Applications of a New Volatile Mass Spectrometry/Gas Chromatography Column for US EPA Methods 8260, 524.2, 624, 8240, and OLM 04.1 (04.2).

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ABSTRACT

In the past, GC stationary phases were designed without consideration for their final application. This resulted in long analysis times, high bleed, and coelutions. Column choice becomes even more of an issue in the separation of volatile organic compounds (VOCs) because US EPA Method updates, such as Method 524.2, rev. IV, have added coeluting compounds having minor ions of one compound interfering with the quantitation of another compound (e.g., methyl acrylate/ propionitrile and 1,1-dichloro-2-propanone/4-methyl-2-pentanone). These compounds can be resolved on the typically-used "624/1301" phase, but with a longer column and longer analysis times.

With the use of computer stationary phase modeling, it is possible to design a column that achieves the fewest number of coelutions and the fastest analysis time for the separation of VOCs found in US EPA Methods 8260, 524.2, 624, 8240, and OLM O4.1 (O4.2). This paper will explain the limitations of current columns and the thermodynamic modeling used to develop a new stationary phase, taking into account the specific requirements of the MS system. Compound lists and phase requirements were determined using EPA methods and collaboration with environmental laboratories across the country. These columns will result in increased sample throughput via shorter analysis times and better resolution of critical compounds, with the aid of extracted ion detection. Five EPA Methods compound lists will be presented on several column dimensions, including "added" compounds commonly requested with these analysis.

INTRODUCTION

Volatile organic compounds (VOCs) are classified as having low solubility in water and high vapor pressure. Their boiling points range from -24°C for the light gases to 220°C for naphthalene and the trichlorobenzenes. When VOCs are present in water or a solid matrix, they readily partition themselves into the gaseous phase and therefore are often concentrated from the sample matrix using purge and trap. Having such a diverse range of pollutants places significant demands on the analytical column. The column must have a selective stationary phase to resolve the volatile pollutants, a sufficient film thickness to retain and resolve the low-boiling volatile compounds (e.g. purgeable gases and Freons®), and must be thermally stable to elute the high-boiling volatile compounds, such as hexachlorobutadiene.

Many capillary columns have been designed for the separation of VOCs by GC/MS. Traditionally, the "624" stationary phase was the column of choice because it provided the best resolution of the early eluting gases. However, this phase often is unable to resolve all compounds of interest that share quantitation ions without resulting in a sacrifice in in analysis time (<24 minutes).

The Rtx®-VMS column was designed to provide the fastest analysis times for volatile organics, such as those listed in US EPA Methods 8260, 524.2, 624, 8240, and OLM 04.2.

EXPERIMENTAL

The Rtx®-VMS phase was designed to provide excellent resolution of the gases (Applications #1, 2, 3, 4 & 5). Initial starting temperatures of up to 60°C are possible with this column (Application #1). This higher temperature provides the required separation and allows for a faster oven cycle time. The Rtx®-VMS phase was also designed to resolve all compounds by primary quntitation ions using extracted ion chromatography. This is important because US EPA method updates, such as Method 524.2, rev. IV, have added new compounds with minor ions that interfere with the quantitation ions of other target compounds. An example of this problem occurs when using the "624/1301" 75m x 0.53mm ID column for methyl acrylate and propionitrile. The quantitation ion for methyl acrylate is 55. Propionitrile has a minor ion of 55, which can interfere with determining actual concentrations of methyl acrylate in "real world" samples. Another difficult pair to resolve on the "624/1301" column is 1,1-dichloro-2-propanone and 4-methyl-2-pentanone, which share ion 43. These compounds can be resolved using the "624/1301" 60m x 0.32mm ID column in more than 30 minutes. The only difficult pair for the VMS phase to resolve in US EPA Method 524.2 rev. IV are 2-nitropropane and 1,1-dichloro-2-propanone which share ion 43 (Application # 2).

US EPA Method 8260 contains many mid-range volatile compounds, which are the most commonly found non-petroleum contaminants in the environment. Unfortunately, mid-range volatile compounds tend to exhibit broad peak shapes due to poor sample transfer from the purge and trap, making them difficult to resolve. The Rtx®-VMS column was designed to have better solubility of these analytes into the stationary phase, thereby providing the greatest degree of separation for these compounds. This tuned selectivity separates tetrahydrofuran/2-butanone, carbon tetrachloride/1,1,1-trichloroethane, and methyl acrylate/propionitrile. Although these compounds share common ions and have very similar spectra, they are resolved by retention time difference on the Rtx®-VMS column (Applications #1 & 3).

Higher boiling volatile compounds are made up of branched and substituted aromatic compounds, and possess their own set of analytical challenges. Isomers of the branched aromatic compounds share the same parent ions and cannot be identified accurately by MS alone. This new column was modeled for maximum separation of the substituted aromatic isomers, such as 2- and 4- chlorotoluene (Applications #1 and 2). This tuned selectivity allows a rapid final GC oven ramp rate of 40°C/min or faster, yielding faster analysis times.

Client target lists may remain the same as the compound list given for Method 8240, however, the calibration criteria and low detection limits set by Method 8260 are enforced (Application #3). The chromatograms showing the 8240 compound list are run under different GC oven conditions, different compound concentrations, and altered MS scan windows. The analysis of alcohols require scanning below 35amu because many of the fragments used to identify the spectra for

these compounds are found between 25 and 35amu. A good example is 2-chloroethanol; this target analyte purges poorly and does not respond well by MS detection. The best way to increase sensitivity with the detector is by changing the scan rate to include ion 31, the base peak. This increases the ability of the software and the user to better identify alcohols because it gives more spectral data. The disadvantage of this technique is an increase in noise, resulting in an overall decrease in sensitivity for all compounds. In Application #3 the second chromatogram shows the increase in baseline noise as a result of the lowered scan window. A direct comparison of the two chromatograms for peak 38 (2-chloroethanol) clearly shows a significant increase in response despite a lower concentration.

EPA Method 624 is generally analyzed using capillary chromatographic techniques (Application #4). The compound list includes 35 commonly analyzed aromatic and halogenated compounds in waste water. This analysis can be done on many different capillary column dimensions. The Rtx®-VMS 30m x 0.25mm ID column shows baseline gas separation (Application #4).

The US EPA has recently awarded contracts for organic low medium (OLM) concentration samples within the Superfund program under the 04.2 revision Statement of Work. These compounds are well resolved even on a large-bore column. Faster runtimes are possible using narrowbore VMS columns (Application #5).

CONCLUSION

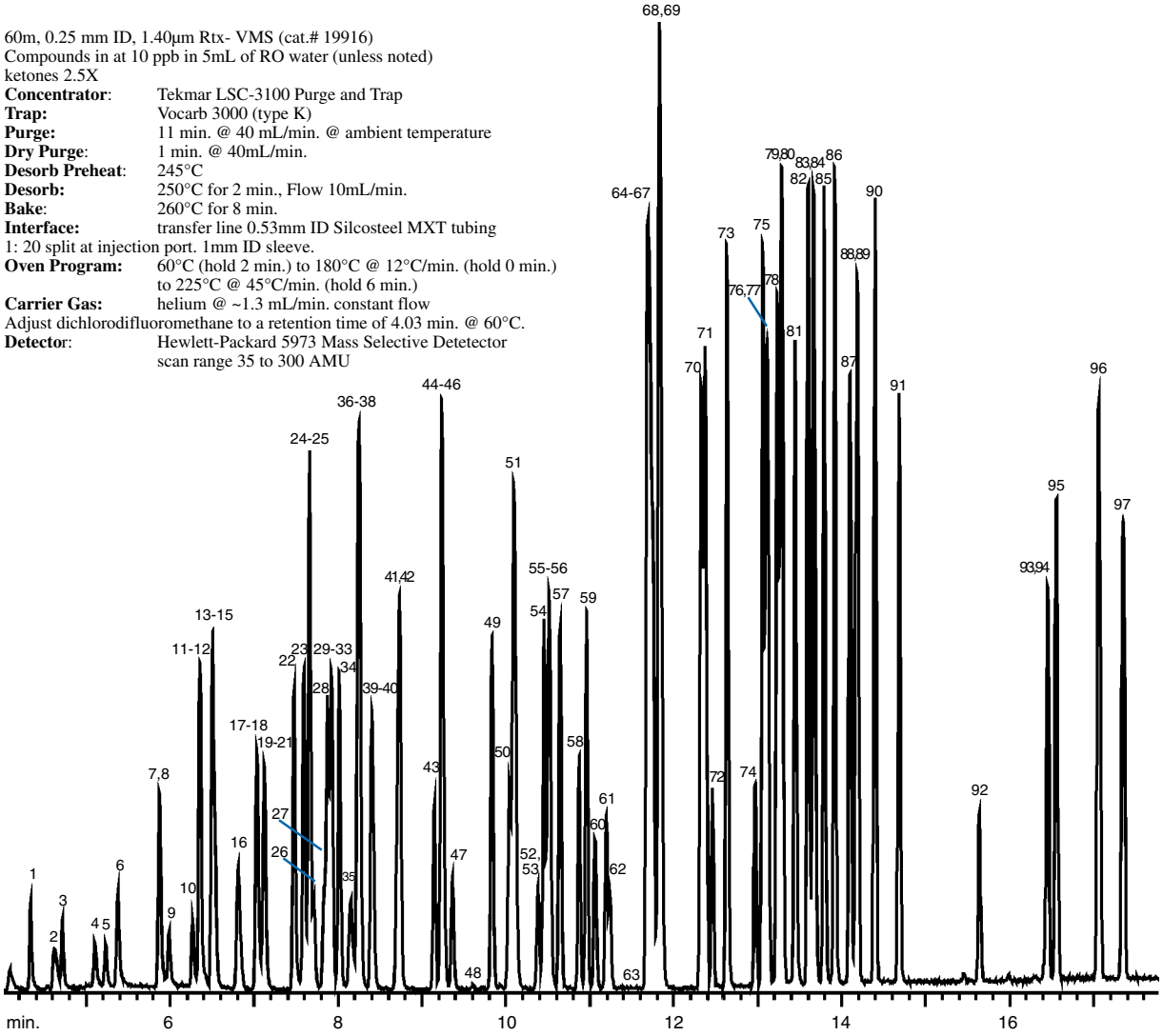
This new column shows excellent selectivity for EPA volatile purge and trap methods with an overall faster runtime than traditional phases currently on the market. Decreasing oven cycle time is the most important factor to increasing productivity. The Rtx®-VMS column can start at temperatures of 60°C (Application #1), thereby allowing a shorter total cycle time without a significant sacrifice in gas resolution.

Application #1

Volatile Organics EPA Method 8260B Rtx®-VMS

60m, 0.25 mm ID, 1.40µm Rtx- VMS (cat.# 19916)
Compounds in at 10 ppb in 5mL of RO water (unless noted)
ketones 2.5X

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.
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
1: 20 split at injection port. 1mm ID sleeve.
Oven Program: 60°C (hold 2 min.) to 180°C @ 12°C/min. (hold 0 min.) to 225°C @ 45°C/min. (hold 6 min.)
Carrier Gas: helium @ ~1.3 mL/min. constant flow
Adjust dichlorodifluoromethane to a retention time of 4.03 min. @ 60°C.
Detector: Hewlett-Packard 5973 Mass Selective Detetector scan range 35 to 300 AMU

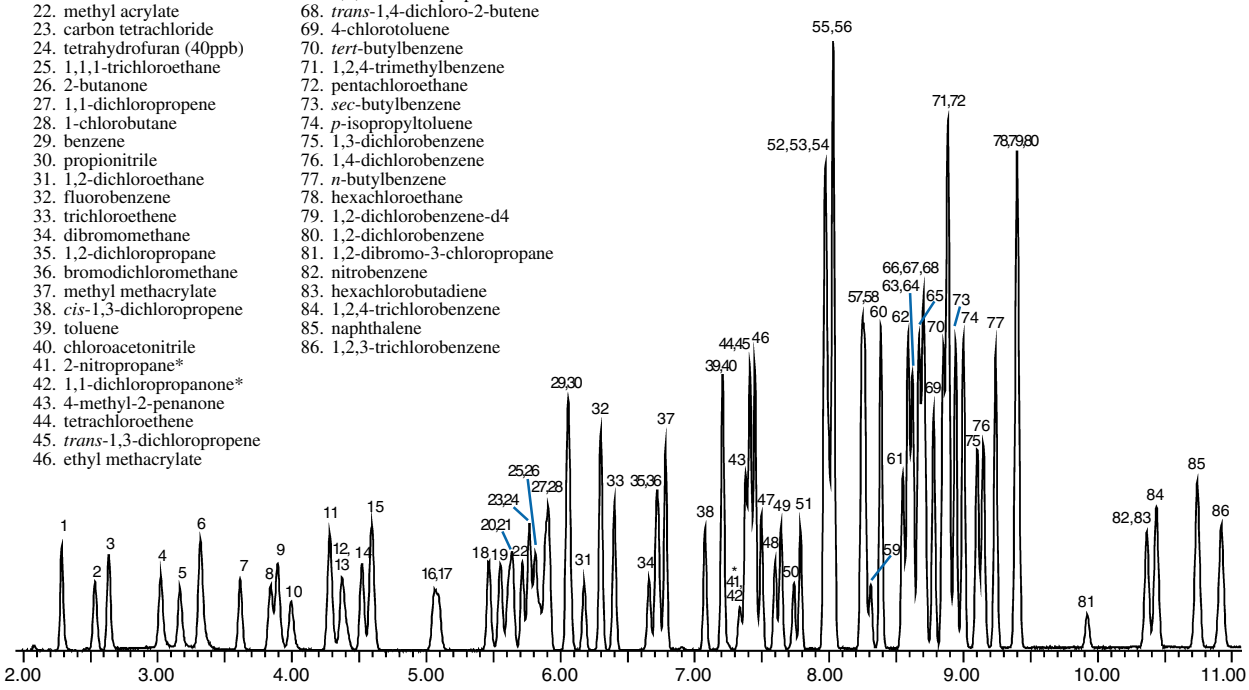


1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. ethanol (2500ppb)
8. 1,1-dichloroethene
9. carbon disulfide (40ppb)
10. allyl chloride
11. methylene chloride
12. acetone
13. *trans*-1,2-dichloroethene
14. *tert*-butyl alcohol (100ppb)
15. methyl *tert*-butyl ether
16. diisopropyl ether
17. 1,1-dichloroethane
18. acrylonitrile
19. vinyl acetate
20. allyl alcohol (250ppb)
21. ethyl-*tert*-buyl ether
22. *cis*-1,2-dichloroethene
23. 2,2-dichloropropane
24. bromochloromethane
25. chloroform
26. ethyl acetate
27. methyl acrylate
28. propargyl alcohol (500ppb)
29. dibromofluoromethane (SMC)
30. tetrahydrofuran
31. carbon tetrachloride
32. 2-butanone
33. 1,1,1-trichloroethane
34. 1,1-dichloropropene
35. pentafluorobenzene(IS)
36. *tert*-amyl-methyl ether
37. benzene
38. isobutyl alcohol (500ppb)
39. 1,2-dichloroethane
40. isopropyl acetate
41. 1,4-difluorobenzene(SMC)
42. trichloroethene
43. dibromomethane
44. bromodichloromethane
45. 1,2-dichloropropane
46. methyl methacrylate
47. *n*-propyl acetate
48. 2-chloroethanol (2500ppb)
49. *cis*-1,3-dichloropropene
50. toluene-d8(SMC)
51. toluene
52. 4-methyl-2-penanone
53. pyridine (250ppb)
54. *trans*-1,3-dichloropropene
55. ethyl methacrylate
56. tetrachloroethene
57. 1,1,2-trichloroethane
58. dibromochloromethane
59. 1,3-dichloropropane
60. *n*-butyl acetate
61. 1,2-dibromoethane
62. 2-hexanone
63. 2-picoline (250ppb)
64. ethylbenzene
65. chlorobenzene-D5(IS)
66. chlorobenzene
67. 1,1,1,2-tetrachloroethane
68. *m*-xylene
69. *p*-xylene
70. *o*-xylene
71. stryrene
72. bromoform
73. isopropylbenzene
74. 4-bromo-1-fluorobenzene (SMC)
75. *n*-propylbenzene
76. 1,1,2,2-tetrachloroethane
77. bromobenzene
78. 1,3,5-trimethylbenzene
79. 2-chlorotoluene
80. 1,2,3-trichloropropane
81. 4-chlorotoluene
82. *tert*-butylbenzene
83. 1,2,4-trimethylbenzene
84. pentachloroethane
85. *sec*-butylbenzene
86. *p*-isopropyltoluene
87. 1,3-dichlorobenzene
88. 1,4-dichlorobenzene-d4(IS)
89. 1,4-dichlorobenzene
90. *n*-butylbenzene
91. 1,2-dichlorobenzene
92. 1,2-dibromo-3-chloropropane
93. nitrobenzene (250ppb)
94. hexachlorobutadiene
95. 1,2,3-trichlorobenzene
96. naphthalene
97. 1,2,4-trichlorobenzene

Application #2

EPA Method 524.2, Revision 4 Rtx®-VMS

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. diethyl ether
8. 1,1-dichloroethene
9. carbon disulfide (40ppb)
10. iodomethane (40ppb)
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 (40ppb)
25. 1,1,1-trichloroethane
26. 2-butanone
27. 1,1-dichloropropene
28. 1-chlorobutane
29. benzene
30. propionitrile
31. 1,2-dichloroethane
32. fluorobenzene
33. trichloroethene
34. dibromomethane
35. 1,2-dichloropropane
36. bromodichloromethane
37. methyl methacrylate
38. *cis*-1,3-dichloropropene
39. toluene
40. chloroacetonitrile
41. 2-nitropropane*
42. 1,1-dichloropropanone*
43. 4-methyl-2-penanone
44. tetrachloroethene
45. *trans*-1,3-dichloropropene
46. ethyl methacrylate
47. 1,1,2-trichloroethane
48. dibromochloromethane
49. 1,3-dichloropropane
50. 1,2-dibromoethane
51. 2-hexanone
52. ethylbenzene
53. chlorobenzene
54. 1,1,1,2-tetrachloroethane
55. *m*-xylene
56. *p*-xylene
57. *o*-xylene
58. stryrene
59. bromoform
60. isopropylbenzene
61. 4-bromofluorobenzene
62. *n*-propylbenzene
63. bromobenzene
64. 1,1,2,2-tetrachloroethane
65. 1,3,5-trimethylbenzene
66. 2-chlorotoluene
67. 1,2,3-trichloropropane
68. *trans*-1,4-dichloro-2-butene
69. 4-chlorotoluene
70. *tert*-butylbenzene
71. 1,2,4-trimethylbenzene
72. pentachloroethane
73. *sec*-butylbenzene
74. *p*-isopropyltoluene
75. 1,3-dichlorobenzene
76. 1,4-dichlorobenzene
77. *n*-butylbenzene
78. hexachloroethane
79. 1,2-dichlorobenzene-d4
80. 1,2-dichlorobenzene
81. 1,2-dibromo-3-chloropropane
82. nitrobenzene
83. hexachlorobutadiene
84. 1,2,4-trichlorobenzene
85. naphthalene
86. 1,2,3-trichlorobenzene



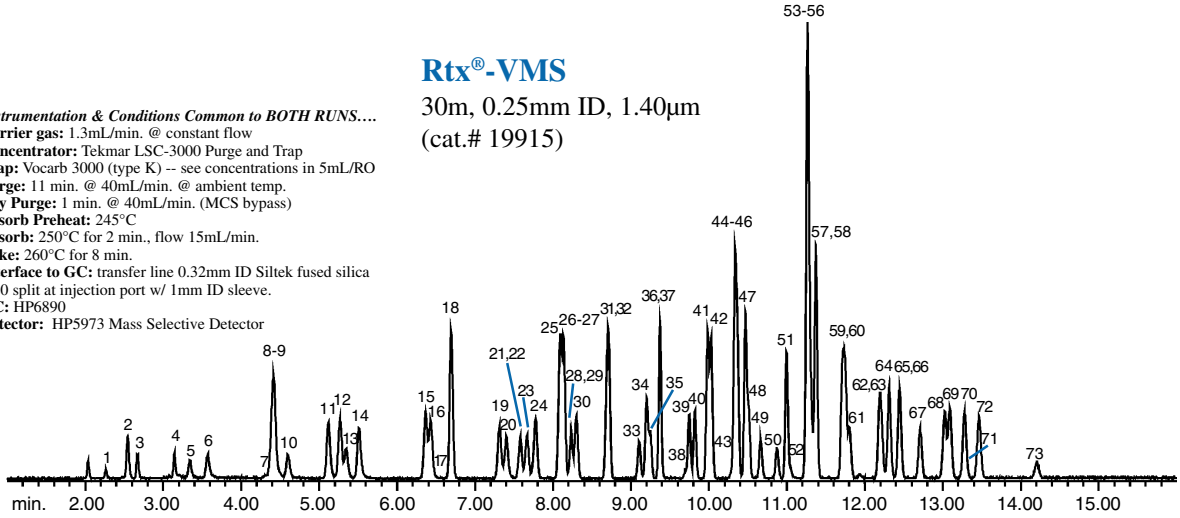
30m, 0.25mm ID, 1.4µm Rtx®-VMS (cat.# 19915)
Carrier gas: helium @ ~1.3mL/min. constant flow
Adjust dichlorodifluoromethane to a retention time of 2.29 min. @ 45°C
Concentrator: Tekmar LSC-3000 Purge and Trap
Oven program: 45°C (hold 2 min.) to 85°C @ 14°C/min.
to 210°C @ 40°C/min. (hold 4 min.)
GC: HP6890 Series II
Trap: Vocarb 3000
Purge: 11 min. @ 40mL/min.
Dry purge: 1 min. @ 40mL/min. (MCS bypassed)
Desorb preheat: 245°C
Desorb: 250°C for 2 min.
Bake: 260°C for 8 min.
Interface: 1:10 split in port
Transfer line: 5m, 0.32mm ID Siltek™ tubing (cat.# 10027)
Detector: Hewlett-Packard 5973 Mass
Selective Detetector scan range 35 to 300 AMU

Standards:
20ppb in 5mL of RO water (unless otherwise noted); ketones in 40ppb.
502.2 Cal Mix #1 (cat.# 30042)
502.2 Cal2000 MegaMix™ (cat.# 30431)
524 Cal Mix 7A & 7B (cat.# 30202)
524 Cal Mix #8 (cat.# 30203)
524 IS/SS Mix (cat.# 30201)

**These peaks (41 and 42) share a quantitation ion (43)*

Application #3

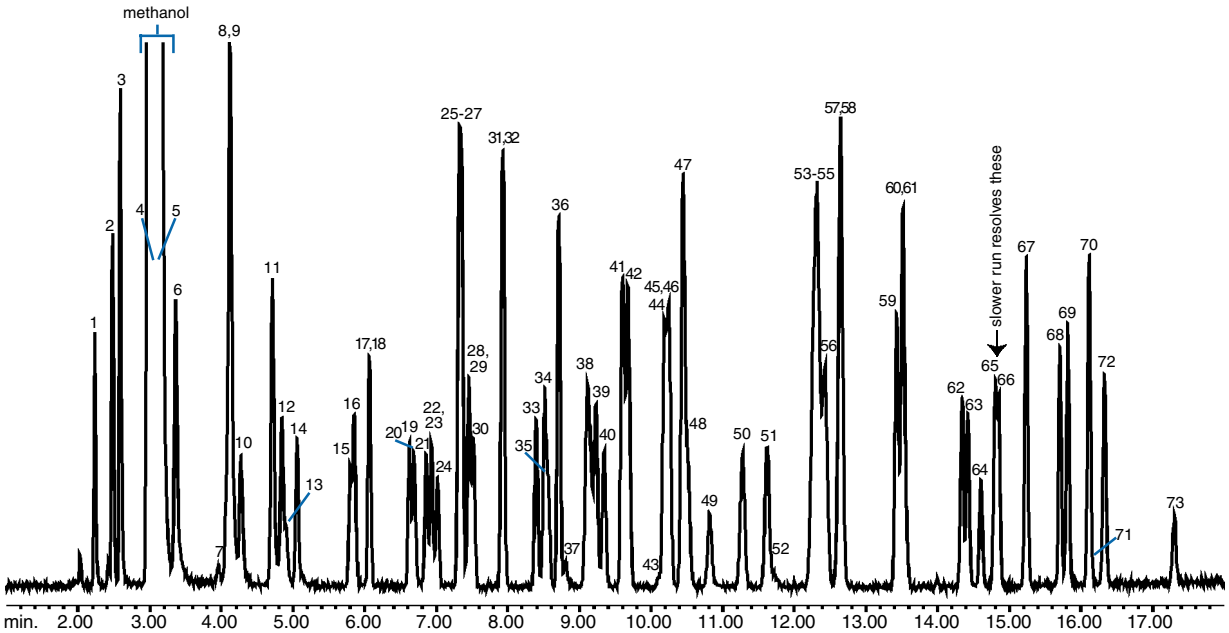
Volatile Organics EPA Method 8240 (8260 Short List) Rtx®-VMS



Instrumentation & Conditions Common to BOTH RUNS....
Carrier gas: 1.3mL/min. @ constant flow
Concentrator: Tekmar LSC-3000 Purge and Trap
Trap: Vocarb 3000 (type K) -- see concentrations in 5mL/RO
Purge: 11 min. @ 40mL/min. @ ambient temp.
Dry Purge: 1 min. @ 40mL/min. (MCS bypass)
Desorb Preheat: 245°C
Desorb: 250°C for 2 min., flow 15mL/min.
Bake: 260°C for 8 min.
Interface to GC: transfer line 0.32mm ID Siltek fused silica 1:20 split at injection port w/ 1mm ID sleeve.
GC: HP6890
Detector: HP5973 Mass Selective Detector

Top chromatogram:
Oven Conditions: 40°C (hold 4 min.) to 90°C at 16°C/min. (no hold) to 210°C at 32°C/min. (hold 5 min.)
Adjust dichlorodifluoromethane to a retention time of 2.27 min. @ 40°C.
MS Scan Range: 35-300amu
compound concentrations, by mix: (in 5mL of RO water)
Compounds in at 100ppb (cat.# 30213, 30004, 30006, 30011, 30042)
Alcohols in at 1ppm (cat.# 30214) except 2Cl ethanol at 10ppm.
vinyl acetate in at 500ppb (cat.#30216)
8240 nitrile mix at 200 ppb (cat.# 30215)
8240 mix 1A at 300 ppb (cat.# 30217)
8240 Mix 2A at 500 ppb (cat.# 30218)

Bottom chromatogram:
Oven Conditions: 45°C (hold 4 min.) to 110°C at 19°C/min. (hold 5 min.) to 220°C at 32°C/min. (hold 5 min.)
Adjust dichlorodifluoromethane to 2.23 min. @ 45°C.
MS Scan Range: 29-260amu, for 2Cl ethanol response
compound concentrations, by mix: (in 5mL of RO water)
Compounds in at 100ppb (cat.# 30213, 30004, 30006, 30011, 30042)
Alcohols in at 1ppm (cat.# 30214) (see MS scan)
vinyl acetate in at 100ppb (cat.# 30216)
8240 nitrile mix at 400 ppb (cat.# 30215)
8240 mix 1A at 300 ppb (cat.# 30217)
8240 Mix 2A at 500ppb (cat.# 30218)

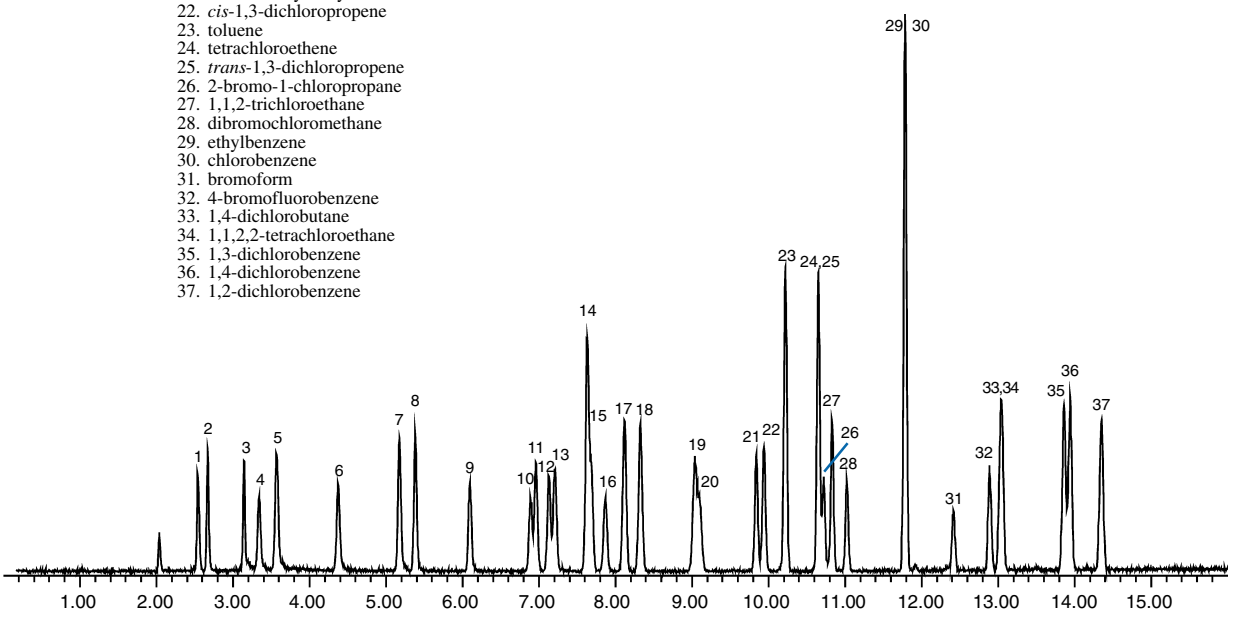


1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. ethanol
8. 1,1-dichloroethene
9. carbon disulfide
10. iodomethane
11. allyl chloride
12. methylene chloride
13. acetone
14. *trans*-1,2-dichloroethene
15. 1,1-dichloroethane
16. acrylonitrile
17. allyl alcohol
18. vinyl acetate
19. bromochloromethane
20. chloroform
21. carbon tetrachloride
22. propargyl alcohol
23. 1,1,1-trichloroethane
24. 2-butanone
25. benzene
26. propionitrile
27. methacrylonitrile
28. 1,2-dichloroethane-d4
29. isobutyl alcohol
30. 1,2-dichloroethane
31. trichloroethene
32. 1,4-difluorobenzene
33. dibromomethane
34. 1,2-dichloropropane
35. bromodichloromethane
36. methyl methacrylate
37. 1,4-dioxane
38. 2-chloroethanol
39. 2-chloroethyl vinyl ether
40. *cis*-1,3-dichloropropene
41. toluene-d8
42. toluene
43. pyridine
44. 4-methyl-2-pentanone
45. tetrachloroethene
46. *trans*-1,3-dichloropropene
47. ethyl methacrylate
48. 1,1,2-trichloroethane
49. dibromochloromethane
50. 1,2-dibromoethane
51. 2-hexanone
52. 2-picoline
53. chlorobenzene-D5
54. ethylbenzene
55. chlorobenzene
56. 1,1,1,2-tetrachloroethane
57. *m*-xylene
58. *p*-xylene
59. *o*-xylene
60. stryrene
61. bromoform
62. 4-bromo-1-fluorobenzene
63. *cis*-1,4-dichloro-2-butene
64. 1,1,2,2-tetrachloroethane
65. 1,2,3-trichloropropane
66. *trans*-1,4-dichloro-2-butene
67. pentachloroethane
68. 1,3-dichlorobenzene
69. 1,4-dichlorobenzene
70. benzyl chloride
71. malononitrile
72. 1,2-dichlorobenzene
73. 1,2-dibromo-3-chloropropane

Application #4

EPA Method 624 Rtx®-VMS

1. chloromethane
2. vinyl chloride
3. bromomethane
4. chloroethane
5. trichlorofluoromethane
6. 1,1-dichloroethene
7. methylene chloride
8. *trans*-1,2-dichloroethene
9. 1,1-dichloroethane
10. bromochloromethane
11. chloroform
12. carbon tetrachloride
13. 1,1,1-trichloroethane
14. benzene
15. pentafluorobenzene
16. 1,2-dichloroethane
17. fluorobenzene
18. trichloroethene
19. 1,2-dichloropropane
20. bromodichloromethane
21. 2-chloroethyl vinyl ether
22. *cis*-1,3-dichloropropene
23. toluene
24. tetrachloroethene
25. *trans*-1,3-dichloropropene
26. 2-bromo-1-chloropropane
27. 1,1,2-trichloroethane
28. dibromochloromethane
29. ethylbenzene
30. chlorobenzene
31. bromoform
32. 4-bromofluorobenzene
33. 1,4-dichlorobutane
34. 1,1,2,2-tetrachloroethane
35. 1,3-dichlorobenzene
36. 1,4-dichlorobenzene
37. 1,2-dichlorobenzene



30 m, 0.25mm ID, 1.40µm Rtx-VMS (cat#19915)

Concentration of Analytes:	20 ppb in 5ml of RO water
Concentrator:	Tekmar LSC-3000 Purge and Trap
Trap:	Vocarb 3000 (type K)
Purge:	11 min. @ 40mL/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:	transfer line 0.32mm ID Siltek fused silica 1: 10 split at injection port. 1mm ID sleeve.
GC:	HP6890
Oven Program:	40°C (hold 4 min.) to 95°C @ 24°C/min. (hold 3 min.), to 210°C @ 40°C/min. (hold 6 min.)
Carrier Gas:	helium @ ~1 mL/min. constant flow

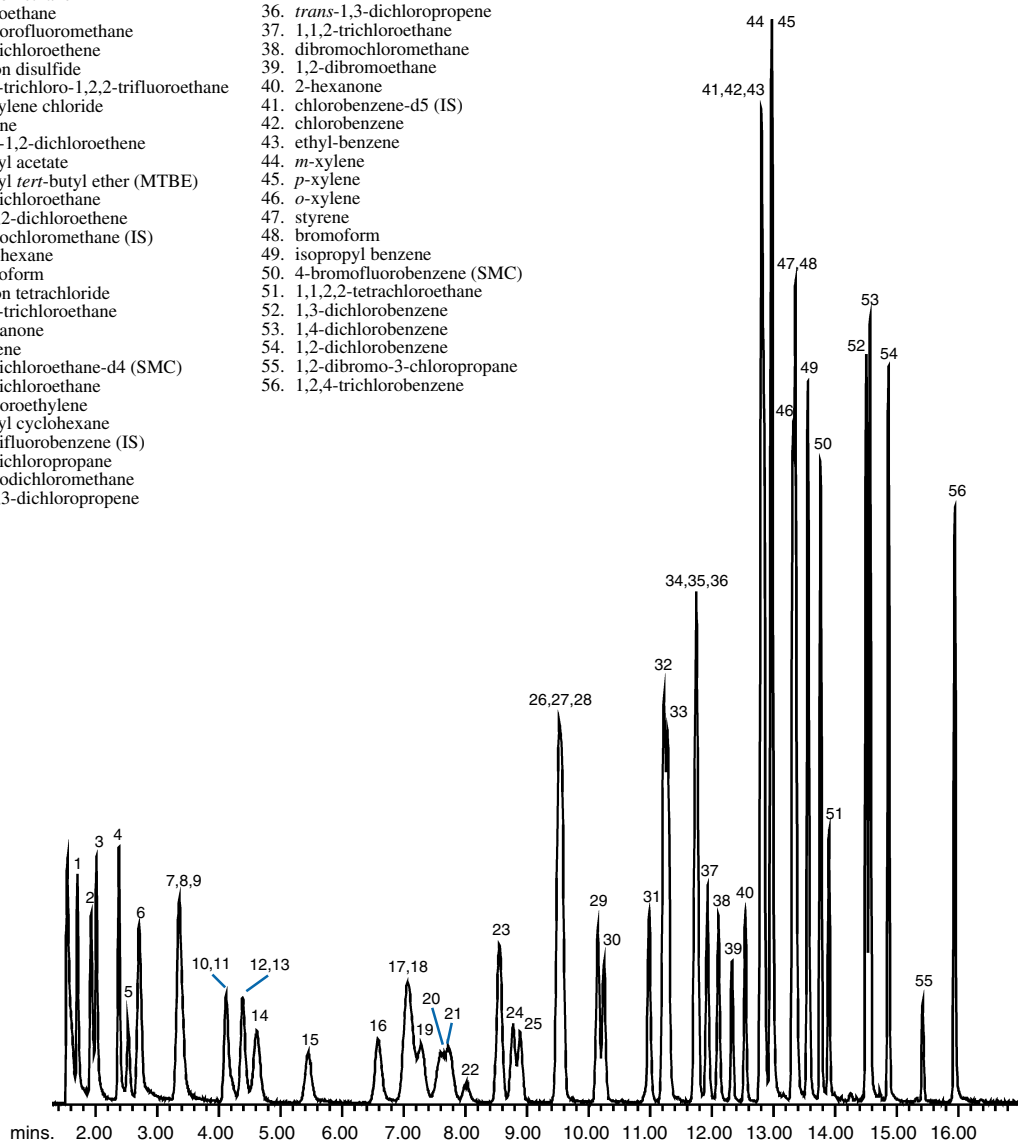
Adjust dichlorodifluoromethane to a retention time of 2.54 min. @ 40°C

Detector:	Hewlett-Packard 5973 Mass Selective Detetector scan range 25 to 300 AMU

Application #5

Volatile Organics OLM 04.1(04.2) Rtx®-VMS

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. carbon disulfide
9. 1,1,2-trichloro-1,2,2-trifluoroethane
10. methylene chloride
11. acetone
12. *trans*-1,2-dichloroethene
13. methyl acetate
14. methyl *tert*-butyl ether (MTBE)
15. 1,1-dichloroethane
16. *cis*-1,2-dichloroethene
17. bromochloromethane (IS)
18. cyclohexane
19. chloroform
20. carbon tetrachloride
21. 1,1,1-trichloroethane
22. 2-butanone
23. benzene
24. 1,2-dichloroethane-d4 (SMC)
25. 1,2-dichloroethane
26. trichloroethylene
27. methyl cyclohexane
28. 1,4-difluorobenzene (IS)
29. 1,2-dichloropropane
30. bromodichloromethane
31. *cis*-1,3-dichloropropene
32. toluene-d8 (SMC)
33. toluene
34. 4-methyl-2-pentanone
35. tetrachloroethene
36. *trans*-1,3-dichloropropene
37. 1,1,2-trichloroethane
38. dibromochloromethane
39. 1,2-dibromoethane
40. 2-hexanone
41. chlorobenzene-d5 (IS)
42. chlorobenzene
43. ethyl-benzene
44. *m*-xylene
45. *p*-xylene
46. *o*-xylene
47. styrene
48. bromoform
49. isopropyl benzene
50. 4-bromofluorobenzene (SMC)
51. 1,1,2,2-tetrachloroethane
52. 1,3-dichlorobenzene
53. 1,4-dichlorobenzene
54. 1,2-dichlorobenzene
55. 1,2-dibromo-3-chloropropane
56. 1,2,4-trichlorobenzene



EPA Method OLM 04.1 SOW
60m, 0.45mm ID, 2.55µm Rtx-VMS (cat.# 19909)

100ppb in 25mL of RO Water (ketones in @ 250ppb);
Concentrator: Tekmar LSC-3000 Purge & Trap;
Trap: Vocarb 3000;
Purge: 11 min. @ 40mL/min.;
Dry Purge: 1 min. @ 40mL/min. (MCS OFF)
Desorb Preheat: 245°C;
Desorb Flow: 10mL/min.;
Desorb: 250°C for 2 min.;
Bake: 260°C for 8 min.;

GC Interface: direct, using 0.32mm ID Siltek transfer line;
GC: HP 5890 Series II;
GC Program: 40°C (hold 7 min.) to 50°C @ 9°C/min. (hold 0 min.) to 110°C @ 27°C/min. (hold 1 min.) to 225°C @ 40°C/min. (hold 5 min.);
Carrier: Helium;
Adjust dichlorodifluoromethane to a retention time of 1.72 min. @ 40°C;
Flow Rate: 10mL/min. constant pressure to OSI (EZ-Vent 3000), 1mL/min. to source, 10:1 split;
Detector: HP 5971A MSD;
Scan Range: 35-300 AMU.