Analysis of Semivolatile Organics

Using the new Rxi™-5ms Capillary GC Column

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- Low column bleed, outstanding inertness, excellent column-to-column reproducibility.
- Symmetric peaks and good response factors for acidic or basic analytes.
- Resolve 93 analytes in less than 18 minutes.

Sub-nanogram Analysis of Semivolatile Organics

Analyzing basic or acidic semivolatile environmental pollutants at low nanogram-on-column concentrations puts demands on the entire analytical system. Using our new Rxi™-5ms column, we have developed an analytical procedure that assures good performance for both acids and bases.

Rxi™-5ms Column Offers Sensitivity for Acids and Bases

One of the most active basic compounds listed in semivolatiles methods is pyridine. This early-eluting compound can elicit poor performance in the injection port and on the column, and many currently available columns give a poor peak shape for pyridine. Columns with a slightly basic surface can perform well with pyridine, but will perform poorly with the acidic compounds, such as 2,4-dinitrophenol.

Figure 1 combines extracted ion chromatograms for the initial three US EPA Method 8270D target compounds, at 0.5ng per compound on-column. The extracted ion for 1,4-dioxane shows that injection port and oven conditions were optimized. The pyridine and N-nitrosodimethylamine peaks are symmetric, even at this low level of detection. An excessively tailing pyridine peak, or a pyridine peak smaller than that for 1,4-dioxane at the same concentration, would indicate on-column activity. With an Rxi™-5ms column, and the conditions listed for Figure 3, pyridine can be detected reliably at low concentrations.

Analytically, 2,4-dintrophenol is considered the most problematic compound in the Method 8270 target list. 2,4-Dinitrophenol and the other system performance check compounds (SPCC) - N-nitroso-di-*n*-propylamine, hexachlorocyclopentadiene, and 4-nitrophenol - must exhibit a minimum average response factor (RF) of 0.050. An optimized system generally will provide response factors greater than 0.1 for these compounds, but the lower the calibration curve for these compounds, the more difficult it is to achieve passing response factors. If any of these compounds fails to meet the Method 8270 response factor criterion, system maintenance must be performed to bring response factors to passing before samples can be

Figure 1 An Rxi™-5ms column provides sharp, easily quantified peaks for active analytes (extracted ion chromatograms).

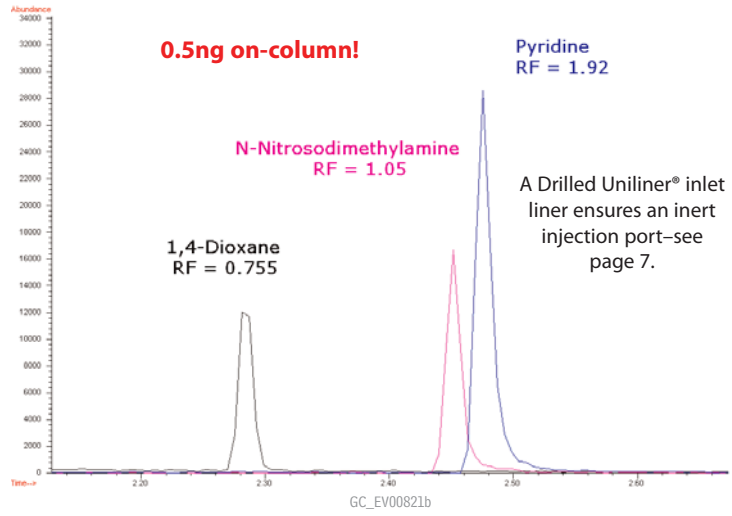


Figure 2 Excellent response for 0.5ng 2,4-dintrophenol on an Rxi™-5ms column.

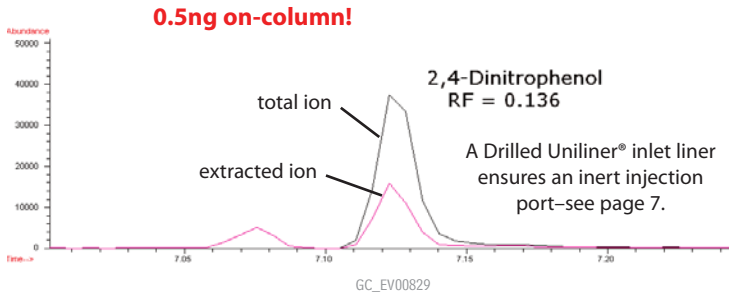
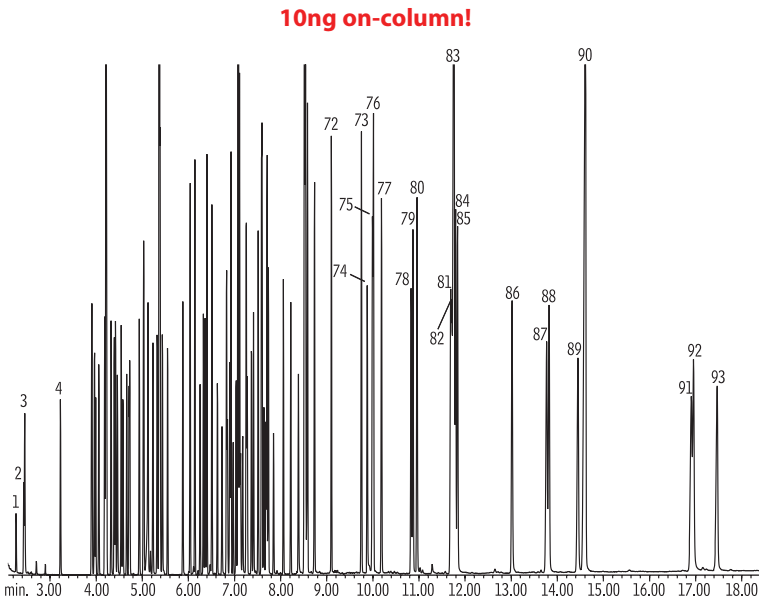


Figure 3 Separate 93 semivolatile compounds in less than 18 minutes, using an Rxi™-5ms column.



For complete identifications, please visit www.restek.com/rxi

GC_EV00823

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

Column: Rxi™-5ms, 30m, 0.25mm ID, 0.25µm (cat.# 13423)
Sample: US EPA Method 8270D mix: 8270 MegaMix™ (cat.# 31850), Benzoic Acid Standard (cat.# 31879), Benzidine Standard (cat.# 31852), Acid Surrogate Mix (cat.# 31025), B/N Surrogate Standard Mix (cat.# 31887), 1,4-Dioxane (cat.# 31853)
Inj.: 1.0µL, 10ppm each analyte (10ng on column), splitless (hold 0.1 min.)
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 0.5 min.) to 265°C @ 25°C/min., to 330°C @ 6°C/min. (hold 2 min.)
Det.: Agilent 5973 GC/MS
Transfer line temp.: 280°C
Scan range: 35-550 amu
Solvent delay: 2 min.
Tune: DFTPP
Ionization: EI

analyzed. Figure 2 shows the inertness of the Rxi™-5ms column, which exhibits a response factor of 0.136 for 0.5ng on-column of 2,4-dinitrophenol.

The total ion chromatogram for our optimized analysis is shown in Figure 3. There are at least five scans across each target analyte, which assures good spectral integrity and good peak shape, and the last compound is eluted in less than 18 minutes.

The Result

The Rxi™-5ms column introduces a new generation of Restek columns that exhibit low bleed, outstanding inertness, and excellent column-to-column reproducibility.

An Rxi™-5ms column, used in an optimized system, provides excellent chromatography for Method 8270 semivolatile compounds, including difficult-to-analyze acidic or basic compounds, at low on-column concentrations. These new columns give the performance needed, at the sensitivity required, column after column.

Rxi™-5ms Columns (fused silica)

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

ID	df (µm)	temp. limits	length	cat. #	price
0.18mm	0.18	-60 to 330/350°C	20-Meter	13402	$370
0.18mm	0.36	-60 to 330/350°C	20-Meter	13411	$370
0.20mm	0.33	-60 to 330/350°C	12-Meter	13497	$230
0.20mm	0.33	-60 to 330/350°C	25-Meter	13498	$365
0.20mm	0.33	-60 to 330/350°C	50-Meter	13499	$630
0.25mm	0.25	-60 to 330/350°C	15-Meter	13420	$260
0.25mm	0.25	-60 to 330/350°C	30-Meter	13423	$435
0.25mm	0.25	-60 to 330/350°C	60-Meter	13426	$780
0.25mm	0.50	-60 to 330/350°C	15-Meter	13435	$260
0.25mm	0.50	-60 to 330/350°C	30-Meter	13438	$435
0.25mm	0.50	-60 to 330/350°C	60-Meter	13441	$780
0.25mm	1.00	-60 to 330/350°C	15-Meter	13450	$260
0.25mm	1.00	-60 to 330/350°C	30-Meter	13453	$435
0.25mm	1.00	-60 to 330/350°C	60-Meter	13456	$780
0.32mm	0.25	-60 to 330/350°C	15-Meter	13421	$280
0.32mm	0.25	-60 to 330/350°C	30-Meter	13424	$460
0.32mm	0.25	-60 to 330/350°C	60-Meter	13427	$820
0.32mm	0.50	-60 to 330/350°C	15-Meter	13436	$280
0.32mm	0.50	-60 to 330/350°C	30-Meter	13439	$460
0.32mm	1.00	-60 to 330/350°C	15-Meter	13451	$280
0.32mm	1.00	-60 to 330/350°C	30-Meter	13454	$460
0.32mm	1.00	-60 to 330/350°C	60-Meter	13457	$820
0.53mm	0.25	-60 to 330/350°C	15-Meter	13422	$310
0.53mm	0.25	-60 to 330/350°C	30-Meter	13425	$515
0.53mm	0.50	-60 to 330/350°C	15-Meter	13437	$310
0.53mm	0.50	-60 to 330/350°C	30-Meter	13440	$515
0.53mm	1.00	-60 to 330/350°C	15-Meter	13452	$310
0.53mm	1.00	-60 to 330/350°C	30-Meter	13455	$515
0.53mm	1.50	-60 to 330/350°C	15-Meter	13467	$310
0.53mm	1.50	-60 to 330/350°C	30-Meter	13470	$515

tech tip

A Drilled Uniliner® inlet liner helps ensure reliable results for active compounds—see information on **page 7**.