Excellent Responses in GC/MS Analysis of Semivolatiles

For Low Level GC/MS Analysis

By Robert Freeman, Environmental Innovations Chemist

- Inert, low-bleed column for reliable results from low-level GC/MS analyses.
- Save time – analyze acidic and basic compounds under the same conditions.
- Guaranteed reproducible performance, column to column.

The second column in our new Rxi™ GC column line – the Rxi™-1ms column – will provide the same outstanding performance as the Rxi™-5ms column, with equally superior inertness, ultra-low bleed, and excellent batch to batch reproducibility.

Our first test for this 100% dimethylpolysiloxane phase column was an analysis of a complex mixture of semivolatile organic compounds. The extensive target list was comprised of many classes of compounds including chloroacetanilides, chlorotriazines, triazinones, uracils, polycyclic aromatic hydrocarbons, and phthalates. Figure 1 shows peak shape and selectivity are equally good for all of these diverse compounds, and all are eluted in an acceptable analysis time.[1]

Excellent Inertness

In addition to analyzing these compounds, we analyzed an acidic compound (2,4-dinitrophenol) and a basic compound (pyridine), each at 0.5ng on column, to assess column inertness. Column activity reveals itself through poor response and peak tailing for such active compounds, and these two compounds present both varying difficulties in a GC/MS analysis and differing modes of degradation. Figure 2 shows the excellent peak shapes and responses for these compounds on the 30m x 0.25mm ID, 0.25µm film column.

Phenols are notorious for breakdown and peak tailing, caused by interaction with the surface of an active inlet liner or an active column. Nitrophenols and pentachlorphenol, for example, very often exhibit poor peak shape and/or poor response. Figure 3 shows the 30m x 0.25mm ID, 0.25µm Rxi™-1ms column provides very good peak shapes for phenols. Peak responses are well above method requirements.

Ultra-Low Bleed

In addition to excellent inertness, Rxi™-1ms columns exhibit very low bleed. Figure 4 is focused on the end of the chromatogram for semivolatiles. At 330°C, bleed is much lower than the signals for 0.5ng of target analytes. This exceptional signal-to-noise differential for late eluting compounds assures better detection limits.

Based on these results, we highly recommend the new Rxi™-1ms column for low-level analyses that require a 100% dimethylpolysiloxane phase.

- **GC Column**
  Rxi™-1ms Columns
- **GC Accessories**
  4mm Drilled Uniliner® Inlet Liner

ALSO OF INTEREST

- A 12-Minute Analysis for Volatiles
- Improved SPE Cartridges for Massachusetts EPH Analysis
- New Reference Mix of Canadian Drinking Water Volatiles
- Analysis of Semivolatile Organics

FOOTNOTE

1 The Drilled Uniliner®

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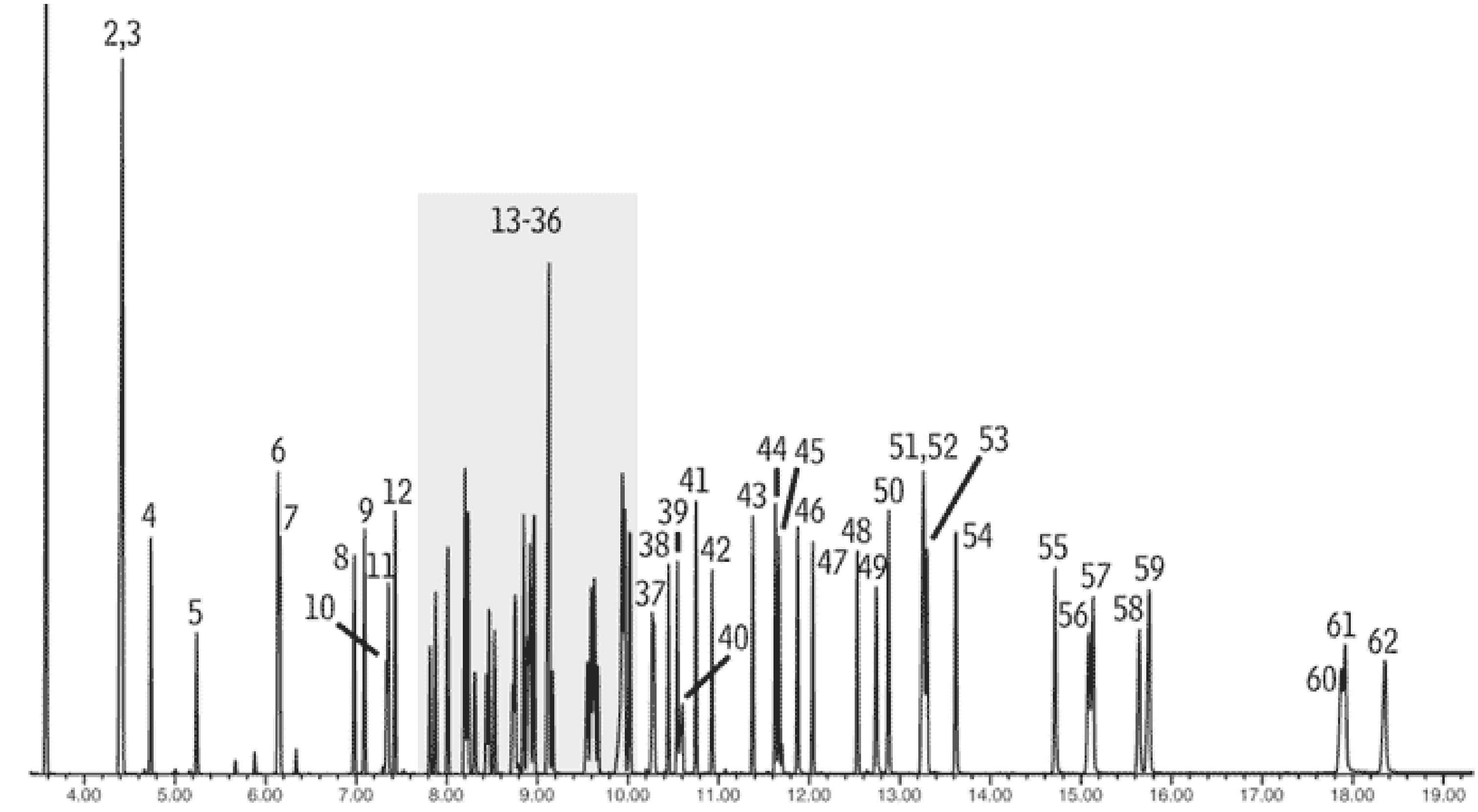
Figure 1 Excellent selectivity and peak shapes for common drinking water semivolatiles at 10ng, using an Rxi™-1ms column.

1. 2-fluorophenol (surr.)
2. bis(2-chloroethyl)ether
3. phenol-d6 (surr.)
4. 1,4-dichlorobenzene-d4 (int. std.)*
5. nitrobenzene-d5 (surr.)
6. naphthalene-d8 (int. std.)*
7. naphthalene
8. 1-methylnaphthalene
9. 2-methylnaphthalene
10. hexachlorocyclopentadiene
11. EPTC
12. 2-fluorobiphenyl (surr.)
13. 2,6-dinitrotoluene
14. dimethylphthalate
15. acenaphthylene
16. acenaphthene-d10 (int. std.)*
17. acenaphthene
18. 2,4-dinitrotoluene
19. 1-naphthalenamine
20. molinate
21. 2-naphthaleneamine
22. 5-nitro-*o*-toluidine
23. diethylphthalate
24. fluorene
25. propachlor
26. diphenylamine
27. 2,4,6-tribromophenol (surr.)
28. simazine
29. prometon
30. atrazine
31. hexachlorobenzene
32. 4-aminobiphenyl
33. terbacil
34. phenanthrene-d10 (int. std.)*
35. phenanthrene
36. anthracene
37. metribuzin
38. acetochlor
39. alachlor
40. bromacil
41. di-*n*-butylphthalate
42. metolachlor
43. fluoranthene
44. pyrene
45. butachlor
46. *p*-terphenyl-d14 (surr.)
47. *p*-dimethylaminoazobenzene
48. benzyl butyl phthalate
49. 2-acetylaminofluorene
50. bis(2-ethylhexyl)adipate
51. benzo(a)anthracene
52. chrysene-d12 (int. std.)*
53. chrysene
54. bis(2-ethylhexyl)phthalate
55. di-*n*-octylphthalate
56. benzo(b)fluoranthene
57. benzo(k)fluoranthene
58. benzo(a)pyrene
59. perylene-d12 (int. std.)*
60. indeno(1,2,3-cd)pyrene
61. dibenzo(a,h)anthracene
62. benzo(ghi)perylene



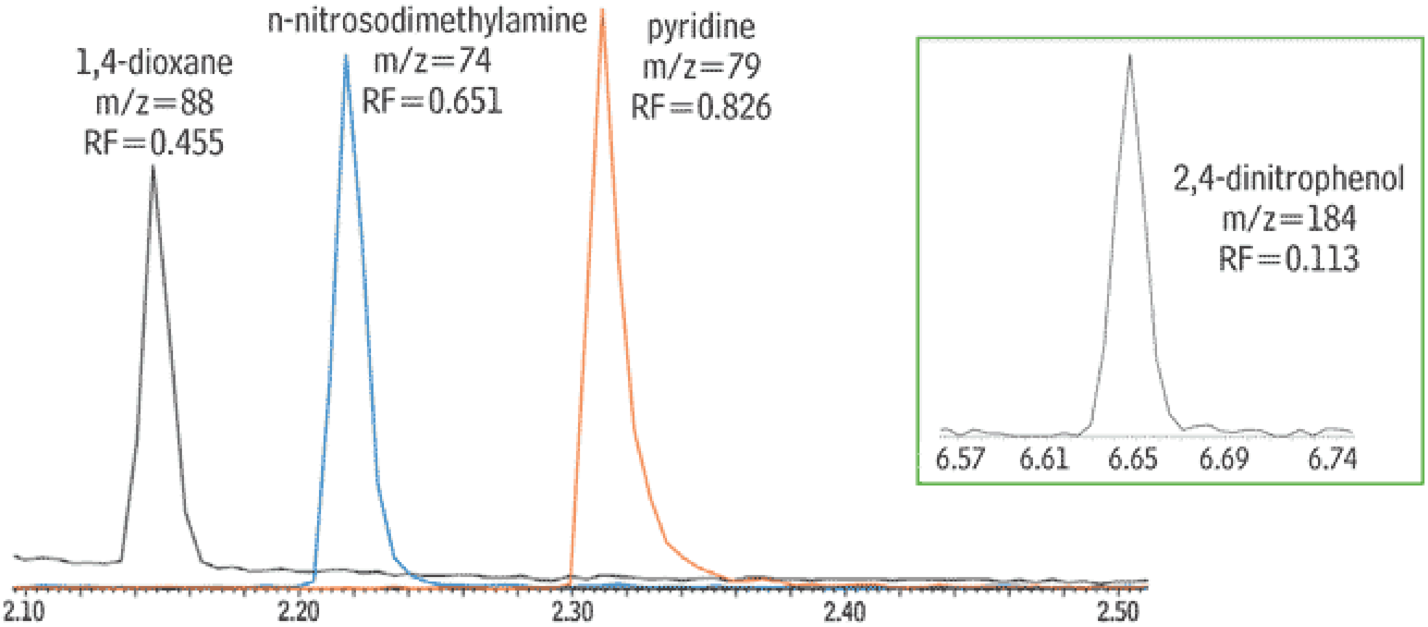
GC_EV00833

Column:	Rxi™-1ms, 30m, 0.25mm ID, 0.25µm (cat.# 13323)
Sample:	US EPA Method 525.2 mix: custom 525.2 calibration mix, SV Internal Standard Mix (cat.# 31206), B/N Surrogate Mix (4/89 SOW) (cat.# 31024), Acid Surrogate Mix (4/89 SOW) (cat.# 31025)
Inj.:	1.0µL, 10µg/mL each analyte (internal standards 100µg/mL), split (10:1) 4mm Drilled Uniliner® inlet liner (hole at bottom) (cat.# 20756)
Instrument:	Agilent 6890
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1.2mL/min.
Oven temp.:	50°C (hold 1 min.) to 265°C @ 20°C/min., to 330°C @ 6°C/min. (hold 1 min.)
Det.:	Agilent 5973 MSD

Transfer line temp.:	280°C
Scan range:	35-550 amu
Solvent delay:	3.20 min.
Tune:	DFTPP
Ionization:	EI

*Internal standards at 100ng on-column.

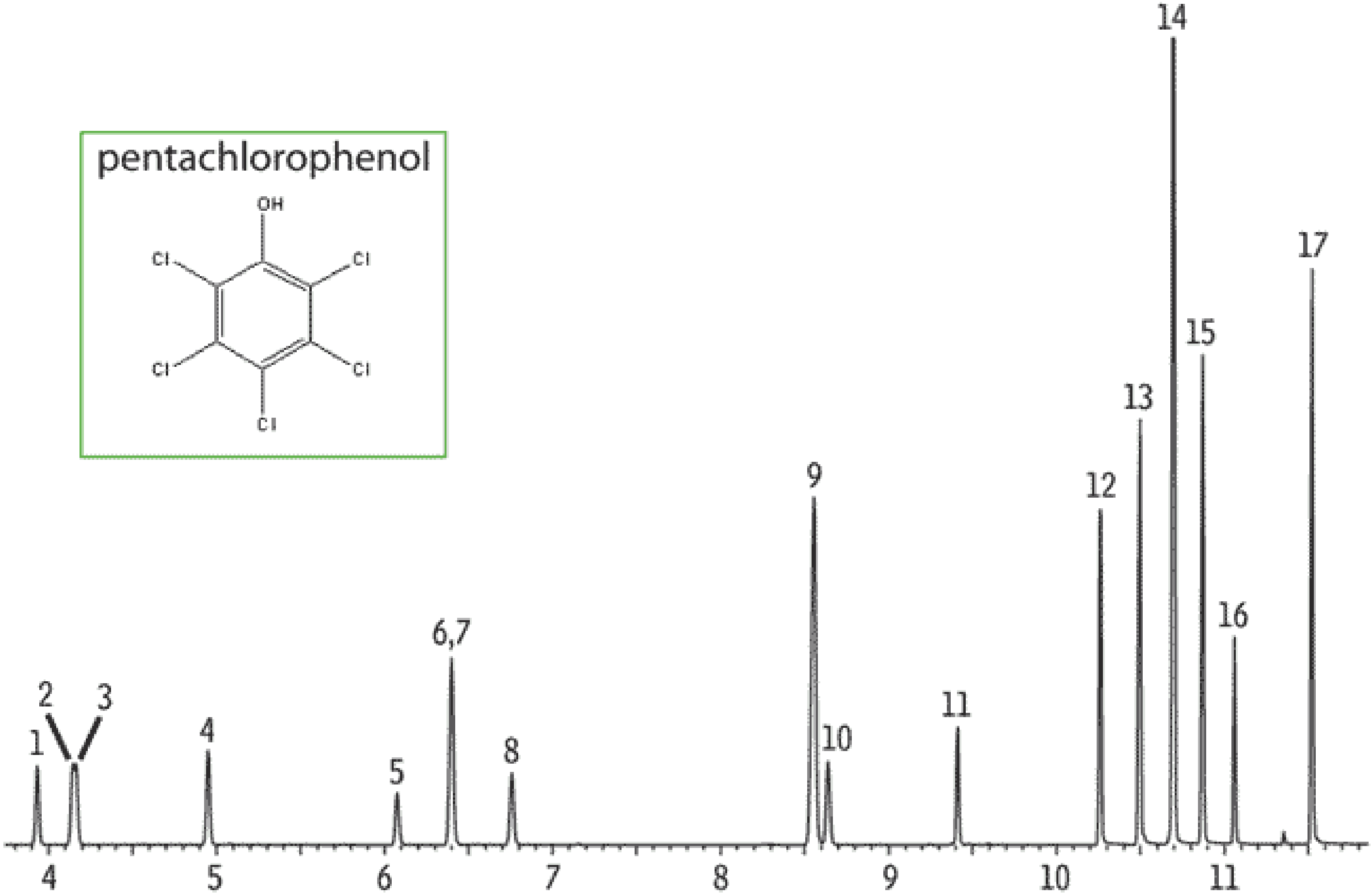
Figure 2 An Rxi™-1ms column has excellent selectivity for basic or acidic compounds, under the same conditions (0.5ng each; extracted ion chromatograms).



GC_EV00830

Figure 3 Symmetric peaks for acidic analytes at 5.0ng on an Rxi™-1ms column (extracted ion chromatogram).

1. phenol
2. 2-chlorophenol-d4 (surr.)
3. 2-chlorophenol
4. 2-methylphenol (*o*-cresol)
5. 2-nitrophenol
6. 2,4-dimethylphenol-3,5,6-d3 (surr.)
7. 2,4-dimethylphenol
8. 2,4-dichlorophenol
9. 3-nitro-*o*-xylene (int. std.)
10. 4-chloro-3-methylphenol
11. 2,4,6-trichlorophenol
12. 2,4-dinitrophenol
13. 4-nitrophenol
14. 2,3,4,5-tetrachlorophenol (int. std.)
15. 2-methyl-4,6-dinitrophenol
16. 2,4,6-tribromophenol (surr.)
17. pentachlorophenol

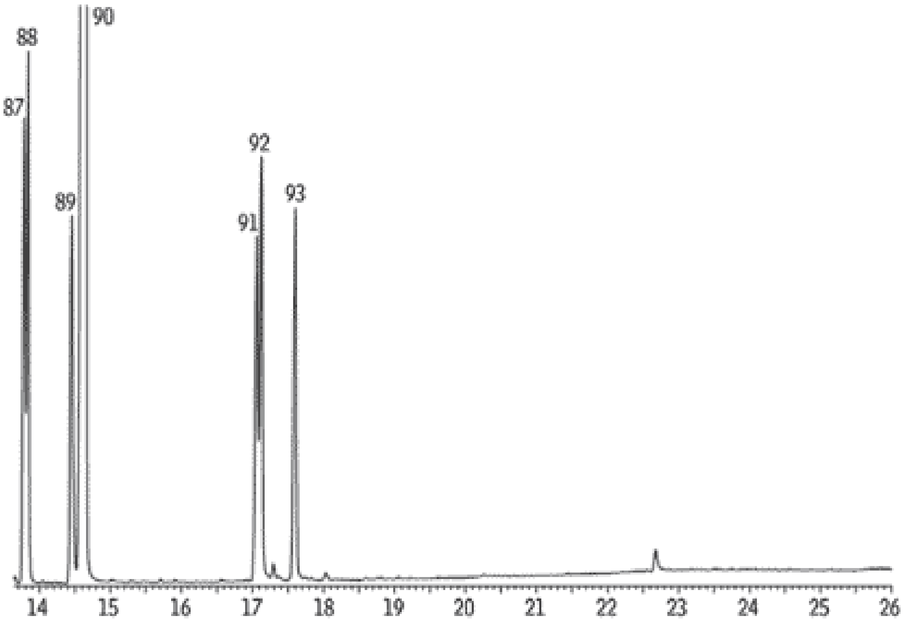


GC_EV00834

Column:	Rxi™-1ms, 30m, 0.25mm ID, 0.25µm (cat.# 13323)
Sample:	US EPA Method 528 Mix: Phenols Fortification Mix, EPA 528 (cat.# 31695), Internal Standard Mix, EPA 528 (cat.# 31696), Surrogate Standard Mix, EPA 528 (cat.# 31697)
Inj.:	1.0µL, 5µg/mL each analyte (internal standards 25µg/mL), split (10:1) 4mm Drilled Uniliner® inlet liner (hole at bottom) (cat.# 20771)
Instrument:	Agilent 6890
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1.2mL/min.
Oven temp.:	70°C (hold 0.5 min.) to 130°C @ 8°C/min., to 300°C @ 50°C/min. (hold 1 min.)
Det.:	Agilent 5973 MSD

Figure 4 Negligible bleed for an Rxi™-1ms column at 330°C (end of analysis for 5ng on-column standard).

87. benzo(b)fluoranthene
88. benzo(k)fluoranthene
89. benzo(a)pyrene
90. perylene-d12 (int. std.)
91. indeno(1,2,3-cd)pyrene
92. dibenzo(a,h)anthracene
93. benzo(ghi)perylene



GC_EV00856

Column:
Rxi™-1ms, 30m, 0.25mm ID, 0.25µm (cat.# 13323)

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The Drilled Uniliner® (Footnote 1)
To reduce the effects of surface activity in the injection port liner, and focus on the effects of the column on active analytes, we used a Drilled Uniliner® inlet liner for this work. This liner connects directly to the column, eliminating contact between the active compounds and active metal surfaces in the injector, and ensuring an inactive sample pathway for analyte transfer from the injection port to the column.