

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.

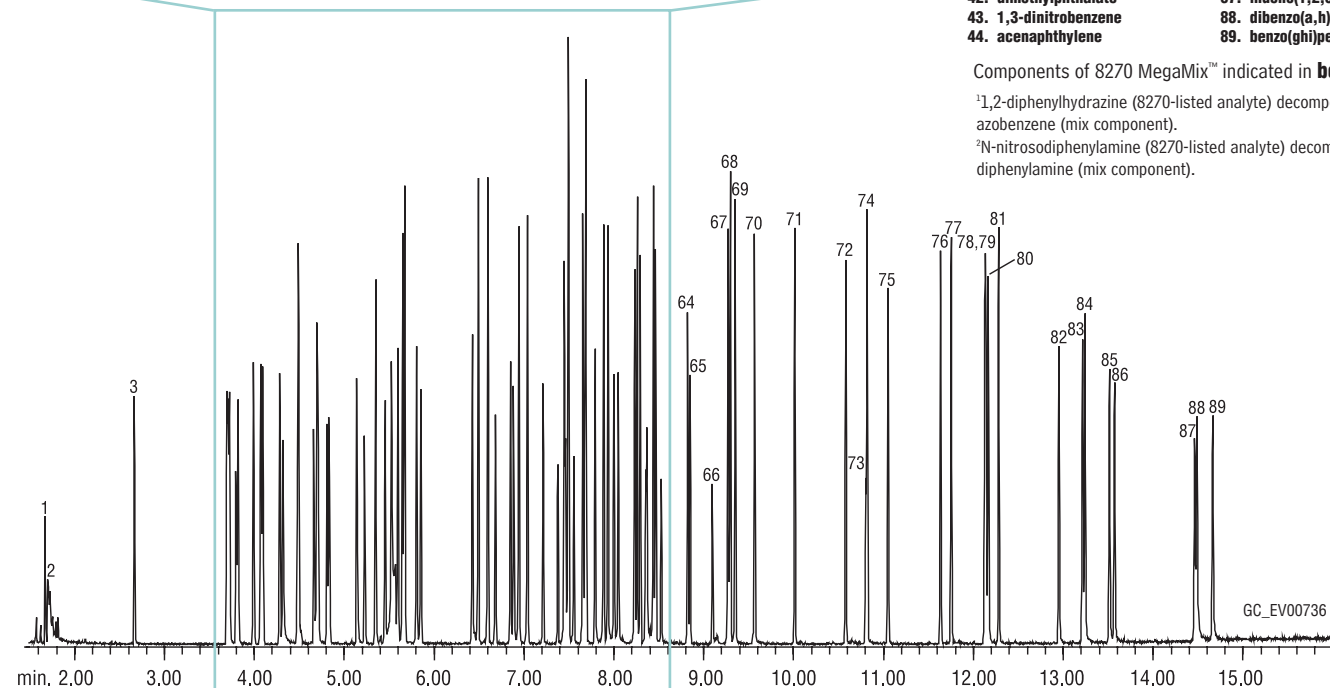
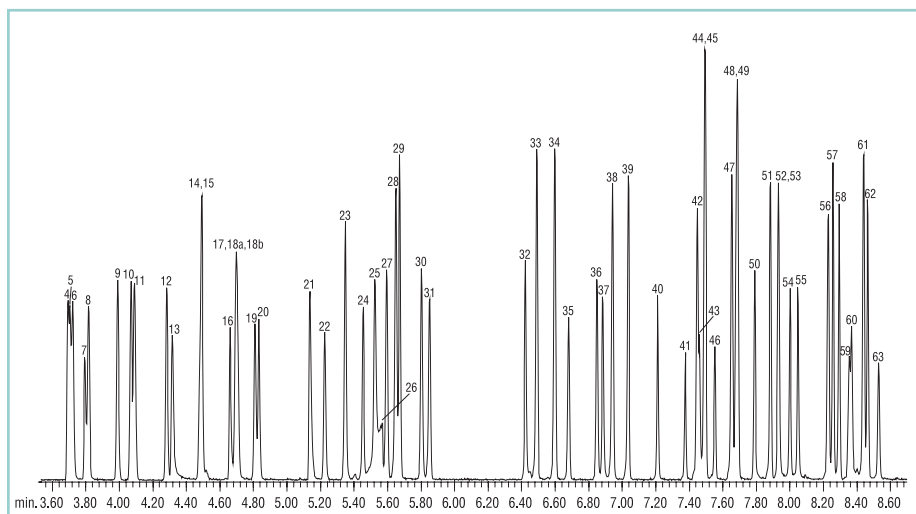
US EPA Method 8270D is one of the GC/MS methods followed to determine concentrations of semivolatile organic compounds in solid waste, soil, water, and air matrices. In a single analysis, environmental laboratories following Method 8270D typically evaluate 100 or more semivolatile organic compounds representing 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-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 (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. diphenylamine²**
- 62. azobenzene¹**
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**.

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

²N-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 semivolatiles 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 than 15 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 270°C 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 14°C/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).

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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)

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 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

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

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, form-

ing 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.