to reactions and adsorption on the inner surfaces of monitoring equipment. In order to accurately sample and quantify mercury in all forms, it is important to use inert sample pathways. Laboratory testing and field results have proven that Sulfinert® treated sampling and testing equipment is essentially inert to active molecules[3], including mercury.

Siltek®/Sulfinert® treatment can be applied to many of the components in a mercury sampling stream, including probe tubing, impingers, fittings, filters, housings, and transfer tubing (Figure 1). Treating all of the components of a stack or continuous emission monitoring system will greatly improve analytical reliability and sensitivity, which will be needed as regulations are brought on line and emission quotas are enforced. Fast and accurate testing, without re-work, can save a great deal of time and money.

Similarly, a Siltek®/Sulfinert® treated sampling system will improve the reliability of data for sulfurous oxides and nitrous oxides (SO_x and NO_x). As with mercury, it is difficult to reliably transfer these compounds through untreated sampling equipment.

In addition to preventing adsorption of reactive compounds, Siltek®/Sulfinert® treatment will act as a barrier, protecting and prolonging the lifetime of treated equipment. The durable layer will withstand temperatures to 400°C.

We offer Siltek®/Sulfinert® treated tubing, sample cylinders, and other components from stock; to discuss custom treatment of system components, please contact the Restek Performance Coatings team.

Restek offers treated and untreated tubing, fittings, and valves, passive air sampling kits, air sampling canisters and miniature air canisters, sample loops, and more. For more information, request our catalog or visit us online. **www.restekcoatings.com**

Table I Typical costs of mercury sampling (U.S.).[2]

Method	Approx. Cost of Analysis
US EPA 29	$300
US EPA 101A	$100
ASTM D6784-02	$250
US EPA 324	$430
FAMS	$640

References

1. Pottinger, M., S. Stecklow, and J.J. Fialka, *Invisible Export, A Hidden Cost of China's Growth: Mercury Migration* The Wall Street Journal Online, Dec. 17, 2004.
2. Serne, J.C., *An Overview and Comparison of Available Mercury Emission Test Methods for Boilers* Symposium on Air Quality Measurement; Methods and Technology 2005, San Francisco, CA; Air & Waste Management Association. paper no. 439, pg. 9.
3. Barone, G., M. Higgins, D. Smith, S. Rowan, W.J. Gross, and P. Harris, *The Surface for Sulfurs* Hydrocarbon Engineering, Dec. 2004, pp 47-50.
4. Proposed Method 324. *Determination of Vapor Phase Flue Gas Mercury Emissions from Stationary Sources Using Dry Sorbent Trap Sampling* United States Environmental Protection Agency. Washington, D.C. p. 5.

Identify and Quantify Adulterants in Seized Cocaine

Using GC/MS (Rtx®-440 Column) and HPLC/RI (Pinnacle II™ Amino Column)

By Kristi Sellers, Clinical/Forensic Innovations Chemist, and Rick Morehead, R&D GC Column Group Leader

- Low bleed Rtx®-440 column improves resolution and inertness for adulterants by GC/MS.
- GC/MS provides positive identification for all adulterants except sugars; data can be used as evidence.
- HPLC is the preferred chromatographic method for identifying sugars as adulterants.

Illicit cocaine is commonly "cut" with adulterants or diluents to increase the amount of product available for sale. Because the composition of an illicit cocaine mixture can be specific to one dealer, identification of adulterants and diluents in seized cocaine is critical in determining the possible routes of distribution and sales.

Either GC or HPLC can be used to identify cocaine adulterants such as sugars, anesthetics, analgesics, and stimulants. GC is the most common analytical technique used for analyzing all cocaine adulterants except sugars. Although sugars can be derivatized for analysis by GC, they are more easily detected using HPLC.

GC

Cocaine mixture components can be detected using flame ionization detection (FID, Figure 1), nitrogen-phosphorus detection (NPD), or mass spectrometry (MS). Although FID or NPD provide good sensitivity for the adulterants, GC/MS is the most widely accepted detection method. MS is very sensitive, provides positive identifications based on mass spectra, and MS data are accepted as confirming evidence in courts of law.

Among the column types we evaluated, only Rtx®440 columns resolved lidocaine and caffeine to baseline (Figure 1). To evaluate the columns, we prepared mock samples of illicit cocaine by adding equal concentrations of a variety of adulterants and diluents to cocaine hydrochloride. We used stimulants, including caffeine, local anesthetics, such as lidocaine, and over-the-counter analgesics, such as phenacetin, and followed a simple "dilute and shoot" sample preparation scheme to dissolve the samples for analysis.

We developed a GC/MS method that enabled us to identify each adulterant or diluent, focusing on maximizing resolution while minimizing total analysis time in order to increase sample throughput. In the optimized GC/MS method (Figure 2), total analysis time was 6.5 minutes. Unlike in the GC/FID analysis (Figure 1), caffeine and lidocaine were not resolved to baseline, but were resolved by approximately 40% (Figure 2), due to MS vacuum effects on sample flow through the column. Caffeine and lidocaine have very different mass spectra, however, and extracted ion analysis ensured

Figure 1 An Rtx®-440 column resolves lidocaine/caffeine, and other cocaine adulterants, to baseline.

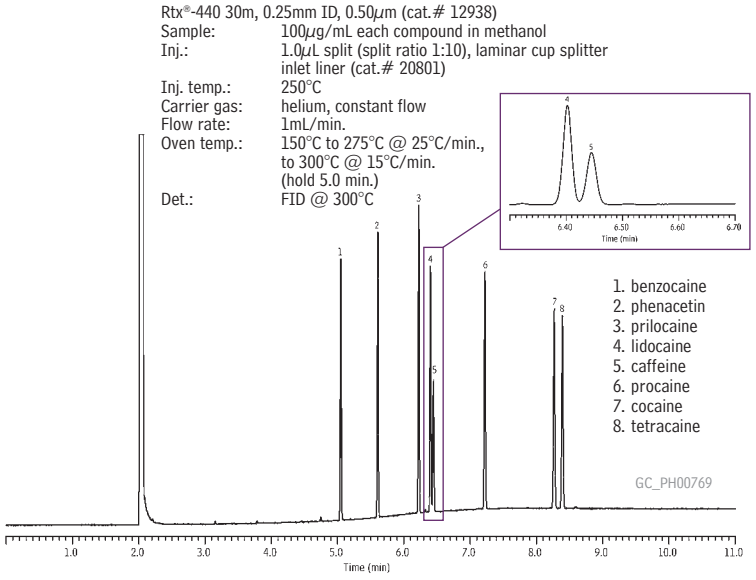
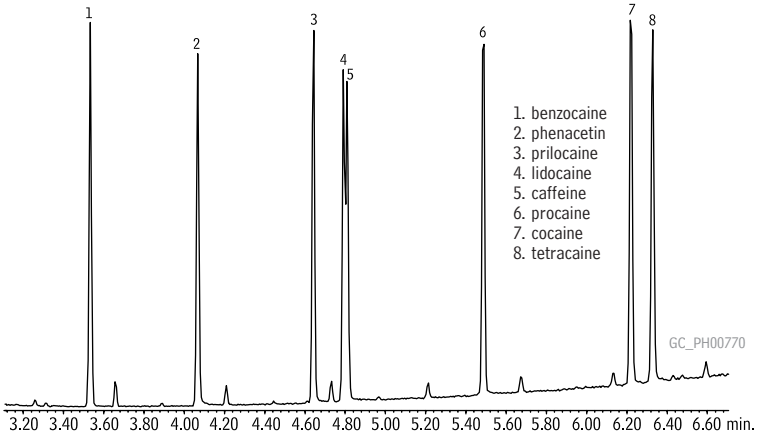
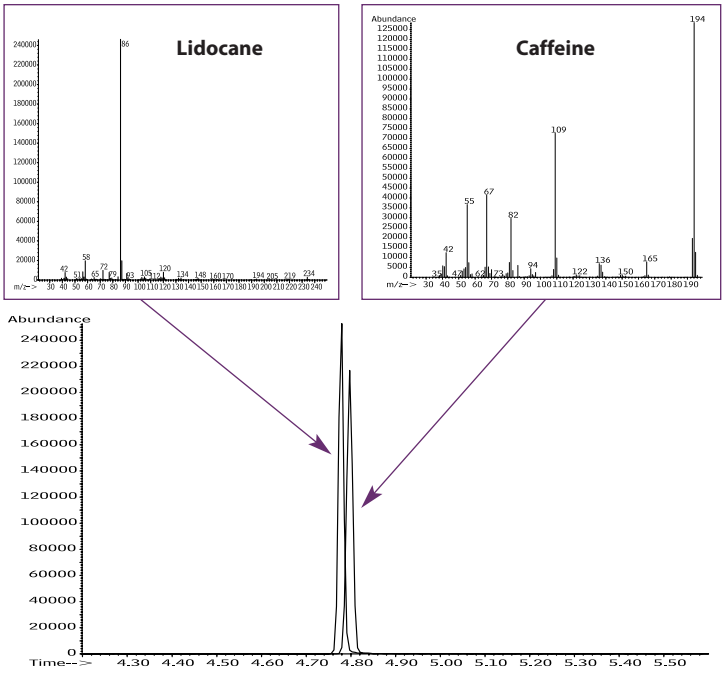


Figure 2 Analyze cocaine adulterants in 6.5 minutes, using an Rtx®-440 column in a GC/MS analysis.



Column:	Rtx®-440 30m, 0.25mm ID, 0.50µm (cat.# 12938)
Sample:	100µg/mL each compound in methanol
Inj.:	1.0µL split (split ratio 1:10), laminar cup splitter inlet liner (cat.# 20801)
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1mL/min.
Oven temp.:	150°C to 275°C @ 25°C/min., to 300°C @ 15°C/min. (hold 5.0 min.)
Det.:	MS
Transfer line temp.:	180°C
Scan range:	35-550amu
Ionization:	EI
Mode:	scan

Figure 3 Distinctive mass fragments ensure positive identification of lidocaine and caffeine.



Column:	Rtx®-440 30m, 0.25mm ID, 0.50µm (cat.# 12938)
Sample:	100µg/mL each compound in methanol
Inj.:	1.0µL split (split ratio 1:10), laminar cup splitter inlet liner (cat.# 20801)
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1mL/min.
Oven temp.:	150°C to 275°C @ 25°C/min., to 300°C @ 15°C/min. (hold 5.0 min.)
Det.:	MS
Transfer line temp.:	180°C
Scan range:	35-550amu
Ionization:	EI
Mode:	scan

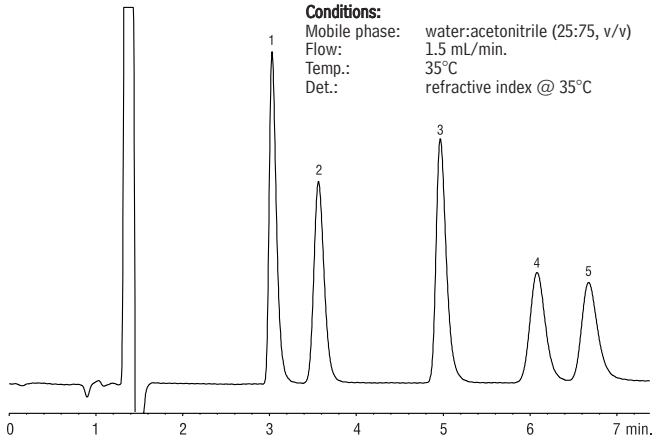
Figure 4 Pinnacle II™ Amino column provides fast, reliable analyses for sugars.

Peak List:	Conc. (mg/mL)
1. fructose	2.0
2. glucose	2.1
3. sucrose	4.0
4. maltose	4.5
5. lactose	4.4

Sample:
Inj.: 5µL
Solvent: mobile phase

Column: **Pinnacle II™ Amino**
Cat. #: 9217365
Dimensions: 150 x 4.6mm
Particle size: 3µm
Pore size: 110Å

Conditions:
Mobile phase: water:acetonitrile (25:75, v/v)
Flow: 1.5 mL/min.
Temp.: 35°C
Det.: refractive index @ 35°C



positive identification and allowed quantification of each compound. Lidocaine and caffeine have distinctive mass fragments of 86m/z and 194m/z, respectively (Figure 3).

HPLC

Sugars are not easily volatilized and, therefore, are difficult to analyze by GC, making HPLC the better chromatographic approach for this analysis. Further, refractive index (RI) or evaporative light-scattering (ELS) detection must be used because sugars have no UV chromophore. HPLC/RI or HPLC/ELS provides reproducible retention times, adequate peak identification and good quantification for sugars, as shown in Figure 4.

HPLC/MS methods for simultaneous analysis of cocaine, sugars, and other classes of adulterants and diluents have not yet been developed, but such methods would enable analysts to evaluate street cocaine mixtures in one analysis. Column parameters and mobile phase composition will be critical parameters to optimize.

Conclusions

Cocaine samples can be "fingerprinted" by identifying and quantifying the adulterants and diluents mixed with the drug. GC/MS provides adequate quantitative information about the concentration of each additive, relative to the cocaine concentration, and provides undisputable identification of a substance (retention time and mass spectrum data). Therefore, GC/MS is the preferred chromatographic method for analyzing cocaine and most cocaine adulterants. Sugars are best analyzed by HPLC.

for more information

Smith, F.P, *Handbook of Forensic Drug Analysis*, pp.235-275, Elsevier, 2005.

Telepchak, M.J., T.F. August, and G. Chaney, *Forensic and Clinical Applications of Solid Phase Extraction*, pp.204-213, Humana Press, 2004.

Rtx®-440 (fused silica)

(proprietary intermediate-polarity Crossbond® phase)

ID	df (µm)	temp. limits	length	cat. #
0.25mm	0.25	20°C to 320/340°C	30-Meter	12923
0.25mm	0.50	20°C to 320/340°C	30-Meter	12938

Pinnacle II™ Amino

3µm Particles, 4.6mm ID	cat. #
150mm	9217365

Carbohydrate HPLC Performance Check Mix

Dry components in 4mL screw-cap vial.
Reconstitute in 1mL acetonitrile:water (75:25) to 2.0, 2.1, 4.4, 4.5, 4.0 mg/mL, respectively.

glucose	2.0mg	maltose	4.5
fructose	2.1	sucrose	4.0
lactose	4.4		

cat. # 31809 (ea.)

No data pack available.

tech tip

We recommend using an HPLC guard column for this application. For Trident™ guard column systems, refer to our catalog, or visit our website at **www.restek.com/hplc**.

New HPLC Confirmation Column for Explosives Analysis

Introducing the Pinnacle II™ Biphenyl Column

By Becky Wittrig, Ph.D., HPLC Product Marketing Manager, Randy Romesberg, HPLC Applications Chemist, and Mike Wittrig, R&D Chemist

- Excellent resolution of US EPA Method 8330 explosives.
- Significantly different selectivity, relative to C18 columns; better resolution than cyano columns.
- Allows quantitative as well as qualitative confirmation.

Testing of residual materials is important when monitoring the disposal of expired or deteriorated munitions. US EPA Method 8330 was developed for quantifying 14 commonly monitored explosives. The method calls for reversed phase HPLC with UV detection, using a primary column and a confirmation column. The primary column contains a C18 stationary phase and, typically, the confirmation column contains a cyano stationary phase. Resolution of the target explosives is poor on cyano stationary phases, however, and the analysis provides qualitative confirmation only.

Restek chemists have developed a superior alternative to cyano phases for explosives analysis. The Pinnacle II™ Biphenyl column provides excellent resolution of EPA Method 8330 explosives, as shown in Figure 1. Further, selectivity is markedly different from that of a C18 column (Figure 2), making the Pinnacle II™ Biphenyl column a true, ideal, confirmation column. Separations on either column are accomplished with a simple, isocratic water:methanol mobile phase.

Restek offers a complete set of analytical reference materials for Method 8330. Our calibration materials for explosives analysis by HPLC are available in two options: as 1000ppm solutions of individual analytes, or as two 7-component mixtures, described on page 7. The internal standard, 3,4-dinitrotoluene, and the surrogate standard, 1,2-dinitrobenzene, also are available as described on page 7.

For superior data from your confirmation analysis for explosives, we highly recommend a Pinnacle II™ Biphenyl HPLC column.

Pinnacle II™ Biphenyl

5µm Particles, 4.6mm ID	cat. #
150mm	9209565

Ultra C18 Columns

5µm Particles, 4.6mm ID	cat. #
250mm	9174575

For individual solutions of EPA Method 8330 analytes, please see our catalog, or visit our website.

Figure 1 Excellent resolution of US EPA Method 8330 explosives, using a Pinnacle II™ Biphenyl column.

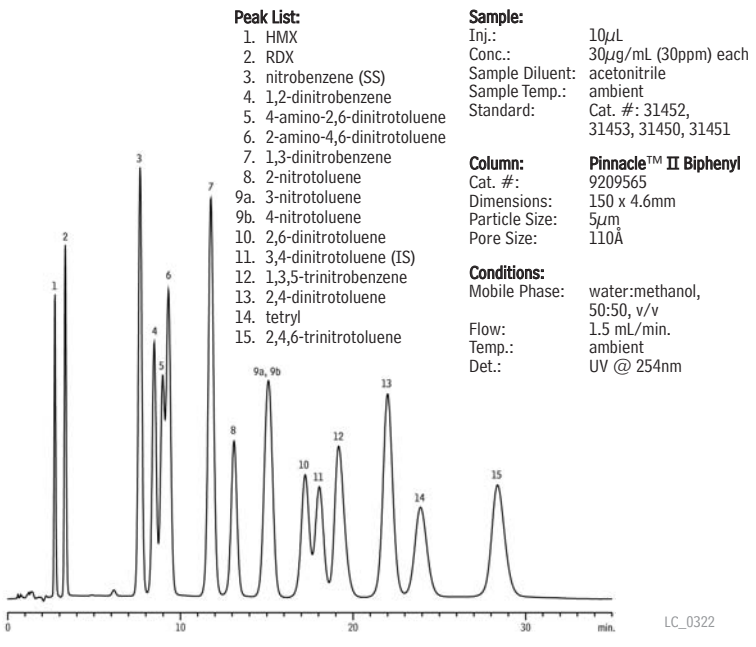
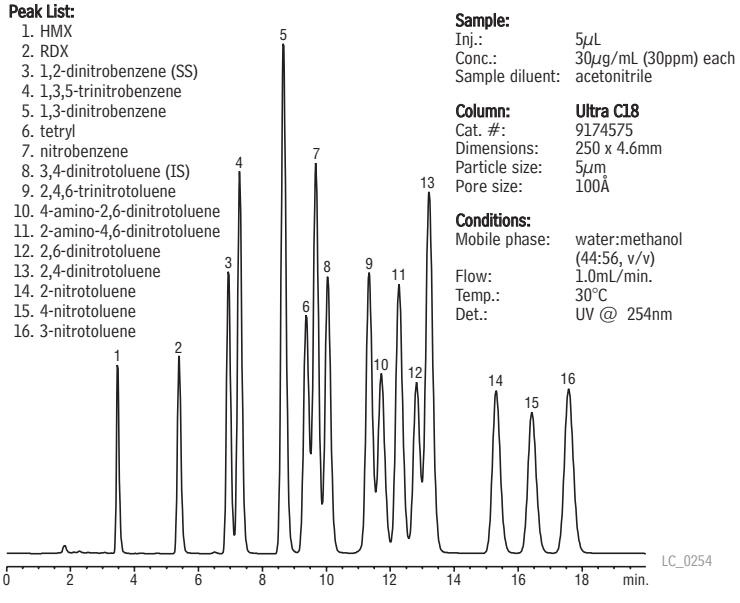


Figure 2 An Ultra C18 column is an outstanding primary column for explosives analysis.



Analytical Reference Materials

High-Purity Explosives - On-Line Data Packs - Custom Mixes

By Ken Herwehe, Analytical Reference Materials Product Marketing Manager

High-Purity Reference Materials for Explosives

HPLC with UV detection is used to measure nitroaromatic and nitramine explosives and their degradation products in water and soil samples.[1] Obtaining pure, neat compounds for these standards can be very difficult. Some of these commercial-grade materials contain desensitizing agents such as beeswax, water, or other manufacturing by-products. Many are shipped wet and must be carefully dried before preparation. To ensure the highest quality standards, Restek chemists carefully purify or synthesize each compound to 98% pure or higher.

Reference

1 US Environmental Protection Agency. *Test Methods for Evaluating Solid Waste*, Physical/Chemical Methods. SW-846 Update III, Office of Solid Waste, Washington, DC, 1997.
(Reference not available from Restek.)

8330 Calibration Mix #1 (7 components)

1,3-dinitrobenzene	RDX
2,4-dinitrotoluene	1,3,5-trinitrobenzene
HMX	2,4,6-trinitrotoluene
nitrobenzene	

1,000µg/mL each in acetonitrile, 1mL/ampul
cat. # 31450 (ea.)

8330 Calibration Mix #2 (7 components)

2-amino-4,6-dinitrotoluene	3-nitrotoluene
4-amino-2,6-dinitrotoluene	4-nitrotoluene
2,6-dinitrotoluene	tetryl
2-nitrotoluene	

1,000µg/mL each in acetonitrile, 1mL/ampul
cat. # 31451 (ea.)

8330 Internal Standard

3,4-dinitrotoluene

1,000µg/mL in methanol, 1mL/ampul
cat. # 31452 (ea.)

8330 Surrogate

1,2-dinitrobenzene

1,000µg/mL in methanol, 1mL/ampul
cat. # 31453 (ea.)

8330 Nitroaromatics Kit

31450: 8330 Calibration Mix #1
31451: 8330 Calibration Mix #2
31452: 8330 Internal Standard Mix
31453: 8330 Surrogate Mix

Contains 1mL each of these mixtures.
cat. # 31454 (kit)

For individual solutions of these analytes, please see our catalog, or visit our website.

free data packs

Restek now offers free downloadable data packs for analytical reference material products. Just visit our website at **www.restek.com/datapacks**. Enter the catalog number and lot number for the product you ordered and obtain a printable PDF file.

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Restek, "the company chromatographers trust™", should be your first choice for custom-made reference materials. Maximum convenience, maximum value, minimum time spent blending calibration mixtures in your laboratory.

- Quotations supplied quickly.
- Mixtures made to your EXACT specifications.
- We have over 2,000 pure, characterized, neat compounds in our inventory!

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Excellent Protein Separations from Viva™ HPLC Columns

Best Performance Among Five Tested Wide Pore Columns

By Bruce Albright, HPLC Chemist; Vernon Bartlett, HPLC Manager; Julie Kowalski, Foods, Flavors, and Fragrances Innovations Chemist; and Becky Wittrig, Ph.D., HPLC Product Marketing Manager

- Best overall performance among five columns evaluated.
- Best resolution and peak symmetry for test proteins.
- C18, C8, C4, and silica columns available; other phases on request.

Reversed phase HPLC is an important technique for separating large biomolecules, such as proteins and peptides. Analysts generally employ C18 stationary phases, because these typically provide the best separations of related compounds, such as genetic variants of a protein or complex tryptic digests. However, limitations often are encountered when analyzing samples containing complex mixtures of closely related analytes. Columns containing wide pore silica (e.g., 300Å) are designed specifically for large molecule analyses, addressing this need for more resolving power.

Developed on Viva™ wide pore silica, Viva™ HPLC columns have ideal performance characteristics for separating large molecules and biomolecules. Using a reversed phase test mix, we compared column efficiency, peak asymmetry, and retention for Viva™ C18 columns and four other C18 wide pore HPLC columns. The Viva™ C18 column ranked highest in retention and selectivity and produced the best peak symmetry measurements (Table I).

To determine overall separating power, retention, and peak shape, we evaluated each column with a protein test mix. The Viva™ C18 column provided excellent resolution and peak shapes, as Figure 1 shows.

300Å silicas enhance resolution of similar or related analytes for several reasons. Large pore materials can provide greater retention because higher molecular weight analytes can enter more of the pores and access more surface area. Theoretically, the more surface 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 yield the needed retention. Further, the mean pore diameter within the distribution (e.g., 250Å vs 350Å) can define the selectivity in some separations, by changing the elution order for certain analytes.

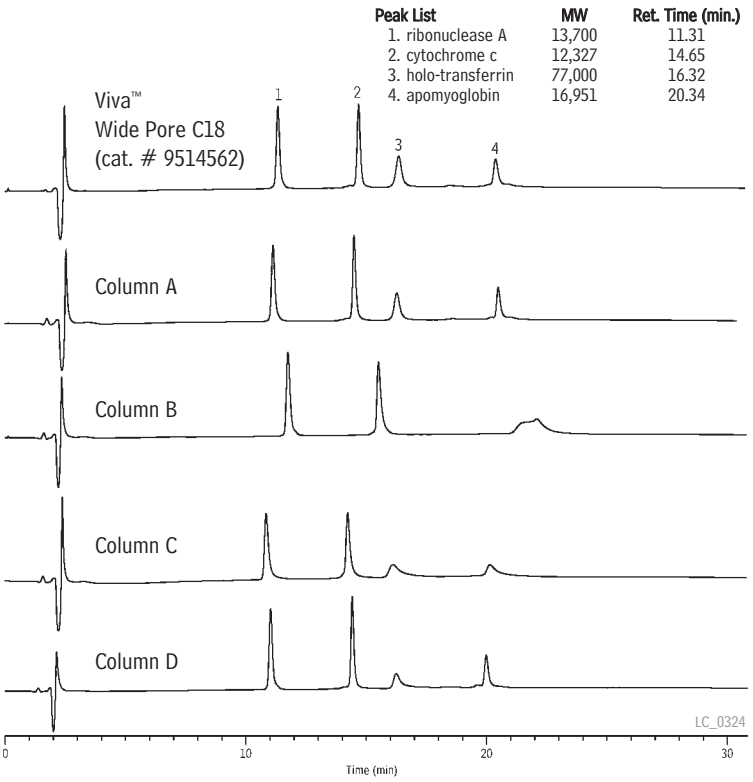
A 250-350Å mean pore diameter also is important because silicas with excessive numbers of pores smaller than 200Å can be more easily fouled by

Table I Viva™ wide pore C18 columns provide the best overall performance among five tested columns.

Column	Efficiency (plates/meter)	Asymmetry (biphenyl)	Retention Time (biphenyl)	Column Pressure (bar)
Viva™ 300 C18	>50,000	1.16	6.30	60
Column A C18	~50,000	1.46	5.77	72
Column B C18	>50,000	1.46	4.96	102
Column C C18	>50,000	1.30	5.89	66
Column D C18	<50,000	1.49	3.79	80

Reversed phase test mix; 150 x 2.1mm C18 phase columns, 5µm particles

Figure 1 Analysis of a four protein test mix shows the superior performance of the Viva™ C18 column.



Sample:
Inj.: 20µL
Conc.: 0.08mg/mL each protein
Sample diluent: 0.10% TFA in water / 0.10% TFA in acetonitrile, 80:20, v/v
Sample temp.: 25°C

Columns:
Dimensions: 150 x 2.1 mm
Particle size: 5µm

Conditions:
Mobile phase: A: 0.10% TFA in water, B: 0.10% TFA in acetonitrile, 20% B to 70% B in 30 min.
Flow: 0.20mL/min.
Temp.: 25°C (or ambient)
Det.: UV @ 214nm

larger molecular weight debris, and silicas with a high percentage of pores larger than 500Å can be impractically fragile for conventional HPLC applications. A narrow distribution around the mean pore diameter is advantageous; it better ensures that proper selectivity is maintained, and aids in separating closely related analytes that differ only slightly in hydrodynamic volume (molecular size in solution).

We developed and introduced Viva™ wide pore silica specifically to meet these challenging criteria. Among the materials we have tested, this new silica provides the greatest available surface area represented by 250-350 Å pores, with a highly desirable pore volume and pore diameter distribution (The Restek Advantage, 2005v1).

Superior physical characteristics and strong test performances show Viva™ HPLC columns are an excellent choice for analyzing proteins, peptides, or other large molecules or biomolecules. C18, C8, C4, and silica columns currently are available; other phases can be prepared on request. If you require a wide pore silica column for your analysis, we highly recommend new Viva™ columns.

Viva™ Wide Pore HPLC Columns

- Excellent for separating peptides or proteins.
- Rugged, spherical particles, with 300Å pore size.
- High proportion of pore/surface area available to large molecules.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
Viva™ Wide Pore C18 Columns, 5µm				
30mm	9514531	9514532	9514533	9514535
50mm	9514551	9514552	9514553	9514555
100mm	9514511	9514512	9514513	9514515
150mm	9514561	9514562	9514563	9514565
200mm	9514521	9514522	9514523	9514525
250mm	9514571	9514572	9514573	9514575
Viva™ Wide Pore Silica Columns, 5µm				
30mm	9510531	9510532	9510533	9510535
50mm	9510551	9510552	9510553	9510555
100mm	9510511	9510512	9510513	9510515
150mm	9510561	9510562	9510563	9510565
200mm	9510521	9510522	9510523	9510525
250mm	9510571	9510572	9510573	9510575

HPLC Reversed Phase Test Mix #1

Routine analysis using this product can assist in determining the need to perform column/system maintenance.

benzene	3.00mg/mL	naphthalene	0.50
uracil	0.02	biphenyl	0.06

In methanol:water (75:25), 1mL/ampul

cat. # 35005 (ea.)

No data pack available.

Survival Kits for HPLC

Invaluable for Keeping Your System Running Smoothly!

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

- Tubing, fittings, and tools for system start-up or maintenance.
- Choose PEEK® or stainless steel components.
- More convenient and more economical than ordering components separately.

Restek HPLC survival kits contain practical selections of tubing, fittings, and tools for setting up or maintaining your HPLC system. The PEEK® Survival Kit contains PEEK® tubing, connectors, and elbows, Teflon® tubing, a tubing cutter and extra blades, a ValvTool wrench, open-end wrenches, and more. The Stainless Steel Survival Kit contains a selection of lengths and IDs of 1/16-inch tubing, plus nuts, ferrules, a ValvTool wrench, and a zero-dead-volume union.

did you **know**?

Restek offers a wide range of HPLC columns, tools, and accessories, and many replacement parts for Agilent, Beckman, Hitachi, PerkinElmer, Shimadzu, Thermo Separation, and Waters instruments. Call us for a copy of our latest HPLC catalog (lit. cat.# 59241B), or visit us on line.

PEEK® Survival Kit for HPLC

The PEEK® Survival Kit is an invaluable parts kit that contains tools and supplies essential for setting up and maintaining your HPLC system.

Description	qty.	cat.#
PEEK® Survival Kit for HPLC	kit	25322

Stainless Steel Survival Kit for HPLC

Contains a wide range of stainless steel tubing, plus fittings and a ValvTool wrench.

Description	qty.	cat.#
Stainless Steel Survival Kit for HPLC	kit	25097

Improve Storage Stability for Sulfur Compounds

Using Sulfinert® Treated Sample Cylinders

By Neil Mosesman, Air Monitoring Product Marketing Manager

- Eliminate sample-surface reactions in sample cylinders, collect and store active compounds.
- Obtain accurate data for active sulfur compounds at ppb levels.
- Many treated system components available from stock.

High-pressure sample cylinders are commonly used for collecting and storing refinery and natural gas samples containing trace amounts of sulfur compounds. These highly active compounds degrade very rapidly in stainless steel sample cylinders, making accurate determination of sulfur compounds virtually impossible. Restek's exclusive Sulfinert® surface treatment eliminates the reactivity of high-pressure sample cylinders and allows collection and stable storage of sulfur compounds, even at ppb levels. Figure 1 shows the recovery of 17ppbv hydrogen sulfide, carbonyl sulfide, methyl mercaptan, ethyl mercaptan, and dimethyl disulfide after 60 hours of storage in a Sulfinert® treated sample cylinder. The data show that these active compounds were unaffected by long-term storage in the Sulfinert® treated cylinder.

In addition to Sulfinert® treated cylinders, we also offer Sulfinert® treated valves, tubing, and sample loops to ensure the entire sample pathway is inert. Custom treatment is available for a wide range of items.

Sulfinert®-Treated Sample Cylinders

- Stable storage of low concentrations of sulfur compounds.
- D.O.T. rated to 1800psi at room temperature.
- 316 stainless steel, 1/4" female NPT threads on both ends.

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

Sulfinert®-Treated Sample Cylinder Valves and Rupture Discs

- All "wetted" valve parts are Sulfinert®-treated.
- Maximum pressure rating, 5000psi.

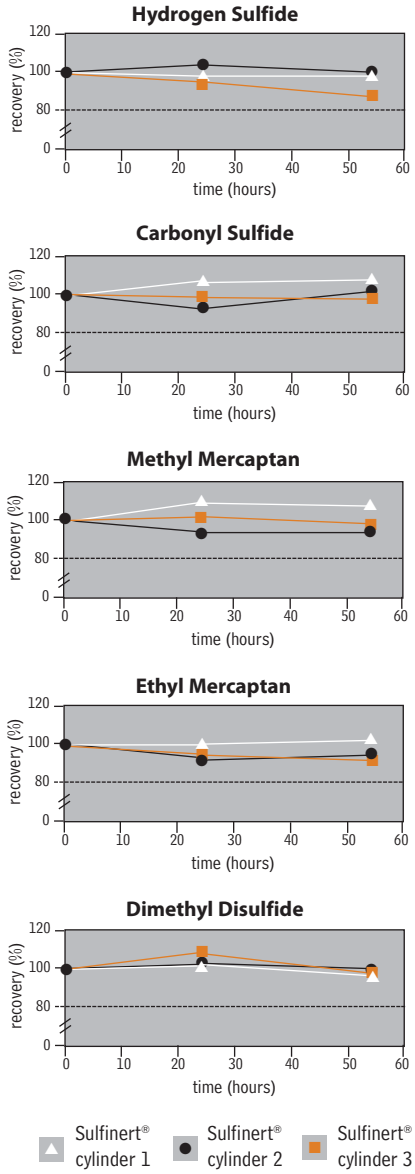
Description	qty.	cat.#
1/4" NPT Exit, Kel-F® Stem Tip	ea.	24127
1/4" Compression Exit, Kel-F® Stem Tip	ea.	24128
1/4" Female NPT Outlet (built-in rupture disc)	ea.	21395

Sulfinert®-Treated Gas Sample Loops

- Ideal for samples containing low concentrations of sulfur compounds.
- Sizes from 5µL to 5cc.; 1/16" fittings, for "W Type" valves.

Size	qty.	cat.#
5µL	ea.	22840
10µL	ea.	22841
20µL	ea.	22842
25µL	ea.	22843
50µL	ea.	22844
100µL	ea.	22845
250µL	ea.	22846
500µL	ea.	22847
1cc	ea.	22848
2cc	ea.	22849
5cc	ea.	22850

Figure 1 Active sulfur compounds are stable in Sulfinert® treated sample cylinders.



17ppbv each compound

for **more** info

Please request Applications Note **#59164B** for more details about storing sulfur compounds in Sulfinert® treated cylinders.

Analysis of Nitrofurans in Honey

Using LC/MS/MS and an Ultra C18 Column

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

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

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

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

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

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

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

Acknowledgement

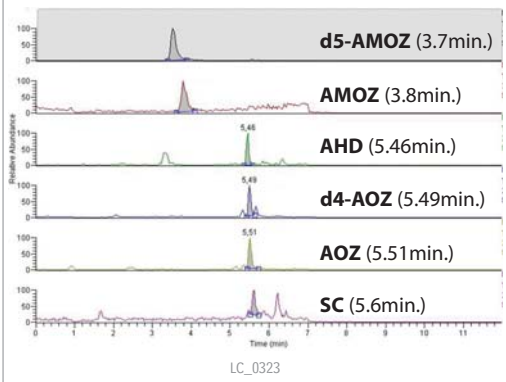
We are grateful to EIDOMET SRL, Restek distributor in Argentina, and application chemist Dr. Alejandro Albornoz, for the analytical work discussed in this article.

Ultra C18 Column

3µm Particles, 2.1mm ID	cat. #
100mm	9174312

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

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



LC_0323

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

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

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

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

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

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

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

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

FID Gas Stations: FID-1000 & New FID-2500

Convenient, Safe Source of Zero Air and Pure Hydrogen

By Kelli Ventura, GC Accessories Associate Product Manager

- Single unit produces zero grade air and 99.9995% pure hydrogen.
- Eliminates inconvenient and dangerous cylinders.
- Silent operation, minimal operator attention required.

Parker Balston FID-1000 and FID-2500 Gas Stations provide both UHP grade hydrogen fuel gas and zero grade air (<0.1ppm total hydrocarbons) for flame ionization detectors on gas chromatographs. The system is designed specifically to supply fuel gas to FIDs and to support flame thermionic and flame photometric detectors. The units produce zero air by purifying compressed air to a total hydrocarbon concentration of 0.1 ppm or less (measured as methane). The hydrogen generators produce hydrogen gas from deionized water, using the principle of electrolytic dissociation of water and hydrogen proton conduction through a proton exchange membrane cell.

These units are designed for universal operation. When ordering an FID Gas Station for use in countries other than the United States, simply add the appropriate international power cord suffix to the catalog number for the gas station.

Description	qty.	cat. #
Model FID-1000 Gas Station (ideal for 1 - 2 FIDs)	ea.	20177
Model FID-2500 Gas Station (ideal for 5 - 6 FIDs)	ea.	24913
Replacement Components for FID Gas Stations		
Resin Bed Cartridge for FID-1000 and FID-2500 Hydrogen Generators	ea.	24914
Desiccant Cartridge	ea.	21671
FID Gas Station Maintenance Kit (Includes 1 desiccant cartridge, 1 resin bed cartridge, 1 filter cartridge)	ea.	24915
International Power Cord Sets	**qty.**	**cat.# suffix**
United Kingdom (230VAC, 50/50Hz)	ea.	-550
European (230VAC, 50/60Hz)	ea.	-551
IEC Connector Only (230VAC, 50/60Hz)	ea.	-552
Japanese (200VAC, 50/60Hz)	ea.	-556
Japanese for Zero Air (100VAC, 50/60Hz)	ea.	-553
Japanese for Hydrogen (100VAC, 50/60Hz)	ea.	-554
Japanese for Nitrogen (100VAC, 50/60Hz)	ea.	-555

Just add the proper suffix to the catalog number for the gas generator you are ordering.

Specifications - FID Gas Stations:

Hydrogen Purity:	99.9995%
Zero Air Purity:	FID-1000: < 0.1ppm total hydrocarbons as methane FID-2500: < 0.05ppm total hydrocarbons as methane
Max. Hydrogen Flow Rate:	FID-1000: 90cc/min. FID-2500: 250cc/min.
Max. Zero Air Flow Rate:	FID-1000: 1000cc/min. FID-2500: 2500cc/min.
Power:	120VAC/amp, 60Hz, 400 watts
Hydrogen Outlet Pressure:	60 psig
Zero Air Outlet Pressure:	40-125 psig*
Inlet Connection:	1/4" NPT (female)
Outlet:	1/8" compression
Dimensions:	16.5"h x 10.5"w x 17"d (42cm x 27cm x 43cm)
Weight:	53 lbs. (24kg)

*Zero air inlet requires minimum of 40psig compressed air pressure.

built to **international** standards

Produced and supported by an ISO 9001 registered organization, Parker Balston hydrogen generators are built to meet the toughest laboratory standards - CSA, UL, CE, and IEC 1010.

Volatile Organic Compounds by GC/MS

Columns and Reference Mixes for US EPA 524.2 Revision IV.

By Christopher English, GC Innovations Group Leader, and Joseph Moodler, Analytical Reference Materials Technical Supervisor

- All 84 compounds listed in Method 524.2 resolved in 12 minutes, using an Rtx®-VMS column.
- MegaMix™ reference mix includes 73 compounds in stable solution.
- Three reference mixes include all 84 compounds.

Initially, US Environmental Protection Agency Method 524.2, a purge and trap, capillary GC/MS method, was used to identify 60 volatile aromatic and halogenated hydrocarbons in municipal drinking water. Revision 4.0 (1992) added 24 polar compounds and, in 2003, California allowed the addition of *tert*-amyl methyl ether (TAME), *tert*-butyl alcohol (TBA), ethyl-*tert*-butyl ether (ETBE), and 1,1,2-trichlorotrifluoroethane (Freon® 113) to the list of target compounds.

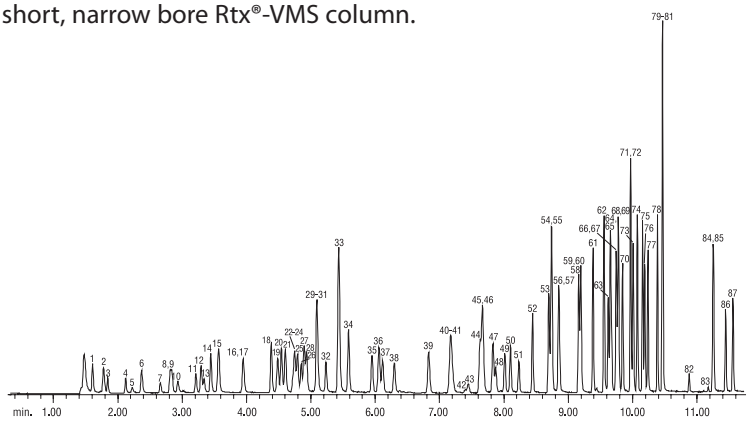
Of these 84 compounds, only the six gases and five ketones are not included in Drinking Water VOA MegaMix™, 524.2 Rev 4.1. To help ensure long-term stability of the mixes, we offer the ketones separately, in purge & trap methanol/water (90:10, v/v). This solvent system protects the keto groups and prevents acetal formation, which is more likely to occur in 100% methanol.

Table 1 Method 524.2 analytes and internal standards.

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. diethyl ether
8. 1,1-dichloroethene
9. carbon disulfide
10. iodomethane
11. allyl chloride
12. methylene chloride
13. acetone
14. *trans*-1,2-dichloroethene
15. methyl *tert*-butyl ether
16. 1,1-dichloroethane
17. acrylonitrile
18. *cis*-1,2-dichloroethene
19. 2,2-dichloropropane
20. bromochloromethane
21. chloroform
22. methyl acrylate
23. carbon tetrachloride
24. tetrahydrofuran
25. 1,1,1-trichloroethane
26. 2-butanone
27. 1,1-dichloropropene
28. 1-chlorobutane
29. propionitrile
30. methacrylonitrile
31. benzene
32. 1,2-dichloroethane
33. fluorobenzene (IS)
34. trichloroethene
35. dibromomethane
36. 1,2-dichloropropane
37. bromodichloromethane
38. methyl methacrylate
39. *cis*-1,3-dichloropropene
40. toluene
41. chloroacetonitrile
42. 2-nitropropane
43. 1,1-dichloro-2-propanone
44. 4-methyl-2-pentanone
45. tetrachloroethene
46. *trans*-1,3-dichloropropene
47. 1,1,2-trichloroethane
48. ethyl methacrylate
49. dibromochloromethane
50. 1,3-dichloropropane
51. 1,2-dibromoethane
52. 2-hexanone
53. chlorobenzene
54. ethylbenzene
55. 1,1,1,2-tetrachloroethane
56. *m*-xylene
57. *p*-xylene
58. *o*-xylene
59. styrene
60. bromoform
61. isopropylbenzene
62. 4-bromofluorobenzene (IS)
63. bromobenzene
64. *n*-propylbenzene
65. 1,1,2,2-tetrachloroethane
66. 2-chlorotoluene
67. 1,2,3-trichloropropane
68. 1,3,5-trimethylbenzene
69. *trans*-1,4-dichloro-2-butene
70. 4-chlorotoluene
71. *tert*-butylbenzene
72. pentachloroethane
73. 1,2,4-trimethylbenzene
74. *sec*-butylbenzene
75. *p*-isopropyltoluene
76. 1,3-dichlorobenzene
77. 1,4-dichlorobenzene
78. *n*-butylbenzene
79. hexachloroethane
80. 1,2-dichlorobenzene-d4 (IS)
81. 1,2-dichlorobenzene
82. 1,2-dibromo-3-chloropropane
83. nitrobenzene
84. hexachlorobutadiene
85. 1,2,4-trichlorobenzene
86. naphthalene
87. 1,2,3-trichlorobenzene

Peaks 1-6 are components of cat. # 30439; peaks 13,26,43,44,52 are components of cat. # 30602; all other analytes except IS are components of cat. # 30601.

Figure 1 12-minute analysis of 84 volatile compounds, using a short, narrow bore Rtx®-VMS column.



Purge and Trap Conditions:

Concentrator:	Tekmar LSC-3100 purge and trap
Trap:	Vocarb 3000 (type K)
Purge:	11 min. @ 40 mL/min. @ ambient temperature.
Dry purge:	1 min. @ 40mL/min. (MCS bypassed using Silcosteel® tubing)
Desorb preheat:	245°C
Desorb:	250°C for 2 min., flow 10mL/min.
Bake:	260°C for 8 min.
Interface:	Silcosteel® transfer line 1:30 split at injection port. 1mm ID split injection sleeve (cat.# 20972).
Column:	Rtx®-VMS, 30m, 0.25mm ID, 1.4µm (cat.# 19915)
Sample:	502.2 Calibration Mix #1 (cat.# 30042) Drinking Water VOA MegaMix™, 524.2 Rev 4 (cat.# 30601) 524 Internal Standard/Surrogate Mix (cat.# 30201) Ketone Mix, EPA Method 524.2 Rev 4.1 (cat.# 30602) Compounds at 20 ppb each in 5mL RO water (ketones at 50ppb; internal standards at 40ppb)
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1.1mL/min.
Dead time:	1.48 min. @ 40°C
Oven temp.:	40°C (hold 2 min.) to 85°C @ 14°C/min. (hold 2 min.) to 220°C @ 30°C/min. (hold 4 min.).
Det:	Agilent 5971A GC/MS
Transfer line temp.:	280°C
Scan range:	35-300 amu
Tune	PFTBA/BFB
Ionization:	EI

A 30m, 0.25mm ID, 1.4µm Rtx®-VMS capillary column (cat.# 19915) is an excellent choice for analyzing the 84 target compounds (Figure 1). This narrow bore column improves resolution of traditionally coeluting compounds, such as carbon tetrachloride / 1,1,1-trichloroethane, while shortening the analysis time. Analysis time is less than 12 minutes, and the cycle time is 16 minutes, which is well below the cycle time of a standard purge and trap system. This allows the fastest runtime attainable using a Tekmar 3100 purge and trap unit coupled to a single GC. A slower initial temperature ramp rate makes additional resolution possible.

We recommend using the 30m, 0.25mm ID column for best resolution of the target gases. At 20ppb in 5mL water, the gases are better than 90% resolved, using an initial temperature of 40°C (Figure 1). We encourage laboratories using either dual purge and trap technology or newer purge and trap systems with rapid cycle times to use a 20m, 0.18mm ID, 1.0µm Rtx®-VMS column for sub-10 minute runtimes.[1,2] Whatever your system for analyzing volatiles, we offer the columns, analytical standards, GC accessories, and technical knowledge to get your laboratory running these analyses quickly and accurately.

References

1. Butler J.C., E. Phillips, and M. Conoley Application Note AN9197, Thermo Electron Corporation, 2215 Grand Avenue Parkway, Austin, TX., 2003.
2. A.L. Hilling and G. Smith, Environmental Testing & Analysis, 10 (3),15-19, 2001.

Rtx®-VMS (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	1.00	-40 to 240/260°C	20-Meter	49914
0.25mm	1.40	-40 to 240/260°C	30-Meter	19915

Drinking Water VOA MegaMix™, 524.2 Rev. 4.1

(73 components—see Table 1)

2,000µg/mL each in P&T methanol, 1mL/ampul
cat. # 30601 (ea.)

Ketones Mix, 524.2 Rev. 4.1 (5 components)

acetone
2-butanone (MEK)
1,1-dichloro-2-propanone
2-hexanone
4-methyl-2-pentanone (MIBK)

5,000µg/mL each in 90% P&T methanol:10% water, 1mL/ampul
cat. # 30602 (ea.)

502.2 Calibration Mix #1 (gases)

bromomethane
chloroethane
chloromethane
dichlorodifluoromethane
trichlorofluoromethane
vinyl chloride

200µg/mL each in P&T methanol, 1mL/ampul
cat. # 30439 (ea.)

2,000µg/mL each in P&T methanol, 1mL/ampul
cat. # 30042 (ea.)

For individual solutions of *tert*-amyl methyl ether, *tert*-butyl alcohol, ethyl-*tert*-butyl ether, and 1,1,2-trichlorotrifluoroethane (Freon® 113), and for internal and surrogate standards, please see our catalog, or visit our website.

Instrument Innovations!

Restek is your #1 source for GC consumables and supplies

by Donna Lidgett, GC Accessories Product Marketing Manager

From Injector to Detector

Restek designs, develops, and markets OEM-equivalent parts and supplies for Agilent, PerkinElmer, Shimadzu, Thermo Finnigan or Varian GC systems. Restek consumables and parts meet or exceed OEM performance, helping you maintain optimum system performance and giving you the convenience and economy of one-stop shopping for all your GC needs.

Check out some of our newest consumables. . .

new! EPC Test Kit for Agilent 6890 GCs

- Kit includes 3 o-rings, 2 plugs, 1 mounting screw and 1 test block.

Description	Similar to Agilent part #	qty.	cat.#
EPC Test Kit for Agilent 6890 GCs	G1530-60960*	kit	24323

*Similar to Agilent part # G1530-60960, but not exact equivalent. Kits differ in parts.

Clear anodized aluminum and high-quality stainless steel.

new! Septum Nut for Shimadzu 17A & 2010 GCs

- One piece design for ease of installation and removal.

Description	Similar to Shimadzu part #	qty.	cat.#
Septum Nut for Shimadzu 17A & 2010 GCs	221-41286-00 221-44584-00	ea.	22079

High quality stainless steel.

new! FID Jet for PerkinElmer Auto SYS™ XL

Description	Similar to PE part #	qty.	cat.#
FID Jet for PerkinElmer Auto SYS™ XL	N6100361	ea.	23038

High quality stainless steel.

new! FID Capillary Column Adaptor for PerkinElmer Auto SYS™ XL

Description	Similar to PE part #	qty.	cat.#
For use with PE style capillary nuts			
FID Capillary Column Adaptor for PerkinElmer Auto SYS™ XL	N6120020	ea.	22608
For use with 1/16" compression style nuts			
FID Capillary Column Adaptor for PerkinElmer Auto SYS™ XL	—	ea.	22609

Clear anodized aluminum/ High quality stainless steel.

new! Septum Cap for PerkinElmer Auto SYS™ XL

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

High quality stainless steel.

Siltek®-treated version for increased inertness.

new! Injector Adaptor for PerkinElmer Auto SYS™ XL

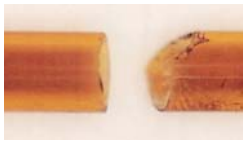
Description	Similar to PE part #	qty.	cat.#
For use with PE style capillary nuts			
Injector Adaptor for PerkinElmer Auto SYS™ XL	N6100157	ea.	22318
Siltek®-Treated Injector Adaptor for PerkinElmer Auto SYS™ XL	—	ea.	22320
For use with 1/16" compression style nuts			
Injector Adaptor for PerkinElmer Auto SYS™ XL	—	ea.	22319
Siltek®-Treated Injector Adaptor for PerkinElmer Auto SYS™ XL	—	ea.	22321

Cool Tools!

Restek Innovations Save You Time and Money

A Clean Square Cut...

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



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

Scoring Wafer with Handle

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

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

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

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

Make clean, square cuts!

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

Ceramic Scoring Wafers

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

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

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

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

Sapphire Scribe

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

One quick stroke...

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

Description	qty.	cat.#
Sapphire Scribe	ea.	20182

Shortix™ Capillary GC Column Cutter

- Consistently make precise, clean, square cuts with a diamond blade.
- Built-in magnifier to verify square cut.
- Use with 0.25mm ID to 0.53mm ID tubing (0.78mm OD max.).
- Maintenance kit includes diamond cutting wheel, O-rings, and a tool to open the column cutter.

Maintenance Kit for Shortix™ Capillary GC Column Cutter

Description	qty.	cat.#
Shortix Capillary GC Column Cutter	ea.	23026
Maintenance Kit for Shortix Capillary GC Column Cutter	kit	23027

Items of Interest

Compiled by the Advantage Staff

Back to School

Widely acclaimed Restek seminars deliver a wealth of information in a professional, non-promotional, multimedia format. Each full-day course is a mix of introductory through advanced elements, making these presentations suitable for novice chromatographers and for experienced analysts who want an introduction to the newest techniques, or a revisit to the fundamentals. Choose the topic and location that match your requirements—or, we'll come to your facility.

For seminar descriptions, please visit our website at **www.restek.com/seminars**.
For availability, please contact your Restek distributor

Detecting Adulterants in Butter

Investigators in the Dipartimento di Scienza degli Alimenti, Università degli Studi di Napoli "Federico II" (Via Università, 100-80055 Portici (NA), Italy) have proposed a new analytical method for detecting extraneous animal fats or vegetable oils added to butter.

Application of a HRGC Method on Capillary Column Rtx® 65-TG for Triglyceride Analysis to Monitor Butter Purity, by Daniele Naviglio and Carlo Raia, was published in Analytical Letters, Vol. 36, No. 14, 2003 (pages 3063-3094). Relative to the official EU method for detecting added animal fats, such as lard or tallow, the authors propose the new method is simple, rapid, and precise, even when quantities of added fats are minimal. The new method is easy to follow, even by nonspecialists. Naviglio and Raia developed their method using a 30m, 0.25mm ID, 0.25um Rtx®-65TG Restek column.

Contact the authors at their university address, above, or by e-mail: **danielenaviglio@inwind.it**

Order the article from : **www.dekker.com/servlet/product/DOI/101081AL120026422#abstract**

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Turning Visions into Reality™

2005 vol. 3

Versatile GC Columns for Forensics

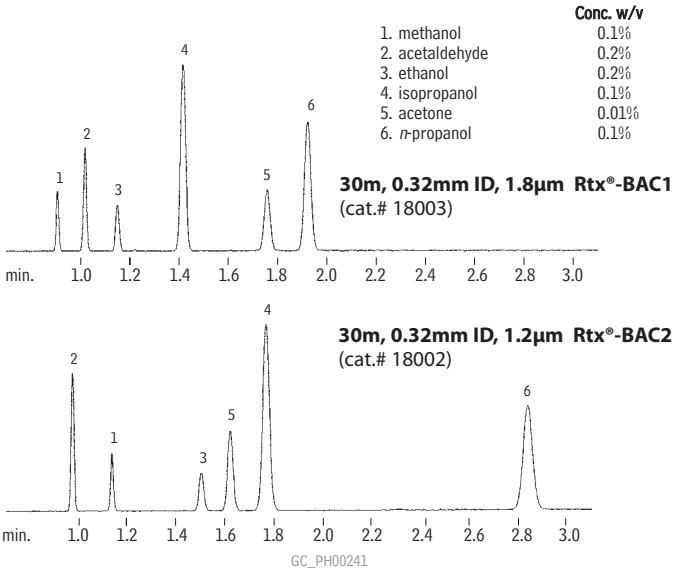
Use an Rtx®-BAC1 / Rtx®-BAC2 Column Set To Evaluate Blood Alcohol, Abused Substances, and Other Materials

By Kristi Sellers, Clinical/Forensic Innovations Chemist

- Unique column set for resolving/ confirming abused substances.
- 3-minute analysis for blood alcohols.
- Reliable data for ethylene glycol or GHB.

Analytical toxicology laboratories assist in criminal investigations by performing analyses for abused substances or poisons. Additional responsibilities include testing for accelerants or explosive materials in investigations of fires or explosions and assessing occupational/environmental exposure. Volatile and non-volatile compounds commonly tested for include blood alcohols, alkyl nitrites, anesthetics, inhalants, glycols, gamma-hydroxybutyrate (GHB), industrial solvents, petroleum hydrocarbons, and nitrogen-containing explosives.

Figure 1 Baseline resolution of all blood alcohol components in less than 3 minutes, using Rtx®-BAC1 and Rtx®-BAC2 columns.



Dual-column analysis using a two-hole ferrule.
1.0mL headspace sample of a blood alcohol mix on a PerkinElmer HS 40 headspace autosampler

Oven temp.:	40°C	Vial sampling time:	0.01 min.
Inj. temp.:	200°C	Transfer line:	0.32mm ID Hydroguard™ fused silica tubing
Carrier gas:	helium		
Sample equilibration:	70°C, 15 min.	Transfer line temp.:	200°C
Vial pressure:	30psi	Injection port sleeve:	2mm ID
Vial pressurization time:	0.15 min.	Split flow:	20mL/min.

in this issue

Where We'll Be in October

October 17-21, 2005
Society of Forensic Toxicologists (SOFT), Renaissance Nashville Hotel, Nashville, TN, booth #19

October 18-20, 2005
Gulf Coast Conference, Moody Gardens Convention Center, Galveston Island, TX

Help Us Celebrate Our 20th Birthday! Visit us at Booth 707, and receive a FREE Restek 20th Anniversary Travel Mug and Tote Bag!

October 25-27, 2005
ISA Expo 2005, McCormick Place Lakeside Center, Chicago, IL, booth #1322

Correction

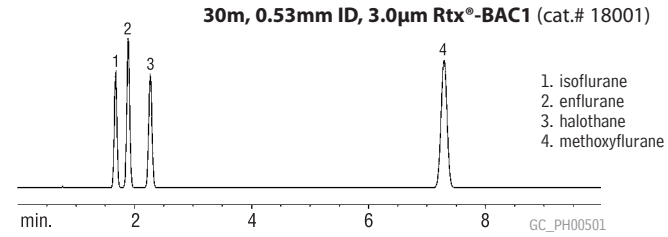
In *Advantage 2005v2, page 11*: Analysis of Nitrofurans in Honey. Mobile phase components A and B contain acetic acid, not formic acid. Honey samples containing nitrofuran metabolites were dissolved in 125mM HCl, derivatized with 2-nitrobenzaldehyde, extracted, evaporated, and reconstituted with mobile phase.

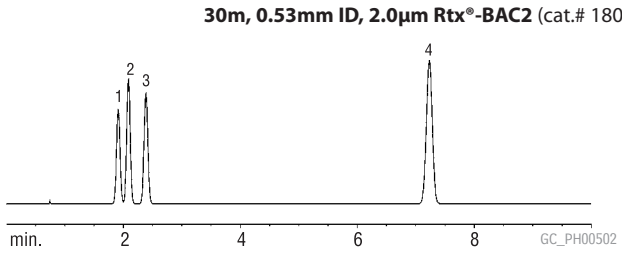
Table I Retention Times for Abused Substances

Compound	Rtx®-BAC1 Elution Order	Rtx®-BAC1 Ret.Time (min.)	Rtx®-BAC2 Elution Order	Rtx®-BAC2 Ret. Time (min.)
methanol	1	1.017	5	1.237
acetaldehyde	2	1.146	1	1.063
ethyl chloride	3	1.275	2	1.071
ethanol	4	1.299	8	1.648
diethyl ether	5	1.574	4	1.167
isopropanol	6	1.607	15	1.945
isoflurane	7	1.661	13	1.922
methylene chloride	8	1.805	11	1.849
Freon® 113	9	1.864	3	1.145
enflurane	10	1.891	16	2.081
tert-butyl alcohol	11	1.926	17	2.154
acetone	12	1.992	10	1.787
acetonitrile	13	1.997	20	2.553
n-propanol	14	2.191	25	3.130
halothane	15	2.267	18	2.383
methyl *tert*-butyl ether	16	2.366	7	1.554
hexane	17	2.495	6	1.386
tert-butyl nitrite	18	2.736	9	1.750
chloroform	19	2.870	27	3.290
sec-butyl alcohol	20	2.962	30	3.793
isobutyl nitrite	21	2.973	12	1.853
sec-butyl nitrite	22	3.059	14	1.939
isobutyl alcohol	23	3.460	32	5.100
tetrahydrofuran	24	3.736	24	2.845
methyl ethyl ketone	25	3.768	26	3.271
ethyl acetate	26	3.800	23	2.785
carbon tetrachloride	27	3.842	21	2.565
1,1,1-trichloroethane	28	3.869	22	2.729
n-butyl nitrite	29	3.879	19	2.469
benzene	30	4.186	28	3.392
n-butyl alcohol	31	4.565	33	6.747
trichloroethylene	32	5.205	31	4.084
isoamyl nitrite	33	6.377	29	3.728
methoxyflurane	34	7.279	36	7.219
isoamyl alcohol	35	7.428	38	9.447
toluene	36	8.358	34	6.944
1,1,2-trichloroethane	37	8.498	39	10.138
methyl isobutyl ketone	38	9.510	37	7.964
tetrachloroethylene	39	9.681	35	7.081
chlorobenzene	40	11.810	41	11.012
ethylbenzene	41	12.279	40	10.704
p-xylene	42	12.726	42	11.038
m-xylene	43	12.727	43	11.046
o-xylene	44	13.733	44	12.280
tetrachloroethane	45	14.106	50	16.968
isopropylbenzene	46	14.845	46	12.962
n-propylbenzene	47	15.966	47	14.124
1,3,5-trimethylbenzene	48	16.565	48	14.711
decane	49	17.166	45	12.369
1,2,4-trimethylbenzene	50	17.586	49	15.904
butylbenzene	51	19.739	51	17.732
tetradecane	52	29.806	52	24.950

30m, 0.53mm ID, 3.0µm Rtx®-BAC1 (cat.# 18001) and
30m, 0.53mm ID, 2.0µm Rtx®-BAC2 (cat.# 18000).
1.0mL headspace sample
Oven temp.: 40°C (hold 5 min.) to 240°C @ 5°C/min.
Inj. & det. temp.: 240°C
Carrier gas: He
Linear velocity: 65cm/sec.

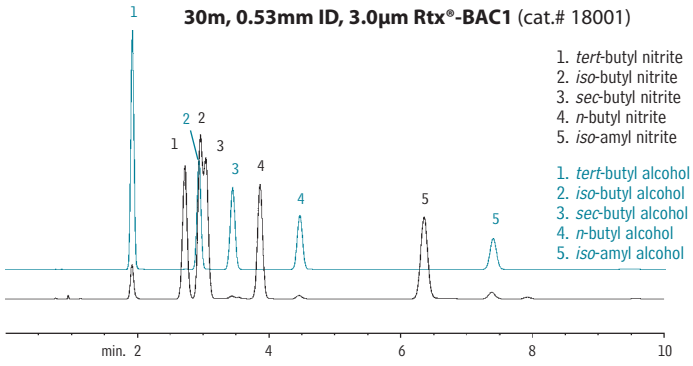
Figure 2 Rapid analysis for volatile anesthetics using Rtx®-BAC1 and Rtx®-BAC2 columns.

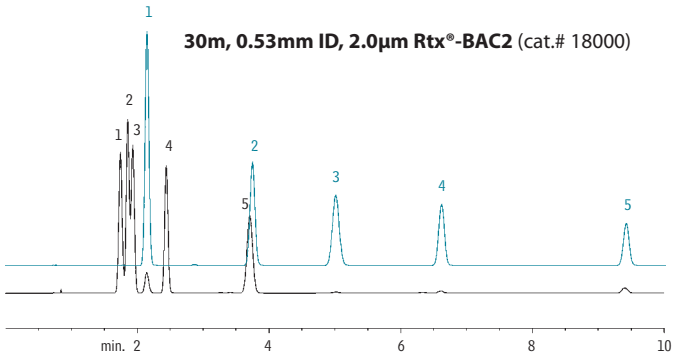




1.0mL headspace sample
Oven temp.: 40°C (hold 5 min.) to 240°C @ 5°C/min.
Inj. & det. temp.: 240°C
Carrier gas: helium
Linear velocity: 65cm/sec.

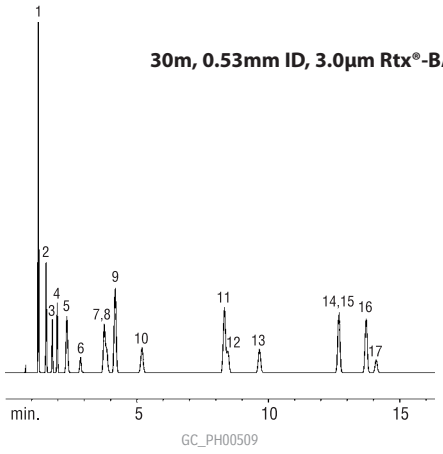
Figure 3 Screen for and confirm alkyl nitrites and metabolites on Rtx®-BAC1 and Rtx®-BAC2 columns.



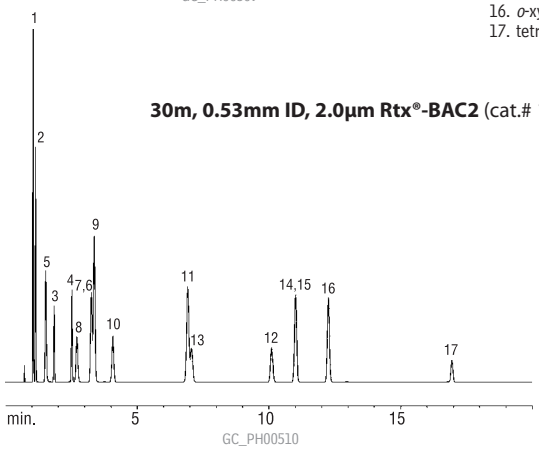


1.0mL headspace sample
Oven temp.: 40°C (hold 5 min.) to 240°C @ 5°C/min.
Inj. & det. temp.: 240°C
Carrier gas: He
Linear velocity: 65cm/sec.

Figure 4 Elution order changes for common industrial solvents on Rtx®-BAC1 and Rtx®-BAC2 columns.

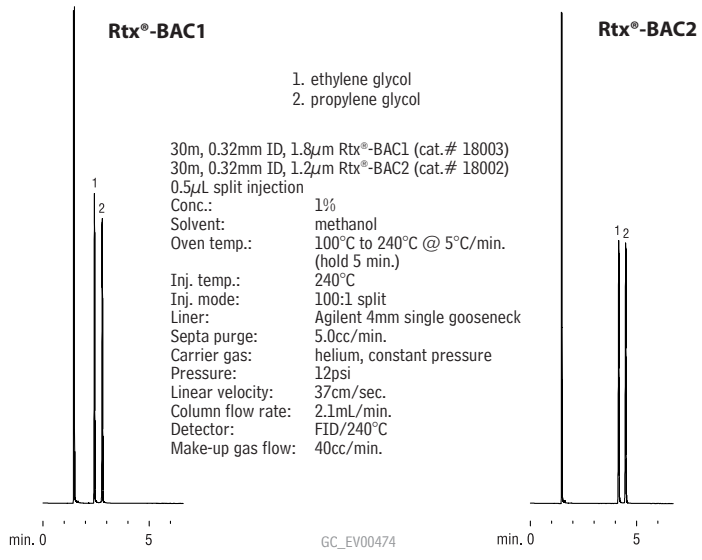


1. ethyl chloride
2. diethyl ether
3. methylene chloride
4. acetonitrile
5. methyl *tert*-butyl ether
6. chloroform
7. methyl ethyl ketone
8. 1,1,1-trichloroethane
9. benzene
10. trichloroethylene
11. toluene
12. 1,1,2-trichloroethane
13. tetrachloroethene
14. *p*-xylene
15. *m*-xylene
16. *o*-xylene
17. tetrachloroethane



1.0mL headspace sample
Oven temp.: 40°C (hold 5 min.) to 240°C @ 5°C/min.
Inj. & det. temp.: 240°C
Carrier gas: helium
Linear velocity: 65cm/sec.

Figure 5 Glycols of forensic interest on Rtx®-BAC1 and Rtx®-BAC2 Columns.



1. ethylene glycol
2. propylene glycol

30m, 0.32mm ID, 1.8µm Rtx®-BAC1 (cat.# 18003)
30m, 0.32mm ID, 1.2µm Rtx®-BAC2 (cat.# 18002)
0.5µL split injection

Conc.:	1%
Solvent:	methanol
Oven temp.:	100°C to 240°C @ 5°C/min. (hold 5 min.)
Inj. temp.:	240°C
Inj. mode:	100:1 split
Liner:	Agilent 4mm single gooseneck
Septa purge:	5.0cc/min.
Carrier gas:	helium, constant pressure
Pressure:	12psi
Linear velocity:	37cm/sec.
Column flow rate:	2.1mL/min.
Detector:	FID/240°C
Make-up gas flow:	40cc/min.

Blood Alcohol Standards

Compound	qty.	cat.#
0.015g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36232
1mL/ampul	10-pk.	36332
5mL/ampul	ea.	36240
20mL/ampul	ea.	36248
0.02g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36233
1mL/ampul	10-pk.	36333
5mL/ampul	ea.	36241
20mL/ampul	ea.	36249
0.025g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36234
1mL/ampul	10-pk.	36334
5mL/ampul	ea.	36242
20mL/ampul	ea.	36250
0.04g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36235
1mL/ampul	10-pk.	36335
5mL/ampul	ea.	36243
20mL/ampul	ea.	36251
0.05g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36257
1mL/ampul	10-pk.	36259
5mL/ampul	ea.	36258
20mL/ampul	ea.	36260
0.08g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36262
1mL/ampul	10-pk.	36264
5mL/ampul	ea.	36263
20mL/ampul	ea.	36265
0.1g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36236
1mL/ampul	10-pk.	36336
5mL/ampul	ea.	36244
20mL/ampul	ea.	36252
0.15g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36237
1mL/ampul	10-pk.	36337
5mL/ampul	ea.	36245
20mL/ampul	ea.	36253
0.2g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36238
1mL/ampul	10-pk.	36338
5mL/ampul	ea.	36246
20mL/ampul	ea.	36254
0.3g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36239
1mL/ampul	10-pk.	36339
5mL/ampul	ea.	36247
20mL/ampul	ea.	36255
0.4g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36266
1mL/ampul	10-pk.	36268
5mL/ampul	ea.	36267
20mL/ampul	ea.	36269

Blood Alcohol Mix Resolution Control Standard (8 components)

acetaldehyde
acetone
acetonitrile
ethanol (NIST certified value)
ethyl acetate
isopropanol
methanol
methyl ethyl ketone

0.100g/dL each in water, 1mL/ampul
cat. # 36256 (ea.)

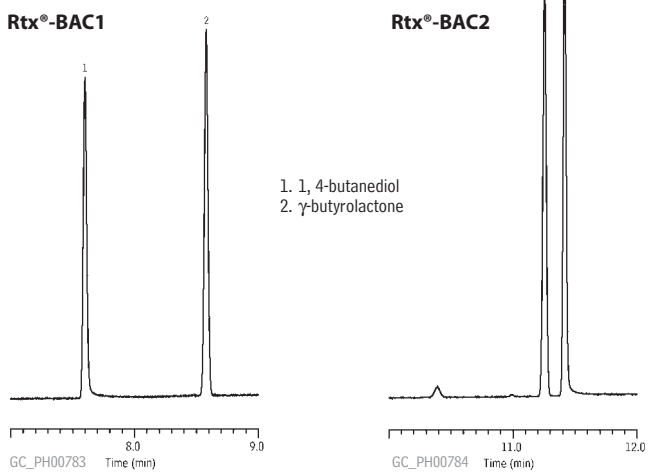
We designed Rtx®-BAC1 and Rtx®-BAC2 columns for blood alcohol analysis by headspace GC/FID (Figure 1), but many other materials of forensic interest also can be analyzed and confirmed using this column pair in a headspace GC/FID system, including inhalant anesthetics, alkyl nitrites, glycols, industrial solvents, and petroleum hydrocarbons. The substances in these target groups are resolved to baseline on one column or the other. Inhalants (Figure 2) or alkyl nitrites and their alcohol metabolites (Figure 3), for example, show excellent resolution and responses, and symmetrical peak shapes, in short analysis times. Similarly, performance is excellent for common industrial solvents (Figure 4). Retention times for many compounds of interest are presented in Table 1.1

For the analysis and confirmation of blood alcohols or other materials on Rtx®-BAC1 and Rtx®-BAC2 columns, we use a GC/FID equipped with a headspace autosampler that simultaneously introduces sample onto the two analytical columns. This dual column technique increases throughput by providing screening and confirmation data from a single injection. By using 0.32mm ID columns and a high carrier gas flow rate, we achieve baseline resolution of blood alcohol compounds in less than 3 minutes (Figure 1).

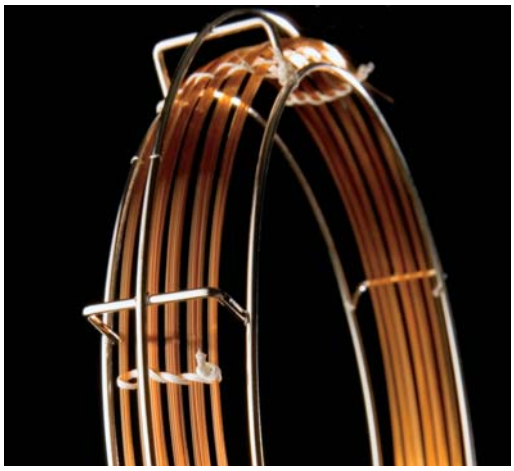
Other abused substances of interest, such as gamma-hydroxybutyrate (GHB, the "date rape drug"), and poisons, such as ethylene glycol, typically are analyzed from liquid injections. The Rtx®-BAC1 / Rtx®-BAC2 dual column system coupled with FID assures excellent responses and peak shapes for ethylene glycol and propylene glycol (Figure 5) or for GHB (Figure 6), which usually is converted to gamma-butyrolactone (GBL) for the analysis.

By analyzing these abused substances and poisons simultaneously on Rtx®-BAC1 and Rtx®-BAC2 columns, compounds coeluting on one stationary phase are resolved on the complementary stationary phase, and analytical and confirmation data are obtained in half the time required with sequential injections. Analytes characteristically are eluted with excellent responses and peak shapes. These example applications establish dual column analysis and confirmation on Rtx®-BAC1 and Rtx®-BAC2 columns as a very useful and highly adaptable forensics technique.

Figure 6 γ-butyrolactone & 1,4-butanediol on Rtx®-BAC1 & Rtx®-BAC2 Columns.



Column:	Rtx®-BAC1 30m, 0.32 mm ID, 1.8μm (cat.# 18003) and Rtx®-BAC2 30m, 0.32 mmID, 1.2μm (cat.# 18002)
Sample:	100μg/mL each γ-butyrolactone (GBL) and 1, 4-butanediol in methanol
Inj.:	1.0μL split (split ratio 1:10), 4mm single gooseneck inlet liner with wool (cat. # 22405)
Inj. Temp.:	240°C
Carrier Gas:	helium, constant flow
Flow Rate:	3.0mL/min.
Oven Temp.:	50°C to 240°C @ 10°C/min. (hold 2 min.)
Det.:	FID @ 240°C



Rtx®-BAC1 Columns (fused silica)

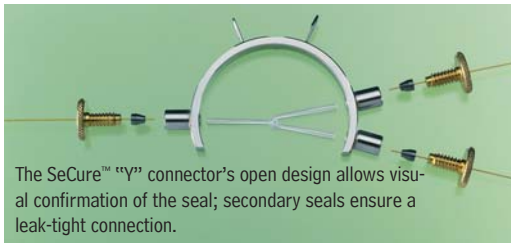
ID	df (μm)	temp. limits	length	cat. #
0.32mm	1.80	-20 to 240/260°C	30-Meter	18003
0.53mm	3.00	-20 to 240/260°C	30-Meter	18001

Rtx®-BAC2 Columns (fused silica)

ID	df (μm)	temp. limits	length	cat. #
0.32mm	1.20	-20 to 240/260°C	30-Meter	18002
0.53mm	2.00	-20 to 240/260°C	30-Meter	18000

SeCure™ "Y" Connector Kit

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



The SeCure™ "Y" connector's open design allows visual confirmation of the seal; secondary seals ensure a leak-tight connection.

Description	Ferrules Fit Column ID	qty.	cat.#
Connector Kit	0.28/0.32mm	kit	20277

Intermediate-Polarity Deactivated Guard Columns/Transfer Lines

Nominal ID	Nominal OD	5-Meter
0.32mm	0.45 ± 0.04mm	10044
0.53mm	0.69 ± 0.05mm	10045

[1]For more information about analyses of anesthetics, or for analyses of petroleum hydrocarbons, please request Application Note 59548 or 59574, respectively.

additional **reading**

Clarke's Analysis of Drugs and Poisons, Third Edition, A.C. Moffat, M.D. Osselton and B. Widdop (editors), Pharmaceutical Press, 2004.

Drug-Facilitated Sexual Assault: A Forensic Handbook, Marc A. LeBeau and Ashraf Mozayani, Academic Press, 2001

Handbook of Forensic Drug Analysis, Frederick P. Smith and Jay A. Siegel (editors), Academic Press, 2004

Optimized, 17-Minute GC Analysis of Semivolatiles

Using a 0.25mm ID Rtx®-5Sil MS Column

By Christopher English, Innovations Group Leader

- Excellent column for many methods, including US EPA methods 8270, 625, and 525.
- Greater on-column sample capacity, longer lifetimes than columns with thinner phase films.
- Analysis optimized for scanning mass spectrometers (ion trap or quadrupole).

Restek Innovations chemists have evaluated many combinations of stationary phase, column dimensions, and analytical conditions for analyzing environmental semivolatile compounds such as those listed in US Environmental Protection Agency Method 8270. Using a typical benchtop quadrupole mass spectrometer, we have achieved a 15-minute analysis, while maintaining a scan rate of at least 5 scans per target analyte.[1] Time-of-flight mass spectrometers (TOFMS) make analysis times under 9 minutes achievable,[2] because they can scan more than 100 times faster than quadrupole instruments.

After developing this analysis on a 0.18mm ID, 0.36µm column,[3] our chemists decided to experiment with a column of standard ID and phase film dimensions (0.25mm ID / 0.50µm film), in an attempt to establish a similarly rapid analysis on a larger bore column. The column they chose was a 30m x 0.25mm ID x 0.5µm Rtx®-5Sil MS column. The target compound list included our 8270 MegaMix™ and Appendix IX Mix #2 mixes, plus internal standards and surrogates—a total of 117 compounds. Advantages of using a 0.25mm ID column with a 0.5µm phase film include increased sample capacity and longer column lifetime, combined with rapid analyses.

Injection Port Optimization

The first step in the experiment was to optimize conditions in the injection port. We found that the inlet liner and seal remain inert longer when we inject only 0.5µL of sample into the injection port. This increases the number of passing calibration checks per liner and seal, and so reduces instrument downtime. The key to maintaining sensitivity when injecting smaller amounts of sample is to attain the most efficient sample transfer possible: we determined a 2mm ID inlet liner most efficiently transfers 0.5µL samples.

Splitless hold time also is important; a change of only several seconds can significantly affect the amount of sample ultimately delivered onto the column. We discovered that a pulsed splitless time, using a 0.4-minute pulse at 30psi (normal column backpressure is 8.8psi at 50°C), dramatically improves sample transfer onto the column. Making the pulse longer than the splitless hold time allows excess solvent to be swept away quickly and dramatically sharpens resolution of the early eluting Method 8270 Appendix IX compounds, such as 1,4-dioxane.

Other Conditions

After optimizing conditions in the injection port, we adjusted other analytical conditions to deliver a fast, rugged analysis on a 0.25mm ID column. In combination, a constant flow of 1.1mL/min., a short initial hold time (0.5 min.) and a fast initial temperature ramp rate (25°C/min.) elute benzo(ghi)perylene in 16.5 minutes. The final temperature ramp rate is a relatively slow 4°C/min., to better resolve benzo(b)fluoranthene and benzo(k)fluoranthene.

With all conditions optimized, the 117 target compounds in our sample are well resolved by quantification ion in one analysis (Figure 1, page 6).

8270 MegaMix™ (76 components)

acenaphthene	2,4-dinitrophenol
acenaphthylene	2,4-dinitrotoluene
aniline	2,6-dinitrotoluene
anthracene	di-*n*-butyl phthalate
azobenzene**	di-*n*-octyl phthalate
benzo(a)anthracene	diphenylamine***
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-cd)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-*n*-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

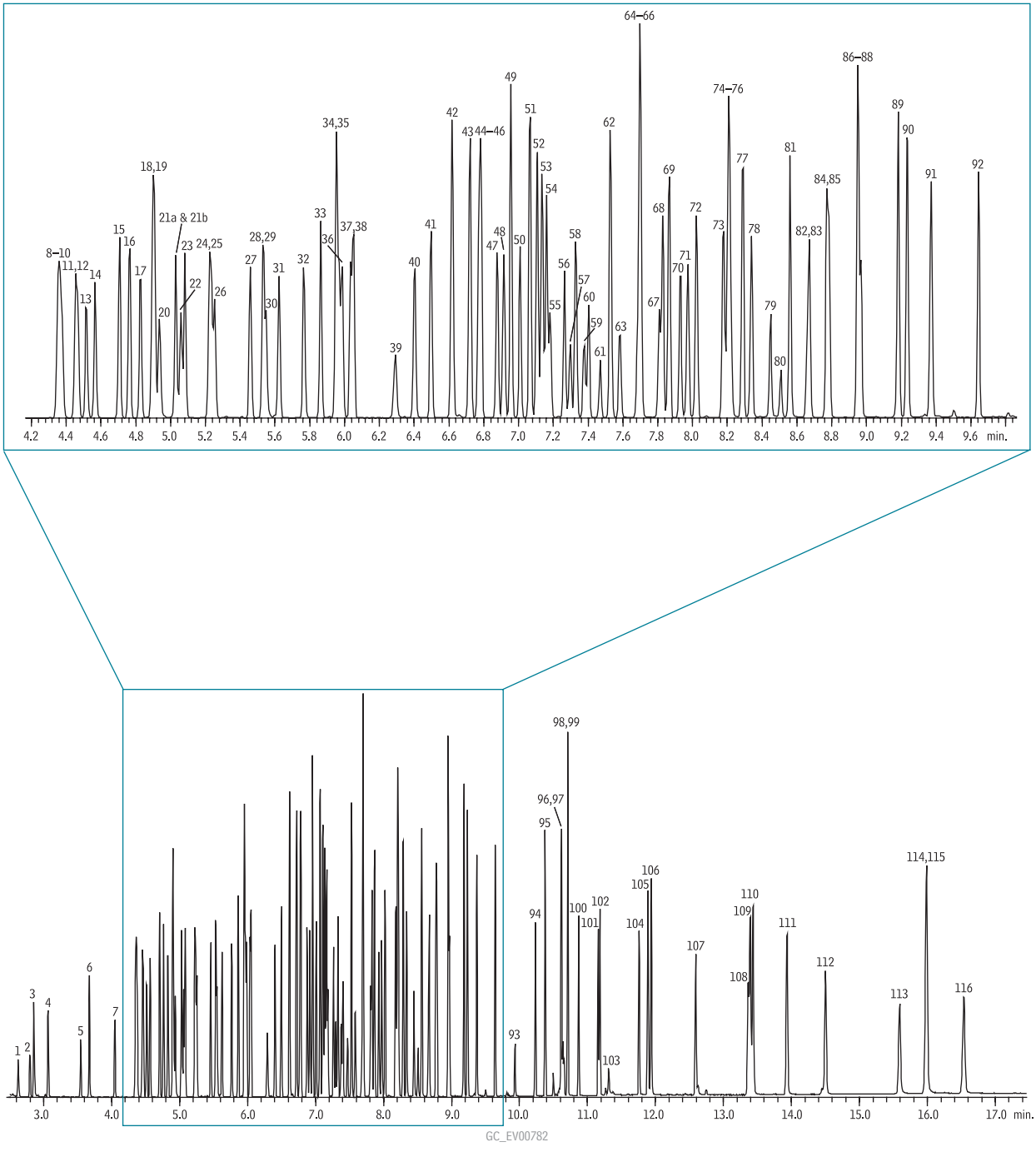
1,000µg/mL each in methylene chloride, 1mL/ampul*
cat. # 31850

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

**1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).

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

Figure 1 117 semivolatile compounds rapidly eluted and well resolved using a 0.25mm ID x 0.50µm Rtx®-5Sil MS column.



1. 1,4-dioxane
2. N-nitrosodimethylamine
3. pyridine
4. ethyl methacrylate
5. methyl methanesulfonate
6. 2-fluorophenol
7. ethyl methanesulfonate
8. phenol-d6
9. phenol
10. benzaldehyde
11. aniline
12. bis(2-chloroethyl)ether
13. pentachloroethane
14. 2-chlorophenol
15. 1,3-dichlorobenzene
16. 1,4-dichlorobenzene
17. benzyl alcohol
18. 2-methylphenol
19. 1,2-dichlorobenzene
20. bis(2-chloroisopropyl)ether
21a. 4-methylphenol
21b. 3-methylphenol
22. N-nitroso-di-*n*-propylamine
23. acetophenone
24. hexachloroethane
25. nitrobenzene-d5
26. nitrobenzene
27. isophorone
28. 2,4-dimethylphenol
29. 2-nitrophenol
30. diallate (isomer)
31. bis(2-chloroethoxy)methane
32. 2,4-dichlorophenol
33. 1,2,4-trichlorobenzene
34. naphthalene
35. 4-chloroanaline
36. 2,6-dichlorophenol
37. hexachloropropene
38. hexachlorobutadiene
39. ε-caprolactam
40. 4-chloro-3-methylphenol
41. isosafrole (*cis*)
42. 2-methylnaphthalene
43. 1-methylnaphthalene
44. hexachlorocyclopentadiene
45. isosafrole (*trans*)
46. 1,2,4,5-tetrachlorobenzene
47. 2,4,6-trichlorophenol
48. 2,4,5-trichlorophenol
49. 2-fluorobiphenyl
50. 2-chloronaphthalene
51. biphenyl
52. safrole
53. 1-chloronapthalene
54. diphenyl ether
55. 2-nitroaniline
56. 1,4-naphthoquinone
57. 1,4-dinitrobenzene
58. dimethylphthalate
59. 1,3-dinitrobenzene
60. 2,6-dinitrotoluene
61. 1,2-dinitrobenzene
62. acenaphthylene
63. 3-nitroaniline
64. 2,4-dinitrophenol
65. acenaphthene
66. 4-nitrophenol
67. 2,4-dinitrotoluene
68. pentachlorobenzene
69. dibenzofuran
70. 2,3,5,6-tetrachlorophenol
71. 2,3,4,6-tetrachlorophenol
72. diethyl phthalate
73. 4-chlorophenyl phenyl ether
74. 4-nitroaniline
75. fluorene
76. 4,6-dinitro-2-methylphenol
77. diphenylamine
78. azobenzene
79. 2,4,6-tribromophenol
80. 1,3,5-trinitrobenzene
81. phenacetin
82. diallate
83. 4-bromophenyl phenyl ether
84. hexachlorobenzene
85. atrazine
86. pronamide
87. pentachlorophenol
88. pentachloronitrobenzene
89. phenanthrene
90. anthracene
91. carbazole
92. di-*n*-butylphthalate
93. 4-nitroquinoline-N-oxide
94. isodrin
95. fluoranthene
96. pyrene
97. benzidine
98. *p*-terphenyl-d14
99. Aramite
100. chlorobenzilate
101. benzyl butyl phthalate
102. bis(2-ethylhexyl)adipate
103. Kepone
104. bis(2-ethylhexyl)phthalate
105. benzo(a)anthracene
106. chrysene
107. di-*n*-octyl phthalate
108. 7,12-dimethylbenzo(a)anthracene
109. benzo(b)fluoranthene
110. benzo(k)fluoranthene
111. benzo(a)pyrene
112. 3-methylcholanthrene
113. dibenzo(a,j)acridine
114. indeno(1,2,3-cd)pyrene
115. dibenzo(a,h)anthracene
116. benzo(ghi)perylene

Column:	Rtx®-5Sil MS 30m, 0.25mm ID, 0.50μm (cat.# 12738)
Sample:	US EPA Method 8270D Appendix IX mix 8270 MegaMix™ (cat.# 31850) Appendix IX Mix #2 (cat.#31806) Acid Surrogate Mix (4/89 SOW) (cat.# 31063) B/N Surrogate Mix (4/89 SOW) (cat.# 31062)
Inj.:	0.5μL, splitless, 100ppm each compound (50ng on column) 2mm Cyclo double gooseneck splitless inlet liner (cat.# 20907), 0.3 min. splitless hold time, 0.4 min. pressure pulse @ 30psi
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1.1mL/min.
Oven temp.:	50°C (hold 0.5 min.) to 310°C @ 25°C/min. (hold 0 min.) to 330°C @ 4°C/min. (hold 4 min.)
Det.:	MS
Det. temp.:	280°C
Transfer line temp.:	280°C
Scan range:	35-550 amu
Solvent Delay:	1 min.
Tune:	DFTPP
Ionization:	EI
Instrument:	Agilent 6890 / 5973

Conclusions

Complex mixtures of semivolatiles can be resolved on an Rtx®-5Sil MS column in a conventional 30m x 0.25mm ID x 0.50μm configuration, without sacrificing the speed associated with shorter, thin phase film columns, and with greater sample capacity. Restek can provide the columns, reference mixes, inlet and other accessories, and technical help you need for reliable, problem-free analyses of semivolatiles by US EPA or other methodology.

References

1. *Fast Analysis of Semivolatile Organic Analytes*, Restek Advantage, 2004, Vol.2 p.2.
2. *Nine-Minute Analysis of Semivolatile Organic Compounds*, Restek Advantage, 2005, Vol.1 p. 8.
3. *Fast GC/MS Analysis of Semivolatile Organic Compounds*, Restek Advantage, 2005, Vol 1 p. 14.

References available on request.

Appendix IX Mix #2 (32 components)

acetophenone	hexachloropropene
Aramite	isodrin
atrazine	isosafrole (*cis* & *trans*)
benzaldehyde	kepone
biphenyl	3-methylcholanthrene
ε-caprolactam	methyl methanesulfonate
chlorobenzilate	1,4-naphthoquinone
1-chloronaphthalene	4-nitroquinoline-N-oxide
diallate	pentachlorobenzene
dibenzo(a,j)acridine	pentachloroethane
2,6-dichlorophenol	pentachloronitrobenzene
7,12-dimethylbenz(a)anthracene	phenacetin
1,4-dioxane	pronamide
diphenyl ether	safrole
ethyl methacrylate	1,2,4,5-tetrachlorobenzene
ethyl methanesulfonate	1,3,5-trinitrobenzene

1,000μg/mL each in methylene chloride, 1mL/ampul
cat. # 31806

Acid Surrogate Mix (4/89 SOW)

2-fluorophenol	2,4,6-tribromophenol
phenol-d6	

2,000μg/mL each in methanol, 1mL/ampul
cat. # 31025

10,000μg/mL each in methanol, 1mL/ampul
cat. # 31063

10,000μg/mL each in methanol, 5mL/ampul
cat. # 31087

B/N Surrogate Mix (4/89 SOW)

2-fluorobiphenyl	*p*-terphenyl-d14
nitrobenzene-d5	

1,000μg/mL each in methylene chloride, 1mL/ampul
cat. # 31024

5,000μg/mL each in methylene chloride, 1mL/ampul*
cat. # 31062

5,000μg/mL each in methylene chloride, 5mL/ampul*
cat. # 31086

*Requires warming and sonication before use.

Rtx®-5Sil MS Columns (fused silica)

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

ID	df (μm)	temp. limits	length	cat. #
0.18mm	0.18	-60 to 325°C	20-Meter	42702
0.18mm	0.36	-60 to 330/350°C	20-Meter	42704
0.25mm	0.25	-60 to 330/350°C	30-Meter	12723
0.25mm	0.50	-60 to 330/350°C	30-Meter	12738

High-Speed Detailed Hydrocarbon Analysis

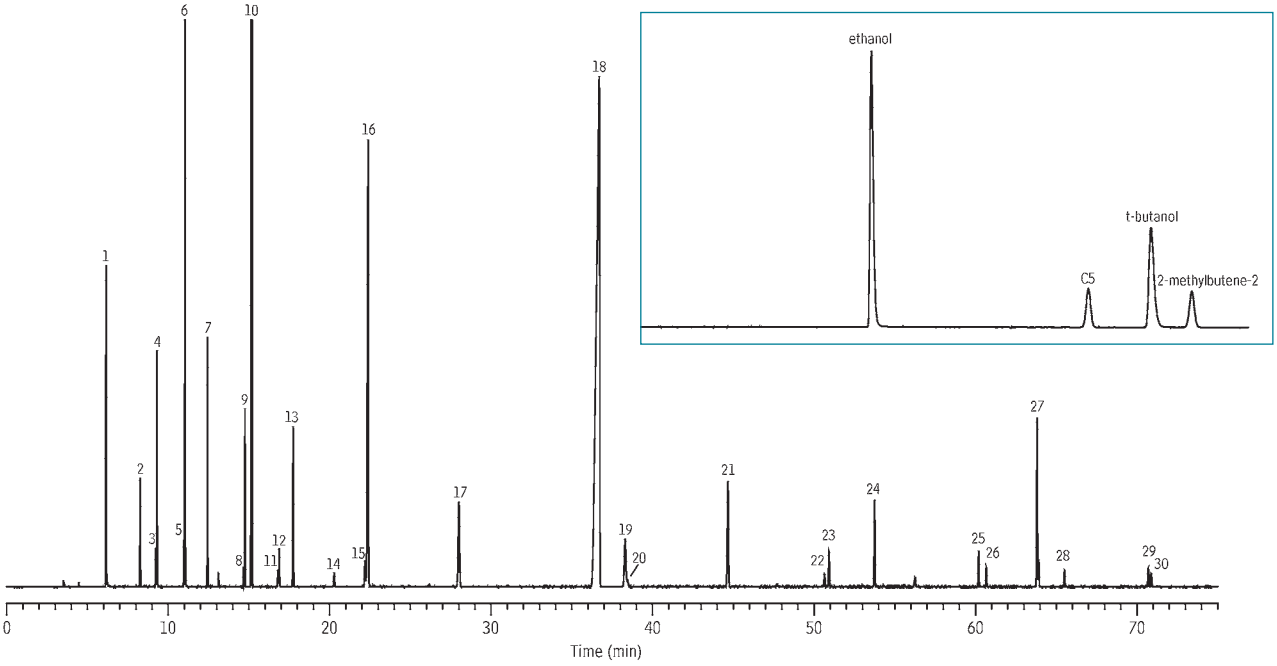
53% Faster Analysis, Using an Rtx®-1 PONA Column and Hydrogen Carrier Gas

By Barry Burger, Petroleum Applications Chemist

- C13 eluted within 70 minutes.
- Excellent response and peak symmetry for polar oxygenates.
- Column meets or exceeds all ASTM D-6730-01 and Canadian General Standards Board method requirements.
- Guaranteed column-to-column reproducibility.

American Society for Testing and Materials method D-6730-01 was designed specifically for determining the individual hydrocarbons in spark ignition fuels and fuel blends containing oxygenates such as methyl *tert*-butyl ether (MTBE), ethyl *tert*-butyl ether (ETBE), *tert*-butanol, and ethanol. To maximize the resolution of these complex mixtures, ASTM recommends a 100 meter x 0.25mm ID capillary column with a 0.5µm film of 100% dimethyl polysiloxane stationary phase as the primary analytical column. To control selectivity for the aromatic compounds, a short tuning column, typically 2-3 meters, containing a 5% diphenyl / 95% dimethyl polysiloxane stationary phase, is connected to the inlet of the primary column. To meet the demanding resolution and retention criteria in ASTM method D-6730-01, and in Canadian General Standards Board (CGSB) methodology for detailed hydrocarbon analysis (DHA), Restek Innovations chemists have reformulated our Rtx®-1PONA column.

Figure 1 Rtx®-1PONA column meets ASTM D-6731-01 resolution and retention specifications.



Column:	Rtx®-1PONA 100m, 0.25mm, 0.5µm (cat.# 10195) plus Rtx®-5PONA tuning column (cat. # 10196), connected via angled Press-Tight® connector (cat.# 20446)
Sample:	DHA/oxygenates setup blend
Inj.:	0.01µL, split (split ratio 150:1), 4mm ID cup inlet liner (cat.# 20709) A: front slice of DHA/oxygenates setup blend B: DHA/oxygenates setup blend
Carrier gas:	hydrogen, constant flow (3.62cc/min.)
Linear velocity:	55cm/sec.
Inj. temp.:	250°C
Oven temp.:	A: 35°C B: 5°C (hold 8.32 min.) (elute C5) to 48°C @ 22°C/min. (hold 26.32 min.) (elute ethylbenzene) to 141°C @ 3.20°C/min. (no hold) (elute C12) to 300°C @ 1°C/min.
Det.:	FID @ 300°C

A: Front end of DHA/oxygenates setup blend
C5 efficiency: 586,825 plates
C5 k': 0.476
tert-butanol skew: 2.10
Resolution *tert*-butanol/ 2-methylbutene-2: 5.39

B: DHA/oxygenates setup blend

1. ethanol
2. C5
3. *tert*-butanol
4. 2-methylbutene-2
5. 2,3-dimethylbutane
6. methyl *tert*-butyl ether (MTBE)
7. C6
8. 1-methylcyclopentene
9. benzene
10. cyclohexane
11. 3-ethylpentane
12. 1,2-dimethylcyclopentane
13. C7
14. 2,2,3-trimethylpentane
15. 2,3,3-trimethylpentane
16. toluene
17. C8
18. ethylbenzene
19. *p*-xylene
20. 2,3-dimethylheptane
21. C9
22. 5-methylnonane
23. 1,2-methylethylbenzene
24. C10
25. C11
26. 1,2,3,5-tetramethylbenzene
27. naphthalene
28. C12
29. 1-methylnaphthalene
30. C13

Chromatogram courtesy of Neil Johansen, Inc., Aztec, New Mexico, in association with Envantage Analytical Software, Inc., Cleveland, Ohio.

Method D-6730-01 suggests using helium as the carrier gas, at a linear velocity of 24cm/sec. (approximately 2.3mL/min.). The tridecane (C13) retention time this combination yields, approximately 146 minutes, greatly limits sample throughput per day. Our enhanced Rtx®-1PONA column meets or exceeds all criteria in the method, but does so in 30% less time: retention time for C13 is 97 minutes, using helium as the carrier gas.[1] In most applications hydrogen is a better alternative to helium as the carrier gas, because it can be used at much higher linear velocities without compromising critical resolutions.

A revision to ASTM D 6730-01 proposed by Neil Johansen Inc. (Aztec, New Mexico), in association with Envantage Analytical Software Inc. (Cleveland, Ohio), has established optimal DHAX (detailed hydrocarbon analysis—extended) parameters, including specifying hydrogen as the carrier gas. Using these conditions, analysis time is reduced to within 71 minutes (C13)—a 53% reduction versus using helium as the carrier gas. The method also is extended to include middle distillates having final boiling points up to 509°C/948°F (*n*-C38).

Restek provided Neil Johansen Inc. with an enhanced Rtx®-1PONA column (100m x 0.25mm ID x 0.5µm df) and an Rtx® 5PONA tuning column for DHAX method development. The Rtx®-1PONA column was connected to 3 meters of the tuning column through a Universal Angled Press-Tight® Connector (cat.# 20446) and was installed in a PerkinElmer AutoSystem XL GC equipped with a flame ionization detector and programmable pneumatic control. The data system used was ChromPerfect Spirit (Justice Laboratory Software, Denville, NJ). Individual compounds were identified by using Dragon- DHA software, developed by Envantage Analytical Software Inc. in association with Neil Johansen Inc., which uses algorithms to process high-resolution chromatographic data. Processed data for the PONA VI reference standard, containing more than 400 individual components of finished gasoline, can be reviewed on the Restek website: www.restek.com/PONA

The proposed DHAX method was optimized with hydrogen carrier gas at a rate of 3.62mL/min, constant flow, producing a linear velocity of 55cm/sec. The new Rtx®-1PONA column was conditioned in less than two hours, as follows: 35°C for 15 min., to 300°C at 10°C/min., hold 30 min., cool to 35°C. Dead time was adjusted to elute methane at 3.50 ±0.05 min., then a DHA/oxygenates setup blend was introduced into the column to determine the column's suitability for the method.

Figure 1A lists the measured critical criteria. Once established that the column met the method criteria, GC oven program parameters were entered and trial injections of the DHA/oxygenates setup blend were begun. The analysis was permitted to run until *n*-C13 was eluted. Based on the resolutions achieved, the length of the tuning column was reduced incrementally until all critical pairs met D-6730-01 specifications. In this example application, the appropriate tuning column length was 2.36 meters. Figure 1B indicates the critical pairs. The full analysis of the 400-plus component PONA VI reference standard, listing retention indices (RIs) calculated using Dragon-DHA software, is posted on the Restek website.

The benefits of using hydrogen carrier gas for the PONA analysis are obvious: all critical components are resolved, per method D-6703-01, in the greatly reduced time of 70.5 minutes (C13), versus 146 minutes or 97 minutes using helium. Relative to the results anticipated in the method, we virtually doubled sample throughput.

We evaluate each column for film thickness, column efficiency, peak skewness, selectivity, resolution, and bleed to guarantee performance and reproducibility from column to column. The redesigned Rtx®-1PONA column earned Restek chemists the Concluded Research Award at the 2004 Gulf Coast Conference. When you use an Rtx®-1PONA column, we think you'll agree the award was well justified.

Rtx®-1PONA Column (fused silica)

(Crossbond® 100% dimethyl polysiloxane)*

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

*Optimized phase for hydrocarbon analysis

Rtx®-5PONA Tuning Column (fused silica)

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

ID	df (µm)	temp. limits	length	cat. #
0.25mm	1.0	-60 to 325°C	5-Meter	10196

Universal Angled Press-Tight® Connectors

- Ideal for connecting a tuning column to an analytical column.
- Inert fused silica.
- Angle reduces strain on the connection.
- Fit all column ODs from 0.33–0.74mm (Restek 0.1mm–0.53mm ID).

5-pk./price	25-pk./price	100-pk./price
Universal Angled Press-Tight® Connectors		
20446	20447	20448
Siltek®-treated Universal Angled Press-Tight® Connectors		
20482	20483	20484

Reference

1. Stidsen, G. and B. Burger, *Enhanced Rtx®-1PONA Column Improves Detailed Hydrocarbon Analysis,* Restek Advantage 2005v1: 12 (2005).

for more **info**!

Processed data for the PONA VI reference standard, containing more than 400 individual components of finished gasoline, can be reviewed on the Restek website: **www.restek.com/PONA**

please **note**

To achieve critical resolutions in detailed hydrocarbon analysis, a 5-meter 5% diphenyl/ 95% dimethyl polysiloxane tuning column (Rtx®-5PONA) is connected to the analytical column and adjusted to the needed length through a series of trial analyses.

Rapidly Determine Benzene and Toluene in Gasolines

Micropacked GC Columns Reduce Analysis Time by 63%

By Barry Burger, Petroleum Applications Chemist

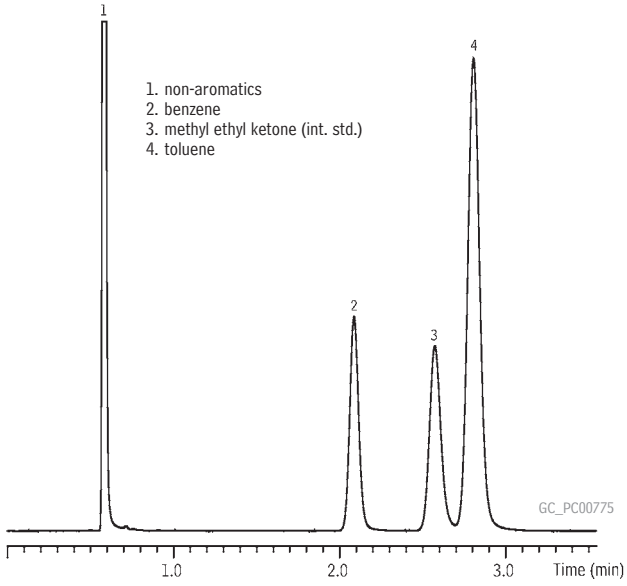
- 3-Minute Cycles for ASTM Method D-3606-99.
- Nearly triple sample throughput.
- Easy set-up, using Restek adaptor kit.

American Society for Testing and Materials test method D-3606-99 is focused on measuring benzene and toluene in finished motor and aviation gasolines: benzene can be determined from 0.1% to 5% by volume and toluene from 2.0% to 20% by volume. The method requires two columns connected in series. Typically, column A is a 0.8 meter x 1/8 inch stainless steel packed column containing a 10% loading of a nonpolar stationary phase, such as Rtx®-1 or OV®-101. This column separates sample components by boiling point. After n-octane (C8) elutes, the column is backflushed to prevent heavier compounds from entering column B, the main analytical column. The light compounds, C8 and below, pass into column B, a 4.0 meter x 1/8 inch stainless steel packed column containing highly polar 1,2,3 tris(2-cyanoethoxy) propane (TCEP). Here, the aromatic compounds are separated from the non-aromatics, and quantitative information is obtained.

For method D-3606-99, micropacked column technology is an efficient, practical, time-saving alternative to 1/8 inch packed columns. Micropacked column A is a 0.8 meter x 1/16 inch x 1mm ID Silcosteel® column packed with 10% Rtx®-1 crosslinked on 60/80 Chromosorb® WAW. Micropacked column B is a 4.0 meter x 1/16 inch x 1mm ID Silcosteel® column packed with 20% TCEP on 80/100 Chromosorb® PAW. We installed the columns in an Agilent 6890 GC capillary inlet, configured in the on-column injection mode using our Micropacked Column Adaptor Kit for On-Column Injection (cat.# 22425). We used hydrogen as the carrier gas and, to attain the 9mL/min. flow rate, we adjusted the column head pressure to 44psig at 145°C.

Figure 1 illustrates the analysis of a sample containing 1% benzene, 2% toluene, and internal standard methyl ethyl ketone (MEK), in *n*-pentane (C5). The cycle time, just under 3 minutes, is greatly reduced, relative to the 8 minute cycle imposed by 1/8 inch packed columns and helium carrier gas. The micropacked column / hydrogen carrier gas combination reduces analysis time by 63%, nearly tripling sample throughput. If you are performing method D-3606-99 analyses, and time is important to you, we highly recommend this micropacked column approach.

Figure 1 Benzene, toluene, and internal standard resolved in 3 minutes, using a micropacked column.



Column:	A: 10% Rtx® 1 on 60/80 Chromosorb® WAW, 0.8m x 1/16 inch x 1mm ID Silcosteel® tubing B: 20% TCEP on 80/100 Chromosorb® PAW, 4.0m x 1/16 inch x 1mm ID Silcosteel® tubing connected in series and using Micropacked Column On-Column Injection Kit (cat.# 22425)
Sample:	benzene, toluene, methyl ethyl ketone in pentane
Inj.:	1.0µL, on-column, 4mm ID splitless inlet liner (cat.# 20772)
Carrier gas:	hydrogen, constant flow
Flow rate:	9.0mL/min.
Inj. temp.:	200°C
Oven temp.:	145°C
Det.:	FID @ 200°C

Micropacked Columns

1/16 inch micropacked columns containing 10% Rtx®-1 on 60/80 Chromosorb® W or 20% TCEP on 80/100 Chromosorb® PAW are prepared on request. For details, please contact your Restek representative.

Micropacked Inlet Conversion Kits

Convert a capillary GC split/splitless inlet for use with 1/16" OD micropacked columns.

- For use with Agilent 5890 and 6890 GCs.
- Sample pathways deactivated for ultimate inertness.

Description	qty.	cat.#
Micropacked Column Adaptor Kit for On-Column Injection* Complete kit with FID and injection port adaptors Kit includes: Dual Vespel® Ring Inlet Seal, large bore; reducing nut, large bore; FID adaptor, large bore; 1/4" ferrule, Vespel®/graphite; 1/4" nut, stainless steel; 1/16" ferrules, Vespel®/graphite (2); Siltek®-treated metal liner installation guide; 1/16" nuts, stainless steel (2)	kit	22425

*For use with packed column FIDs only.

Rapid, Dual Column Analysis for Organochlorine Pesticides

12-Minute Analysis Using Rtx®-CLPesticides2 / Rtx®-440 Capillary GC Columns

By Jason Thomas, Environmental Innovations Chemist

- Analysis and confirmation with a single injection.
- Rapid analysis increases throughput.
- New, thicker Rtx®-CLPesticides2 phase increases column lifetime.

Capillary GC stationary phases for organochlorine pesticides (e.g. US EPA Method 8081) must possess the selectivity needed to resolve target pesticides, yet withstand the rigors of repeated injections of extracts containing harsh residuals from the sample matrix. The US EPA method also requires a suitable counterpart column for confirmation, to quantify potential pesticide "hits." An efficient way to meet these requirements is through dual column analysis, which eliminates the need for a separate confirmation run, or GC/MS analysis. A newly enhanced version of our Rtx®-CLPesticides2 column, with dimensions of 30m x 0.32mm ID x 0.50µm (cat.# 11325) is now available for this application. The Rtx®-CLPesticides2 column, coupled with an Rtx®-440 column of the same dimensions (cat.# 12939), can provide a complete separation of the 20 most commonly analyzed organochlorine pesticides, listed in Method 8081, in less than 12 minutes (Figure 1).

The unique selectivities of the Rtx®-CLPesticides2 column and the Rtx®-440 column enable the analysis to be run quickly, with good resolution and peak shapes, as shown in Figure 1. Both columns produce similar run times, with a set of elution order inversions and, because they share a common stationary phase thickness, they should exhibit similar life expectancies. For accurate, time-saving analyses of organochlorine pesticides, we highly recommend the Rtx®-CLPesticides2 / Rtx®-440 column combination.

Rtx®-CLPesticides2 Column (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	0.50	-60 to 320/340°C	30-Meter	11325

Rtx®-440 Column (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.25mm	0.25	20°C to 320/340°C	30-Meter	12923

SeCure™ "Y" Connector Kit

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

Ferrules Fit Column ID	qty.	cat.#
0.28/0.32mm	kit	20277

Organochlorine Pesticide Mix AB #2

20 components, listed in Figure 1, 8-80µg/mL in hexane:toluene (1:1), 1mL/ampul

cat. # 32292

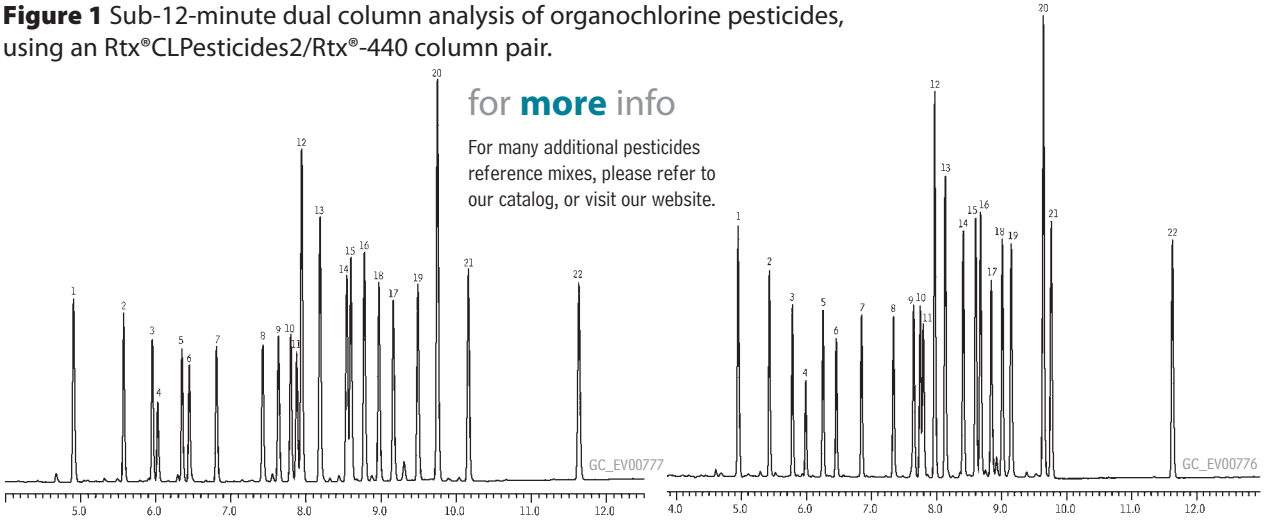
Pesticide Surrogate Mix

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

200µg/mL each in acetone, 1mL/ampul

cat. # 32000

Figure 1 Sub-12-minute dual column analysis of organochlorine pesticides, using an Rtx®CLPesticides2/Rtx®-440 column pair.



Column:	Rtx®-CLPesticides2 30m, 0.32mm ID, 0.50µm (cat.# 11325) and Rtx®-440 30m, 0.32mm ID, 0.50µm (cat.# 12939) with 5m x 0.32mm ID Siltek® treated guard tubing (cat.# 10027), connected using SeCure™ "Y" Connector Kit (cat.# 20277) with Universal "Y" Press-Tight® Connector
Sample:	Organochlorine Pesticide Mix AB #2, 8-80µg/mL each component in hexane/toluene (cat.# 32292), Pesticide Surrogate Mix, 200µg/mL each component in acetone (cat.# 32000)
Inj.:	0.5µL splitless (hold 0.75 min.), 3.5mm Siltek® treated single gooseneck inlet liner (cat.# 20961)
Inj. temp.:	250°C
Carrier gas:	hydrogen, constant pressure
Linear velocity:	60cm/sec. @ 300°C
Oven temp.:	110°C (hold 0.5 min) to 230°C @ 25°C/min., to 330°C @ 15°C/min. (hold 1 min.)
Det.:	ECD @ 330°C
Instrument:	Shimadzu GC-2010 FID/ECD with AOC-20i autosampler

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

New Analytical Reference Materials for Fuels, Accelerants

By Ken Herwehe, Analytical Reference Materials Product Marketing Manager, Mark Badger, Senior Organic Chemist

did you **know**?

We have over 2,000 pure, characterized, neat compounds in our inventory! If you do not see the EXACT mixture you need listed on any of these pages, call us.

For our on-line Custom Reference Materials Request Form visit us on the web at **www.restek.com/solutions**.

tert-Amyl ethyl ether Standard

Oxygenate additive in (US) gasolines.

A US EPA target analyte that is not commercially available, this reference material is prepared from a laboratory-synthesized sample.

2,000µg/mL in P&T methanol, 1mL/ampul
cat. # 30617

Oxygenates Standard

diisopropyl ether (DIPE)	2,000µg/mL
ethyl-*tert*-butyl ether (ETBE)	2,000
tert-amyl ethyl ether (TAEE)	2,000
tert-amyl methyl ether (TAME)	2,000
tert-butyl alcohol (TBA)	10,000

In P&T methanol, 1mL/ampul
cat. # 30619

tert-Butanol-d^9 Standard

An internal standard for oxygenates.

20,000µg/mL in P&T methanol, 1mL/ampul
cat. # 30618

Diesel/Biodiesel 80:20 Blend Standard

The biodiesel component is methyl soyate.

5,000µg/mL in methylene chloride, 1mL/ampul
cat. # 31880

Florida TRPH Standard (17 components)

n-octane (C8)
n-decane (C10)
n-dodecane (C12)
n-tetradecane (C14)
n-hexadecane (C16)
n-octadecane (C18)
n-eicosane (C20)
n-docosane (C22)
n-tetracosane (C24)
n-hexacosane (C26)
n-octacosane (C28)
n-triacontane (C30)
n-dotriacontane (C32)
n-tetratriacontane (C34)
n-hexatriacontane (C36)
n-octatriacontane (C38)
n-tetracontane (C40)

500µg/mL each in hexane, 1mL/ampul
cat. # 31266

2,000µg/mL each in carbon disulfide, 1mL/ampul*
NEW Higher concentration
cat. # 31878

Florida TRPH Surrogate Mix

n-nonatriacontane (C39)

3,000µg/mL in carbon disulfide, 1mL/ampul*
cat. # 31456

3,000µg/mL in carbon disulfide, 10mL/ampul*
NEW Larger volume
cat. # 31877

*Ground transportation shipments only.

About Biodiesel Fuel

In the US, soybean oil is the predominant feedstock for biodiesel fuel. Through transesterification, the oil is converted to methyl soyate, which has the characteristics and physical properties appropriate for a diesel-type fuel. Although biodiesel fuel can be used alone in diesel engines, many fleet operators blend it with petroleum-based diesel fuels to stretch the supply and lessen the cost of using what is still an expensive commodity, as biodiesel fuel still is not produced on a large scale.

Biodiesel fuel in an 80:20 blend (B20) has huge benefits for the consumer, including reduced emissions. The relatively high oxygen content of biodiesel fuel can reduce particulates, and has been shown to reduce NOx emissions and engine wear. New low-sulfur diesel fuels, mandated by the US EPA to reduce SOx emissions, lose some of the inherent lubrication that the sulfur species impart. However, biodiesel fuel has adequate inherent lubrication to offset the effects of the low-sulfur petroleum-based fuels.

Other biodiesel standards, at different blend ratios (e.g., B80 or B100), are available as custom products. In addition, we can custom prepare materials that comply with ASTM D6584, the method used to determine glycerin and free glycols in biodiesel fuel.

Single Source Unleaded Gasoline (ASTM Class 2 Accelerant)

These solutions are prepared from a single source (one refinery) product. Samples of regular and premium grade unleaded gasoline were collected, then blended in equal volumes. The weathered materials indicate the percent weight loss from the original material.

Compound	cat.# (ea.)
5,000µg/mL in P&T methanol, 1mL/ampul	
unleaded gasoline: unweathered	30096
unleaded gasoline: 25% weathered	30097
unleaded gasoline: 50% weathered	30098
unleaded gasoline: 75% weathered	30099
unleaded gasoline: 99% weathered	30436

Kerosene (ASTM Class 4 Accelerant)

These solutions are prepared from a single source (one refinery) product. The weathered materials indicate the percent weight loss from the original material.

Compound	cat.# (ea.)
5,000µg/mL in methylene chloride, 1mL/ampul	
kerosene: unweathered	31229
kerosene: 25% weathered	31230
kerosene: 50% weathered	31231
kerosene: 75% weathered	31232

Diesel Fuel #2 (ASTM Class 5 Accelerant)

These solutions are prepared from a single source (one refinery) product. The weathered materials indicate the percent weight loss from the original material.

Compound	cat.# (ea.)
5,000µg/mL in methylene chloride, 1mL/ampul	
diesel fuel #2: unweathered	31233
diesel fuel #2: 25% weathered	31234
diesel fuel #2: 50% weathered	31235
diesel fuel #2: 75% weathered	31236

Mineral Spirits

The mineral spirit solutions listed below were prepared from an equal volume blend of Type I, II, and III mineral spirits.

Compound	cat.# (ea.)
5,000µg/mL in methylene chloride, 1mL/ampul	
mineral spirits: unweathered	31225
50,000µg/mL in methylene chloride, 1mL/ampul	
mineral spirits: unweathered	31260
50,000µg/mL in methylene chloride, 5mL/ampul	
mineral spirits: unweathered (5mL/ampul)	31261
5,000µg/mL in methylene chloride, 1mL/ampul	
mineral spirits: 25% weathered	31226
5,000µg/mL in methylene chloride, 1mL/ampul	
mineral spirits: 50% weathered	31227
5,000µg/mL in methylene chloride, 1mL/ampul	
mineral spirits: 75% weathered	31228

for **more** info

For blended/composite materials, refer to our catalog, or visit our website at **www.restek.com/standards**

Distillates and Crude Oils

In addition to our stock products, we offer the following materials as custom products.

For details, visit our website: **www.restek.com/standards**

biodiesel (methyl soyate)
diesel #1
diesel #2: straight run - low sulfur - weathered - single source - composite
fuel oil #4
fuel oil #5
fuel oil #6
kerosene: unweathered - weathered
aviation gasoline, 100 octane
jet A: straight run - additized
JP-4
JP-5
JP-8
JP-10
RP-1
unleaded gasoline: oyxgenate free - weathered - single source - composite
naphtha cut
charcoal lighter fluid
Stoddard solvent
mineral spirits: unweathered - weathered
mineral oil
gear oil
hydraulic oil
turbine oil
cutting fluid
vacuum pump oil
motor oil: 10W30 - 10W40 - 20W50 - 5W30 - 30W - 40W - 50W - blend - used composite
creosote oil
light cycle oil (LCO)
refined chemical oil (RCO)
coal tar pitch
asphalt
blacktop patch
Californian heavy crude
Alaskan crude
Pennsylvanian crude

Superior Moisture Dry-Down and Corrosion Resistance

Restek treated tubing and system components improve analytical reliability and prolong lifetimes.

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

- Up to three times faster response to moisture changes in process streams.
- Corrosion resistance improved tenfold, or more—prolongs component lifetime and maintains pure product stream.
- Custom services: can be applied to existing equipment.

did **you** know?

Restek surface treatments deposit an amorphous silicon based layer onto, and into, the steel surface through a chemical vapor deposition (CVD) process. All exposed surfaces are coated. For corrosion resistance, layer depth is optimized at 5 to 10 microns. The amorphous silicon layer can be further functionalized using the patented Siltek® process (US Patent #6,444,326), which reduces moisture hold-up and improves surface inertness.

Introduction

Often, gas transfer systems require low moisture content, low moisture retention, and high resistance to corrosion. The current substrates of choice, including electropolished VIM/VAR (vacuum induction melt/vacuum arc melt) 316L stainless steel, typically are insufficient in these capacities, increasing periodic maintenance, prolonging equilibration times, and allowing system contamination and inaccurate analytical results. In contrast, surface treatments available through the Restek Performance Coatings Group greatly accelerate wet-up and dry-down times and dramatically improve corrosion resistance.

Experiments measuring the response time for moisture content change in Restek treated electropolished stainless steel tubing, untreated electropolished stainless steel tubing, and standard 316L stainless steel tubing, demonstrate a significant advantage in Restek treated substrates.[1] Wet-up curves for Siltek®

Figure 1 Restek treated electropolished tubing stabilizes at 1ppm moisture much faster than conventional surfaces.[1]

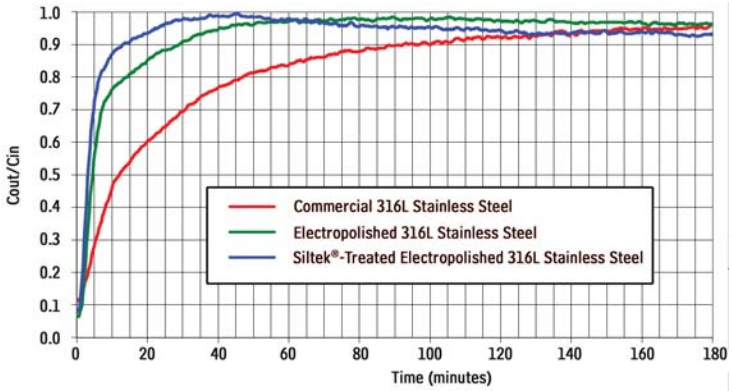
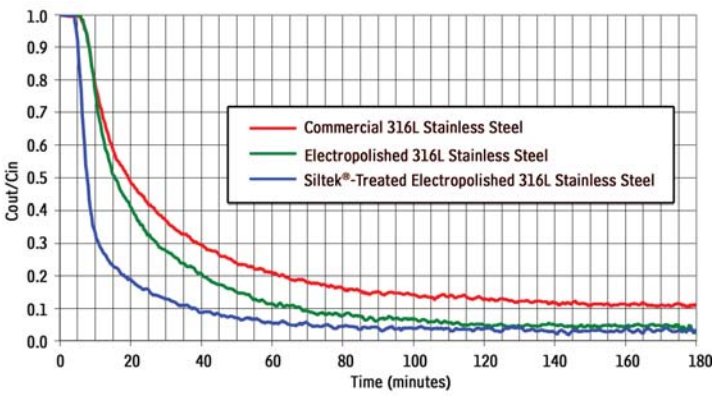


Table I Restek treated electropolished tubing provides the shortest drying times.[1]

Moisture Concentration		Time Required to Detect Change (min.) Treated Electropolished Tubing	Untreated Electropolished Tubing	Standard Tubing
From	To			
10ppm*	5ppm	4	5	13
5ppm	1ppm	22	46	71
1ppm	500ppb	40	63	96
500ppb	100ppb	80	103	153
100ppb	50ppb	98	121	—

*Initial moisture concentration.

Figure 2 Restek treated electropolished tubing dries much faster than conventional surfaces.[1]



treated electropolished, electropolished, and standard tubing are compared in Figure 1. Treated electropolished tubing reached the 98% saturation limit in 30 minutes, compared to 60 minutes for electropolished tubing. Standard tubing could only achieve a 96% uptake, after 180 minutes.

After the tubing was stabilized with 1ppm of moisture, dry-down properties were measured. Moisture dry-down curves for the three tubing treatments show treated electropolished tubing achieved dry-down in 35 minutes, electropolished tubing required 65 minutes, and standard tubing required 175 minutes (Figure 2). Table 1 compares time to various dry-down levels for tubing saturated with 10ppm of moisture.

Superior Corrosion Resistance: Silcosteel®-CR

In addition to rapid wet-up and dry-down, the other key advantage of Restek treatment for 316L stainless steel is a dramatic improvement in corrosion resistance. The amorphous silicon layer is insoluble in many acidic environments. Figures 3, 4, and 5 briefly summarize the results of corrosion testing by ASTM methods. Comparisons between treated and untreated test samples illustrate the improvements in corrosion resistance offered by Silcosteel®-CR treatment. For more information about corrosion resistance, request information packet 59048, or visit our website.

When moisture considerations and corrosion concerns arise in transfer of ultra-high purity gas streams, Restek treated tubing and system components will dramatically improve dry-down, reduce contamination from moisture carryover, and extend periodic maintenance cycles.

Reference

1. *Relative Response Time of True Tube™ when Measuring Moisture Content in a Sample Stream* Test Report, Haritec Scientific & Engineering Support, Calgary, Alberta, Canada, May 2004.

Tubing used in the wet-up / dry-down experiments was supplied by Cardinal UHP (St. Louis, MO). All tubing was tested as 100 foot coils of 1/4" OD x 0.020" wall 316L stainless steel. Electropolished tubing had a surface roughness of 10 to 15 microinches. Siltek® treated tubing was finished with 5µm of amorphous silicon, followed by a surface functionalization to increase inertness and hydrophobicity.

Reference courtesy of O'Brien Corporation, available on request from Restek.

Figure 3 In chloride environments, Silcosteel®-CR treated stainless steel outperforms untreated metal by an order of magnitude (ASTM G 48, Method B).

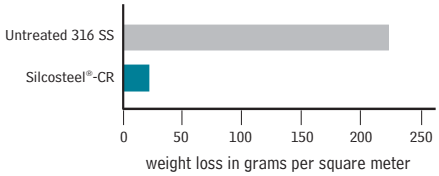
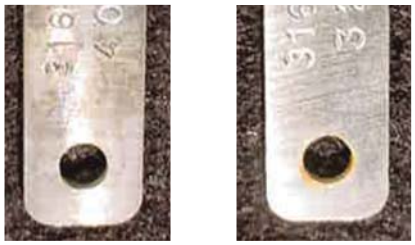


Figure 4 Silcosteel®-CR treated 316L stainless steel shows no sign of attack after 4000-hour salt spray exposure (ASTM B117).



Silcosteel®-CR treated | untreated

Figure 5 Silcosteel®-CR treated 316L stainless steel shows no crevice corrosion and only slight pitting corrosion after 72-hour exposure to ferric chloride; untreated steel exhibits severe crevice corrosion.

Silcosteel®-CR treated | untreated

Siltek®- and Silcosteel®-CR-Treated Electropolished Tubing

- Exceptional inertness.
- Improved reliability and reproducibility; longer lifetime.
- Use with treated fittings for the most inert sample pathway available.

			Price-per-foot			
ID	OD	cat.#	5-24 ft.	25-99 ft.	100-299 ft.	>300 ft.
Siltek®-Treated Electropolished Tubing						
0.085"	1/8"	22538				
0.180"	1/4"	22539				
Silcosteel®-CR-Treated Electropolished Tubing						
0.085"	1/8"	22536				
0.180"	1/4"	22537				

Coiled, Treated, Seamless 316 Grade Stainless Steel Tubing

			Price-per-foot			
ID	OD	cat.#	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
Silcosteel®-CR -Treated 316L Tubing**						
0.055" (1.40mm)	1/8" (3.18mm)	22896				
0.180" (4.57mm)	1/4" (6.35mm)	22897				
Siltek® Treated 316L Tubing**						
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.

**0.035" wall thickness

Note: (required length in meters) x (3.2808) = length in feet.

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Learn more about our precisely applied, highly durable surface treatments:
www.restekcoatings.com

Simple HPLC Analysis for Sudan Dyes

Monitor Sudan I, II, III, and IV in a Single, Isocratic Analysis

By Julie Kowalski, Innovations Chemist

- Ultra Aqueous C18 HPLC column separates the four Sudan dyes in 20 minutes.
- Simple methanol and water mobile phase; two wavelengths detect all four dyes.
- Two wavelengths detect all four dyes.

Sudan dyes are synthetic industrial azo-dyes traditionally used in waxes, plastics, oils, and polishes. Although recognized as carcinogens, Sudan dyes recently have been found in food products in some European countries. They are added to foods such as chili powders to mimic, intensify, and prolong the appearance of natural red hues. In the UK, more than six hundred products containing Sudan dyes have been recalled, the largest food recall in British history.[1]

Sudan dyes are categorized as Class 3 carcinogens by the International Agency for Research on Cancer (IARC) and, therefore, are illegal as food additives according to both the FDA and the EU. The European Commission requires products to have documentation confirming the absence of Sudan dyes.[2,3] Since 2003, European nations have required random product testing and testing of suspected adulterated products. Items found to contain Sudan dyes must be disposed of as hazardous waste.[4]

Laboratories performing analyses for Sudan dyes are not required to follow defined methods. The EU has set detection limits at 0.5-1 mg/kg, and any food material containing more than the limit should be withdrawn from the market.[1] Here, we describe a simple reversed phase HPLC separation of Sudan I, Sudan II, Sudan III, and Sudan IV (Scarlet Red).

We prepared 1mg/mL stock solutions of Sudan I or Sudan II in HPLC grade methanol, and equivalent solutions of Sudan III or Sudan IV in ethyl acetate. To avoid reductive cleavage, we stored the stock solutions at 4°C in foil-wrapped containers. We prepared sample solutions by combining the four stock solutions and diluting with methanol to 20µg/mL each dye. We used a 150 x 4.6mm Ultra Aqueous C18 HPLC column (cat.# 9178565) for the analysis.

Results

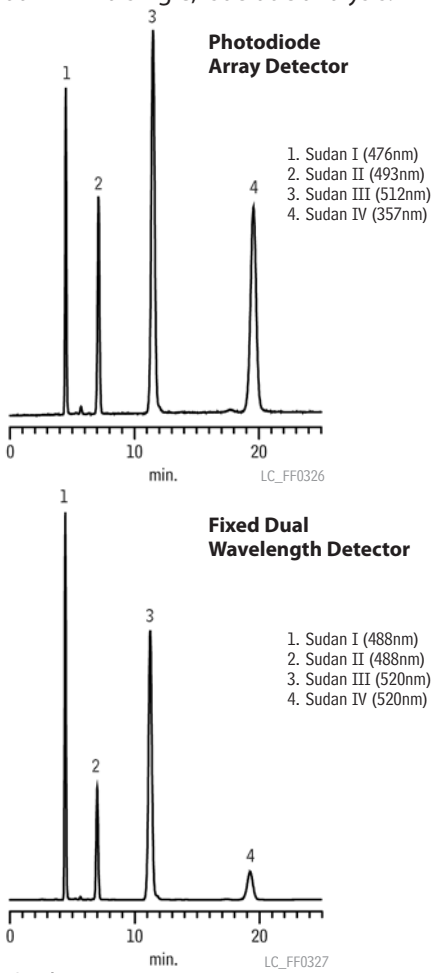
Figure 1 shows the Ultra Aqueous C18 column separates the four dyes in approximately 20 minutes. Sudan I can be detected at 476nm or 418nm, Sudan II at 493nm or 604nm, Sudan III at 508nm to 512nm, and Sudan IV at 357nm or 520nm. For each dye except Sudan III, we observed the higher response at the first listed wavelength; for Sudan III there was little difference. The dyes can be detected by monitoring at 488nm for Sudan I and II and at 520nm for Sudan III and IV, allowing all four dyes to be detected with a fixed dual wavelength instrument.

This method is simple, yet efficient, requiring only a simple mobile phase, isocratic elution, and detection at two wavelengths. The Ultra Aqueous C18 column provides the selectivity needed to assure the separation.

References

1. http://www.ift.org/news_bin/news/newsBody.shtml
2. Commission Decision of 20 June 2003 on emergency measures regarding hot chili and hot chili products, notified under document number C(2003) 1970, (2003/460/EC), OJ L. 154/114, 21.6.2003.
3. Implementation of Commission Decision 2003/460/EC of 21 January 2004.
4. http://www.food.gov.uk/foodindustry/guidancenotes/foodguid/sudanguidance

Figure 1 Monitor Sudan I, II, III and Sudan IV in a single, isocratic analysis.



Sample:
Inj.: 20µL, mixture of Sudan I, Sudan II, Sudan III, Sudan IV
Conc.: 20µg/mL each component
Sample diluent: methanol

Column: **Ultra Aqueous C18 (cat.# 9178565)**
Dimensions: 150 x 4.6 mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: methanol:water, 97:3
Flow: 1mL/min.
Temp.: ambient
Det.: UV @ 476 / 493 / 512 / 357 nm (top) or UV @ 488 / 520 nm (bottom)

for **more** info

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

Ultra Aqueous C18 Column (USP L1)

5µm Column, 4.6mm	cat. #
150mm	9178565

Enhanced Electronic Leak Detector

A Leak-Free System Stabilizes Baselines and Lengthens Column Life

By Donna Lidgett, GC Accessories Product Marketing Manager

new for 2005

- Reliable thermal conductivity leak detector—every analyst should have one.
- Compact, portable, ergonomic design—easy to hold and operate.
- Sensitive—detects helium or hydrogen at $1x10^{-4}$ cc/sec*.
- Fast results—responds to leaks in less than 2 seconds, zeros with the touch of a button.
- Built-in rechargeable battery—charging adaptor included.

tech tip

Avoid poor chromatography caused by leaks—check for leaks with the Restek Leak Detector

In continuing our efforts to provide chromatographers with the best available columns, tools, and accessories, we have enhanced our popular Restek Electronic Leak Detector. New features include internal battery charge capability, a low-battery indicator, a battery charge indicator light, yellow lights to signal a nitrogen leak, a repositioned on/off switch, to eliminate accidentally powering on the unit, and a new probe tip design that prevents debris from entering the unit. The new leak detector retains the microchip technology that enables high sensitivity in a compact unit, the autozero feature that allows instantaneous zeroing with the touch of a button, and the ergonomic design that puts all controls at your fingertips, for maximum ease of use.

Easy-to-clean probe assembly.

The new Restek Electronic Leak Detector is the affordable solution for detecting helium, hydrogen, or nitrogen leaks in your GC system. Leaks can cause detector noise and baseline instability, waste carrier gas, and shorten column lifetimes. The leak detector responds in less than 2 seconds to leaks of gases with thermal conductivities different from air, indicating leaks with both an audible alarm and an LED readout. The leak detector detects minute gas leaks that can go undetected by liquid leak detectors. And, remember—you should never use liquid leak detectors on a capillary system, because liquids drawn into the system through the leaks will contaminate the system.

Description	qty.	cat.#
Leak Detector with 110Volt Battery Charger	ea.	22451
Leak Detector with 220Volt European Battery Charger	ea.	22451-EUR
Leak Detector with 220Volt UK Battery Charger	ea.	22451-UK

Caution: The Restek Electronic Leak Detector is NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.
*Sensitivity measured using helium.

Verify pinpoint leaks with the adaptor fitting.

Leak Detector Accessory Kit

The kit includes an adaptor fitting that fits over the probe assembly to detect very small leaks in hard-to-reach locations, and a mounting bracket that can be affixed to the wall or GC.

Description	qty.	cat.#
Leak Detector Accessory Kit (adaptor fitting for probe, mounting bracket)	kit	22453

Leak Detector is easily accessed when stored in the mounting bracket.

Genuine Restek HPLC Parts and Accessories

The parts and tools you need to keep your HPLC systems running smoothly

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

- Restek quality and reliability.
- Renowned Restek Plus 1™ service.

Genuine Restek Replacement Parts for ThermoSeparation Products HPLC Systems

Restek offers replacement parts for Agilent, Beckman, Hitachi, PerkinElmer, Shimadzu, Waters, and ThermoSeparation Products HPLC systems—all designed to equal or exceed the performance of original equipment manufacturers' parts. We've listed parts for ThermoSeparation Products HPLC Systems here. For parts for other systems, refer to our catalog, or visit our website. Use Genuine Restek Replacement Parts to keep your system in peak condition!

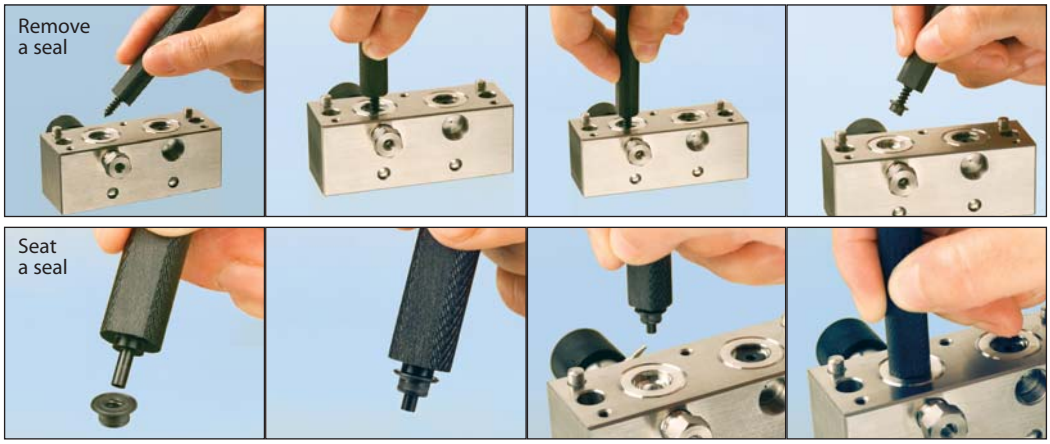
Description	Model #	Similar to SP/TSP part #	qty.	cat.#
Inlet Check Valve Assembly	SP8800 & P-Series Pumps	A3495-010	ea.	25474
Outlet Check Valve Assembly	SP8800 Series Pumps	A3490-010	ea.	25475
Piston	SP8800 & P-Series Pumps	A3102-010	ea.	25476
Back-up Seal	SP8800 & P-Series Pumps	A2963-010	ea.	25477
Plunger Seal, Gold Superseal	SP8800 & P-Series Pumps	A2962-010	ea.	25478
Check Valve and Transducer Assembly	P-Series Pumps	A3990-010	ea.	25479
Kel-F® Washer	P-Series Pumps	A2973-010	ea.	25480
Rotor Seal Assembly, Rheodyne® 7010	TSP AS100, 300, 1000, 3000, 3500, 8875, and 8880 Autosamplers	7010-039	ea.	25481
Syringe Assembly, 250µL	TSP AS100, 300, 1000, 3000, 3500, 8875, and 8880 Autosamplers	A3588-020	ea.	25482
Syringe, 500µL	TSP AS100, 300, 1000, 3000, 3500, 8875, and 8880 Autosamplers	A3588-010	ea.	25483
Lamp, UV	Linear UV-200, 203, 204, 205, 206, and UV 100, 150, 1000, and 2000 Detectors	9551-0023	ea.	25484

Description	Model #	Similar to TSP part #	qty.	cat.#
Check Valve Cartridge	LDC Constametric Pumps	900946	ea.	25485
Sapphire Plunger	LDC Constametric Pumps	801306	ea.	25486
Plunger Seal Kit, Gold	LDC Constametric Pumps	31-36-00754	ea.	25487
Plunger Seal, Black	LDC Constametric Pumps	206129001	ea.	25488
Plunger Seal, Gold	LDC Constametric Pumps	206156001	ea.	25489
Lamp, Deuterium	LDC SM-I, II, III, 3000, 3100, 3100X, and 4000 Detectors	108035	ea.	25490
Lamp, Deuterium Pre-aligned	LDC 3200 and 4100 Detectors	900918001	ea.	25491

Use the flat side of the Piston Seal Insertion Tool to seat a Waters face seal.

HPLC Piston Seal Insertion Tool

Simplify pump maintenance: use one end to remove your old seal, then simply slip your new seal on the other end and push it flush into position. The tool cannot mar the surrounding metal surface of the pump housing.



Description	qty.	cat.#
HPLC Piston Seal Insertion Tool	ea.	21356

PEEK® Unions, Connectors, and Tubing

Restek offers a wide range of PEEK® and stainless steel unions, connectors, and tubing, for installing and maintaining your HPLC systems. For complete listings, see our current catalog, or visit our website.

PEEK® Union Connector

Allows you to quickly and reliably connect two pieces of 1/16-inch tubing. End fittings included.

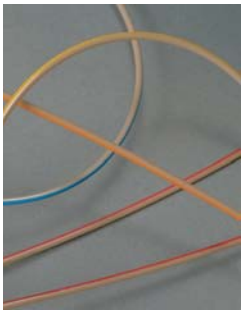
Description	qty.	cat.#
PEEK® Union Connector 1/16"	2-pk.	25323

Universal 10-32 PEEK® Column Connectors and Plugs

Universal PEEK® Connectors allow easy installation of all 1/16-inch tubing, including stainless steel.

Description	qty.	cat.#
PEEK® Column Connector (beige, round body)	10-pk.	25015
PEEK® Column Plug (black)	10-pk.	25016
PEEK® Fingertight Fittings (blue, flat-sided)	10-pk.	25324

Inert PEEK® Tubing



- Replaces stainless steel, titanium, Teflon® or Tefzel® tubing.
- Less oxygen permeable and more temperature resistant (to 250°C) than Teflon® or Tefzel® tubing.
- Use with PEEK® fingertight or flangeless fittings.
- Use to 7,000psi.

Description	Color Code	qty.	cat.#
PEEK® Tubing, 1/16" OD x 0.0025" ID x 1m	natural	3-pk.	25320
PEEK® Tubing, 1/16" OD x 0.005" ID x 3m	red stripe	ea.	25065
PEEK® Tubing, 1/16" OD x 0.007" ID x 3m	yellow stripe	ea.	25066
PEEK® Tubing, 1/16" OD x 0.010" ID x 3m	blue stripe	ea.	25067
PEEK® Tubing, 1/16" OD x 0.020" ID x 3m	orange stripe	ea.	25068

HPLC 30-Column Storage Cabinet

Tired of stacks of HPLC columns on your lab benches? This easy-to-install cabinet saves space and protects columns; the hinged door is clear to allow quick identification of column labels or tags.

Description	dimensions	qty.	cat.#
30 Column Cabinet	17 3/8 x 15 x 2 7/8"	ea.	25159

*Please note: Columns in photograph are not included.

Teflon® Tubing

- Ideal for mobile phase inlet lines.
- Chemically inert.
- Use to 500psi and 80°C.

Description	qty.	cat.#
Teflon® Tubing, 1/8" OD x 0.063" ID x 3m (1.6mm ID)	3m	25306
Teflon® Tubing, 1/8" OD x 0.094" ID x 3m (2.4mm ID)	3m	25307

Opti-Cap™ Bottle Top

The most economical way to helium-sparge and deliver HPLC mobile phases. The Opti-Cap™ top fits all standard GL-45 bottles and has two 1/8-inch holes and one 1/16-inch hole for tubing.

Description	qty.	cat.#
Opti-Cap™ (Cap and PEEK® Plug)	ea.	25300
Opti-Cap™ Kit (Opti-Cap™, 3 meters of tubing, sparging filters)	kit	25301
Opti-Cap™ Kit with 1L Bottle	kit	25302
Opti-Cap™ Kit with 2L Bottle	kit	25303
Related items and replacement parts	**qty.**	**cat.#**
Mobile Phase Sparge Filter: 2µm, stainless steel	ea.	25311
Mobile Phase Inlet Filter: 10µm	ea.	25312
Teflon® Tubing, 1/8" OD x 0.094" ID x 3m (2.4mm ID)	3m	25307
Teflon® Tubing, 1/8" OD x 0.063" ID x 3m (1.6mm ID)	3m	25306
PEEK® Plug, 1/4"-28 threads	3-pk.	25319
1L Graduated Safety-Coated Bottle – GL-45 threads	ea.	25304
2L Graduated Safety-Coated Bottle – GL-45 threads	ea.	25305

Opti-Cap™ Kit with bottle

Genuine Restek Supplies & Accessories for ASE® Systems

Extraction Cell Parts, Collection Vials, PEEK® Washers, Filters

save!

Economical pricing.

By Neil Mosesman, Sample Preparation Product Marketing Manager

Extraction Cell Parts for ASE® Systems

- Designed to meet or exceed performance of original manufacturer's parts.
- Polished inner surfaces for easier cleaning; Siltek® deactivation available.

In addition to stainless steel extraction cell parts, we offer bodies, caps, and frits finished with our innovative Siltek® treatment, to greatly improve inertness and, therefore, the reliability of analytical results for active compounds.

		Stainless Steel		Siltek®-Treated	
Description	Similar to Dionex part #	qty.	cat.#	qty.	cat.#
Parts for ASE® 200 Extraction Cells					
Extraction Cell Body for ASE® 200, 1mL	054973	ea.	26110	ea.	26111
Extraction Cell Body for ASE® 200, 5mL	054974	ea.	26112	ea.	26113
Extraction Cell Body for ASE® 200, 11mL	048820	ea.	26114	ea.	26115
Extraction Cell Body for ASE® 200, 22mL	048821	ea.	26098	ea.	26099
Extraction Cell Body for ASE® 200, 33mL	048822	ea.	26116	ea.	26117
Replacement Extraction Cell End Caps for ASE® 200	049450	2-pk.	26096	2-pk.	26097
Replacement Frits for ASE® 200	049453	10-pk.	26100	10-pk.	26101
Parts for ASE® 300 Extraction Cells					
Extraction Cell Body for ASE® 300, 10mL		ea.	26172	ea.	26173
Extraction Cell Body for ASE® 300, 34mL		ea.	26176	ea.	26177
Extraction Cell Body for ASE® 300, 66mL	056696	ea.	26178	ea.	26179
Extraction Cell Body for ASE® 300, 100mL	056693	ea.	26132	ea.	26133
Replacement Extraction Cell End Caps for ASE® 300	056921	2-pk.	26170	2-pk.	26171
Replacement Frits for ASE® 300/100		6-pk.	26174	6-pk.	26175

Accessories for ASE® Systems

Meet original equipment manufacturer's performance.

PEEK® Washers for ASE® Extraction Units

Description	Similar to Dionex part #	qty.	cat.#
Accessories for ASE® 200 Systems			
PEEK® Washers for ASE® 200	049454	12-pk.	25256
PEEK® Washers for ASE® 200	049454	48-pk.	25257
PEEK® Washers for ASE® 200	049454	250-pk.	26120
Snap Rings for Caps for ASE® 200	049456	10-pk.	26184
Funnel for ASE® 200	056958	ea.	26180
Accessories for ASE® 300/100 Systems			
PEEK® Washers for ASE® 300	061687	12-pk.	25393
PEEK® Washers for ASE® 300	061687	48-pk.	25394
Snap Rings for Caps for ASE® 300/100	056778	12-pk.	26134

20mm Filters for ASE® 200 Extraction Cells

Consistent porosity, to deliver rapid flow rates and protect the metal frit in the cell from contamination.

Description	Similar to Dionex part #	qty.	cat.#
Cellulose Filters for ASE® 200	049458	100-pk.	26118
Glass Fiber Filters for ASE® 200	047017	100-pk.	26119

Diatomaceous Earth

Mix with densely packed samples such as clays to improve extraction efficiencies and absorb excess moisture.

Description	Similar to Dionex part #	qty.	cat.#
Diatomaceous Earth, 30/40 mesh	062819	1kg	26033

60mL Sample Collection Vials

Cleaned to EPA specifications and supplied assembled with caps and septa.

Description	Similar to Dionex part #	qty.	cat.#
60mL Collection Vials, Clear Glass, for ASE® Systems	048784	72-pk.	26121
60mL Collection Vials, Amber Glass, for ASE® Systems	048781	72-pk.	26122

Syringe Filters

Top-Quality Filters—Great Prices

By Neil Mosesman, Sample Preparation Products Marketing Manager

- Nylon - PTFE - PVDF membranes.
- 13mm and 25mm diameter.
- 0.22µm and 0.45µm porosity.
- Color coded for easy identification.
- 100 filters, reusable storage container.

Size	Porosity	qty.	cat.#
Nylon			
13mm	0.22µm	100-pk.	26146
13mm	0.45µm	100-pk.	26147
25mm	0.22µm	100-pk.	26148
25mm	0.45µm	100-pk.	26149
PTFE (polytetrafluoroethylene)			
13mm	0.22µm	100-pk.	26142
13mm	0.45µm	100-pk.	26143
25mm	0.22µm	100-pk.	26144
25mm	0.45µm	100-pk.	26145
PVDF (polyvinyldifluoride)			
13mm	0.22µm	100-pk.	26150
13mm	0.45µm	100-pk.	26151
25mm	0.22µm	100-pk.	26152
25mm	0.45µm	100-pk.	26153

Bulk Adsorbents

For Thorough Sample Preparation and Reliable Results

By Neil Mosesman, Sample Preparation Products Marketing Manager

Florisil® PR

- Pesticide residue grade.
- Each lot certified to meet the requirements of AOAC methodology.
- Packaged in glass containers.

Florisil® PR is commonly used to remove polar interferences from pesticide residues. This bulk material is ideal for labs packing their own chromatography columns for pesticide residue extractions.

Description	qty.	cat.#
Florisil® PR, 60/100 mesh	500gms	26135

Granulated Activated Copper

- Convenient form for removing sulfur from environmental extracts.
- Acidified and activated—ready for use.

Activated copper effectively removes elemental sulfur from environmental extracts. Our acid washed and activated material can be used right out of the package. The 30 mesh granular material eliminates the potential for fine copper particles in filtered extracts.

Description	qty.	cat.#
Granulated Activated Copper, 30 mesh	1kg	26136

Ottawa Sand

- Sample medium for matrix spikes and laboratory control blanks.
- Packaged in convenient 5kg buckets.

Ottawa sand is organics free and is listed in several US EPA methods as the specified medium for matrix spike and laboratory control blanks.

Description	qty.	cat.#
Ottawa Sand	5kg	26137

Instrument Innovations!

Simplify Your Analyses for Volatile Organic Compounds

by Donna Lidgett, GC Accessories Product Marketing Manager

Purge-and-Trap Spargers for Tekmar 2000, 3000, or 3100 GCs

- Available with uniform frits, to ensure maximum purging efficiency.
- Use non-fritted spargers for wastewater samples.
- Manufactured to tight tolerances to ensure a leak-tight seal.

Description	qty.	cat.#
Fritted Spargers, 1/2-inch mount		
5mL Fritted Sparger	ea.	21150
10mL Fritted Sparger	ea.	26138
25mL Fritted Sparger	ea.	21151
Non-Fritted Spargers, 1/2-inch mount		
5mL Non-Fritted Sparger	ea.	26139
10mL Non-Fritted Sparger	ea.	26140
25mL Non-Fritted Sparger	ea.	26141

Moisture Control By-Pass Lines for Tekmar Instruments

- Increase response for ketones, alcohols, and acetates.
- Silcosteel®-deactivated tubing for increased inertness.
- Suitable for US EPA Methods 8260, 524.2, and OLM4.1.
- Easily attaches in minutes.

Description	qty.	cat.#
Moisture Control By-Pass Line for Tekmar 3000 Purge & Trap	ea.	21035
Moisture Control By-Pass Line for Tekmar 3100 Purge & Trap	ea.	21109

ELCD Nickel Reaction Tubes

- Pretreated for maximum sensitivity.
- Quality-controlled for reliability.
- Available for many popular models.

	To replace these instrument part numbers:					Order these Restek part numbers:	
ELCD Model #	Tremetrics	Varian	PerkinElmer	Shimadzu	O.I. Analytical	qty.	cat.#
Hall 700A	115439-0003	00-996724-14	0330-2675	—	—	2-pk.	21580
Hall 1000	117459-0003	00-997625-12	N660-1072	220-90435-00	—	2-pk.	21581
O.I. 4420	—	—	—	—	260323	2-pk.	21582

Cleaned Teflon® Transfer Lines for ELCDs

We stringently clean our ELCD Teflon® transfer lines with an HCl solution to remove any contaminants, then rinse with methanol. Convenient 6.5-inch precut pieces that directly interface the nickel reaction tube and conductivity cell in Tracor, Tremetrics, O.I., and many other ELCDs.

Description	qty.	cat.#
Teflon® Transfer Lines for ELCDs (five 6.5-inch lines)	5-pk.	20121

Replacement Accesories for Hall 1000

ELCD Nickel Reaction Tube Nut

High-quality stainless steel ELCD nut mounts nickel reaction tube into ELCD.

Description	qty.	cat.#
ELCD Nickel Reaction Tube Nut	2-pk.	21584

1/16-Inch Vespel®/Graphite Sealing Ring

Installs onto the nickel reaction tube after the screw. Easily compresses on the reaction tube to provide a leak-tight seal and prevent detector oxidation.

Description	qty.	cat.#
1/16-Inch Vespel®/Graphite Sealing Ring	2-pk.	21583

Cool Tools!

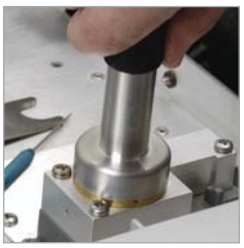
Restek Innovations Save You Time and Money

Spanner Wrench for Agilent 5890/6890 FID Collector Assembly

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

Remove the collector housing...

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



...then turn counterclockwise...

...and remove.

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

Injector Wrench for Agilent 5890/6890/6850 GCs

- Use to remove the septum nut and weldments during GC maintenance.
- High-quality stainless steel construction.
- Meets original equipment performance.

Use the smaller end to remove the septum nut.

Use the larger end to tighten the split/splitless weldment nut.

Description	Similar to Agilent part #	qty.	cat.#
Injector Wrench for Agilent 5890/6890/6850 GCs	19251-00100	ea.	22065

Injector Wrench for Shimadzu 17A and 2010 GCs

- Designed specifically for removing Shimadzu injection ports.
- High-quality stainless steel construction.

Description	Similar to Shimadzu part #	qty.	cat.#
Injector Wrench for Shimadzu 17A and 2010 GCs	221-46977-00	ea.	21159

1/4- to 5/16-inch Open-End Wrench Set

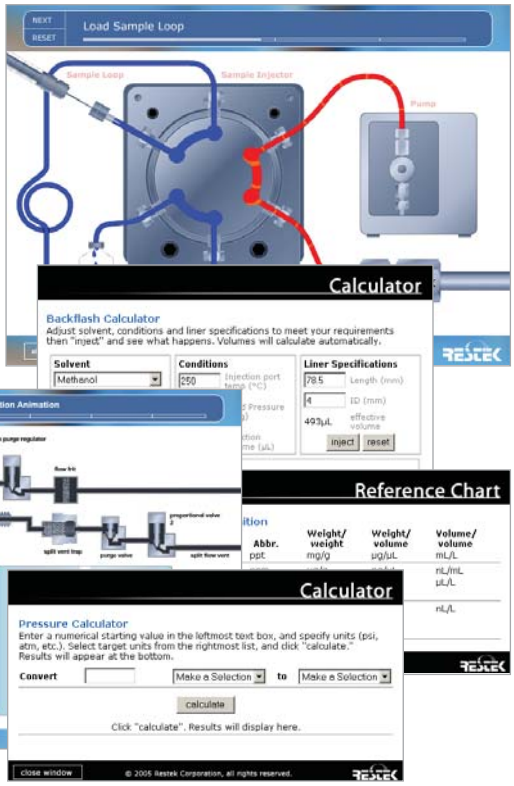
We examined many different wrenches before we decided to offer this high-quality pair for tightening capillary fittings.

Description	qty.	cat.#
Open-End Wrenches (1/4" x 5/16")	2-pk.	20110

www.restek.com/expert

Restek Website Expert Center

Need a conversion factor in a hurry? Want to see where the sample goes in a capillary GC split injection? Visit the Expert Center on the Restek website. Located in the "Info. & Support" menu, the Expert Center includes a tremendous variety of useful information. Our **calculators**, for example, are extremely handy tools. Use the Backflash Calculator to determine how much sample in a particular solvent you can introduce into a capillary inlet liner without the expanding sample backflashing into, and contaminating, the system. Use the Pressure Calculator to quickly interconvert among the various measurements of pressure: psi, atm, kg/cm^2, Torr, inches Hg, kPa, bar.



The **animations** of capillary GC injection techniques and operation of a 6-port HPLC valve enable you to view these processes, and help you appreciate the benefits, and potential problems, associated with each. The subjects in the Expert Center include:

Troubleshooting
- Chromatogram Archive
- Optimization Calculators
- GC Column Selection
- Capillary Column Installation Guide
- Free Technical Literature
- and more

Optimization Calculators and Reference Charts
- Backflash Calculator
- GC Column Characteristics
- Pressure Calculator
- Reference Charts for Setting Deadtime
- Methane Retention
- Pressure Conversion
- Weight/Volume Composition Measures
- Mesh Size Conversions
- Septum Size Chart
- GC Retention Time Indexes

Animations
- Direct Injection
- Split Injection
- Splitless Injection
- HPLC 6-Port Valve

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