

THE RESTEK ADVANTAGE

Turning Visions into Reality

2004 vol. 4



Fast, Sensitive GC Analyses of Semivolatile Organics in Water

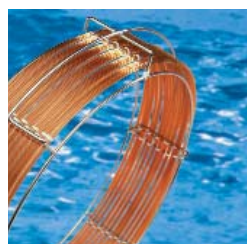
Using an 0.18mm ID Rtx®-XLB Column

new!

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,

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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 →

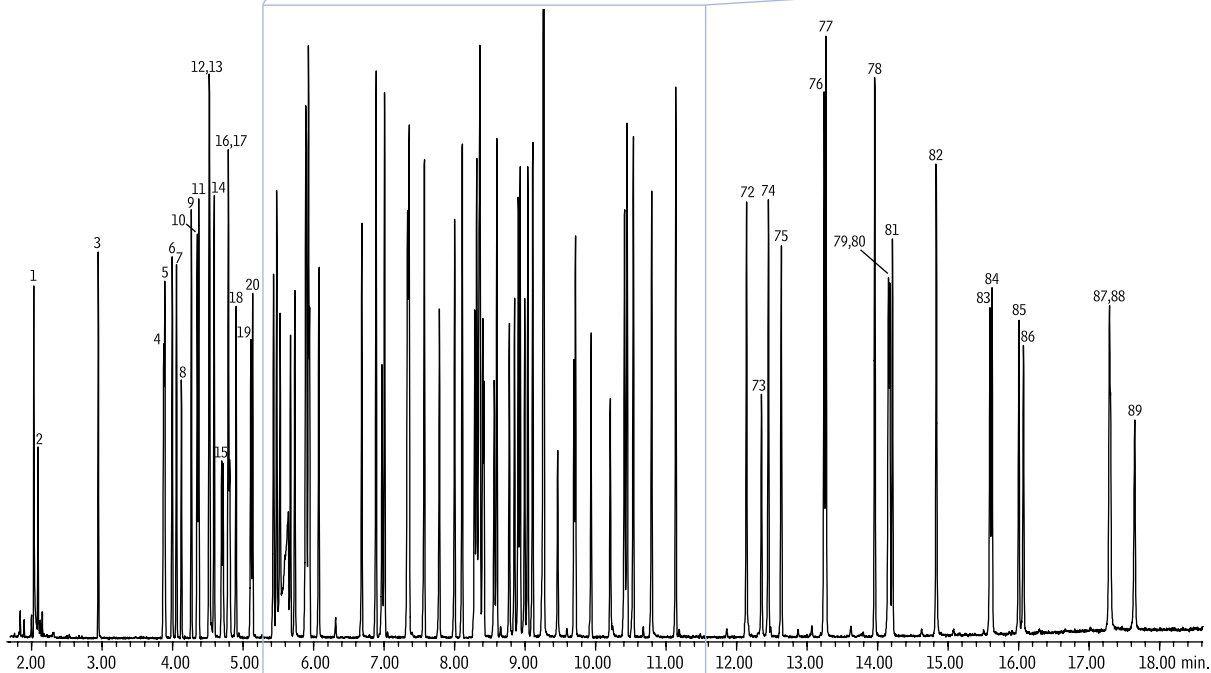
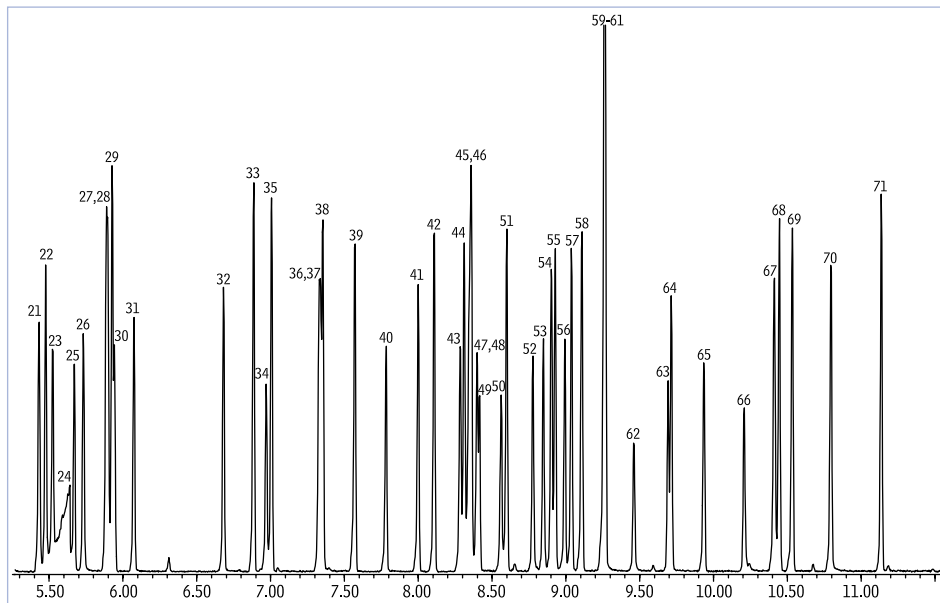
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Figure 1

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



GC_EV00747

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

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



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

**Requires warming and sonication before use.

Benzoic Acid

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

Benzenidine

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.)

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