Analysis of Volatile Organic Compounds by Purge and Trap using GC/MS and a unique Stationary Phase

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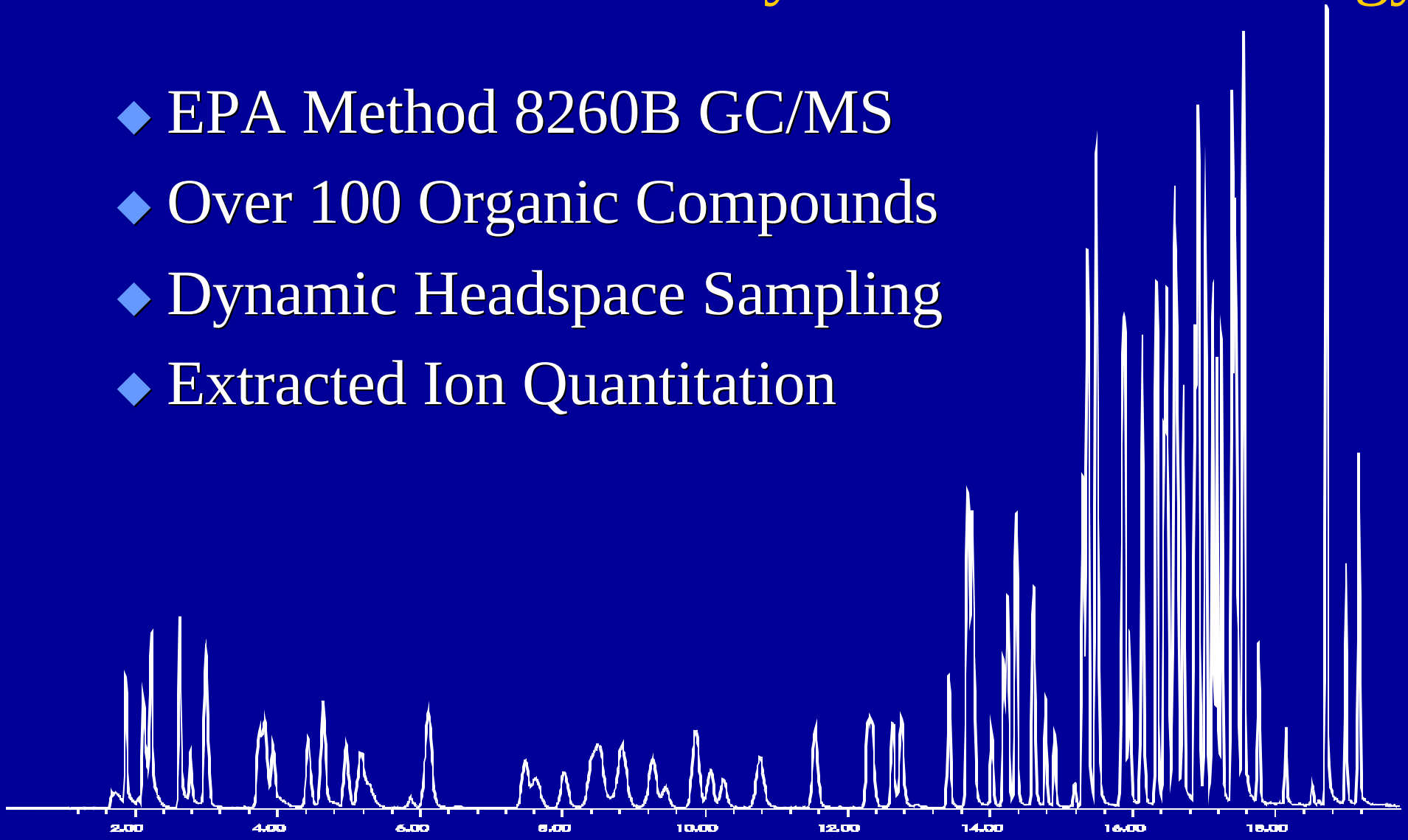
RESTEK
Turning Visions into Reality

GC/MS Volatiles Analysis EPA Methodology

- EPA Method 8260B GC/MS
- Over 100 Organic Compounds
- Dynamic Headspace Sampling
- Extracted Ion Quantitation



Dynamic Headspace Sampling – Purge & Trap

- Purge & Trap increases sensitivity compared to other introduction methods
- However, purging analytes from the sample to the trap, then desorbing the trap, can result in broad peaks for the most volatile compounds.
- Column design needs to take into account the early eluting peak shapes and isomeric pairs (ex. Chlorotoluenes, dichlorobenzenes).

Column Design Criteria

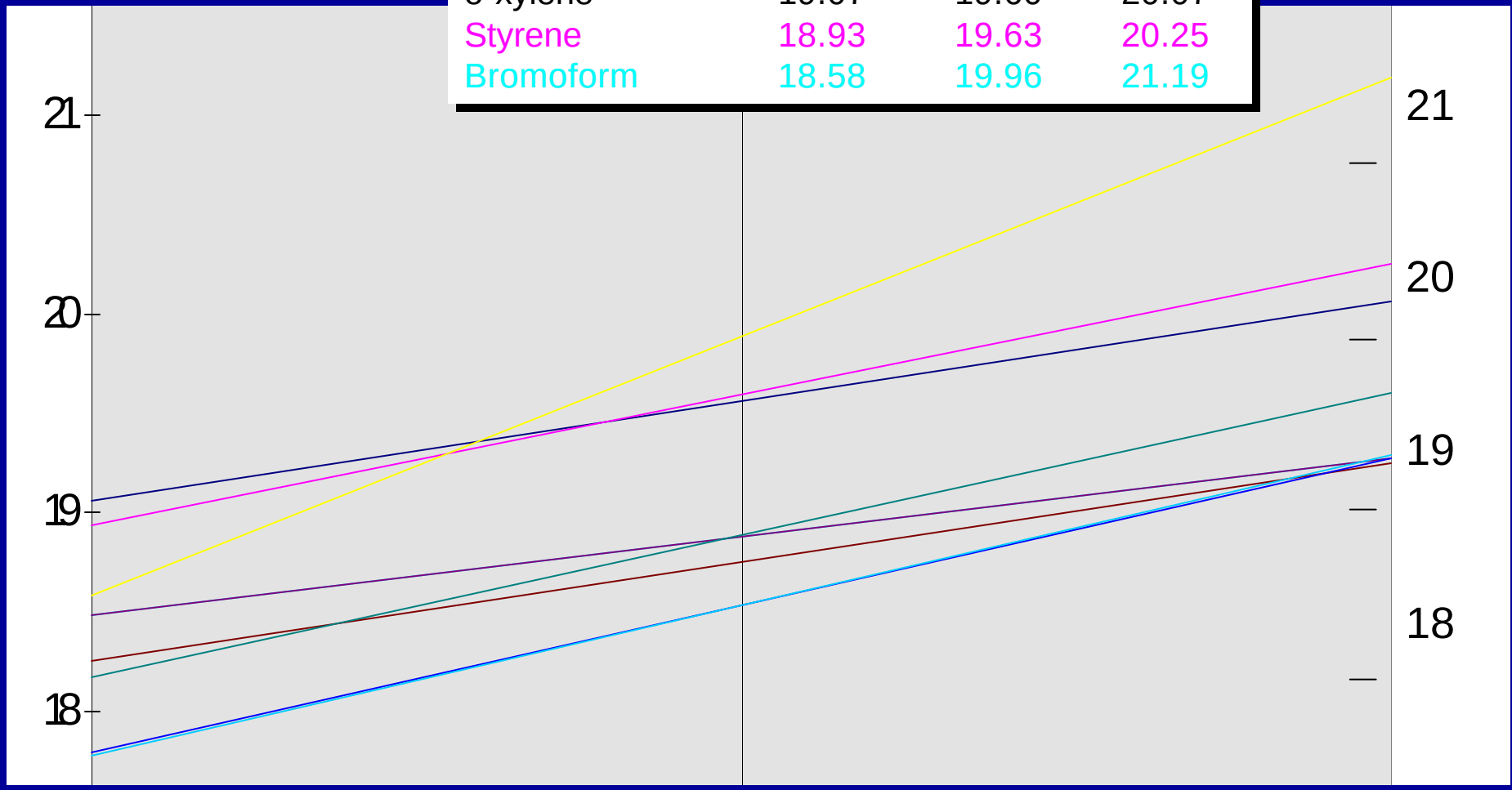
- Analysis Time
- Low Bleed
- Critical Resolution
- No Cryofocusing
- GC/MS Compatible
- Computer Aided Stationary Phase Design (CASPD) can help achieve these criteria

Computer Aided Stationary Phase Design

- Phase modeling involves comparing analyte retention times collected on different stationary phases.
- This data can be used to predict the optimum resolution of compounds with different phase combinations.
- For example, Rtx-1 and Rtx-35 can be modeled to predict a composition for the Rtx-502.2 stationary phase.

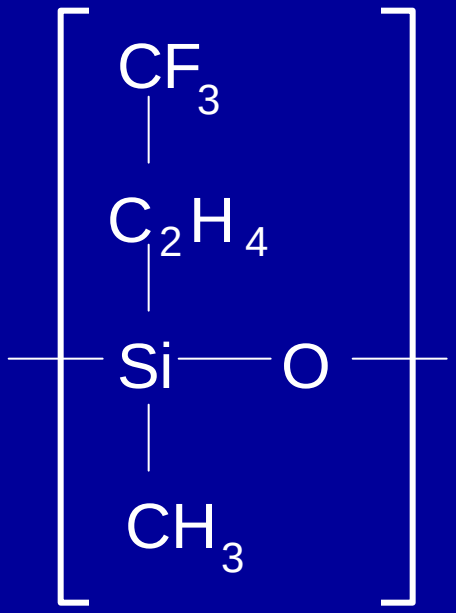
Modeling

	Rtx-1	Rtx-502	Rtx-35
Chlorobenzene	17.79	18.57	19.27
1112te ethane	17.78	18.58	19.29
E benzene	18.26	18.78	19.25
m/p-xylene	18.48	18.90	19.27
o-ClF benzene	18.16	18.93	19.61
o-xylene	19.07	19.60	20.07
Styrene	18.93	19.63	20.25
Bromoform	18.58	19.96	21.19

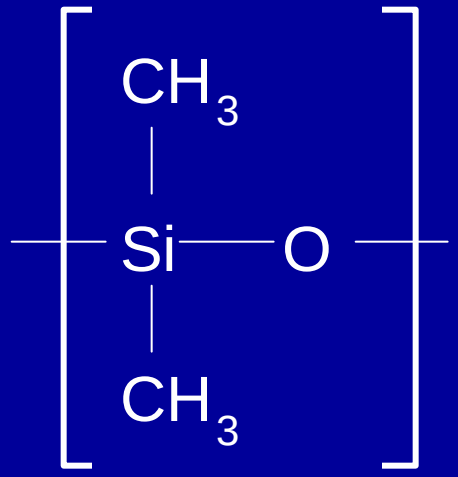


Stationary Phases Used for Modeling

trifluoropropylmethyl polysiloxane

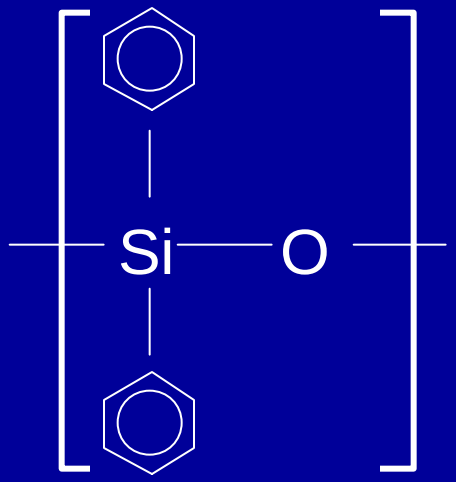


dimethyl polysiloxane

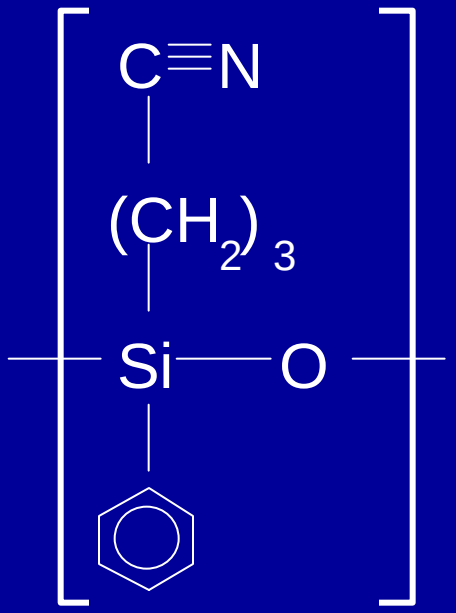


Stationary Phases Used for Modeling

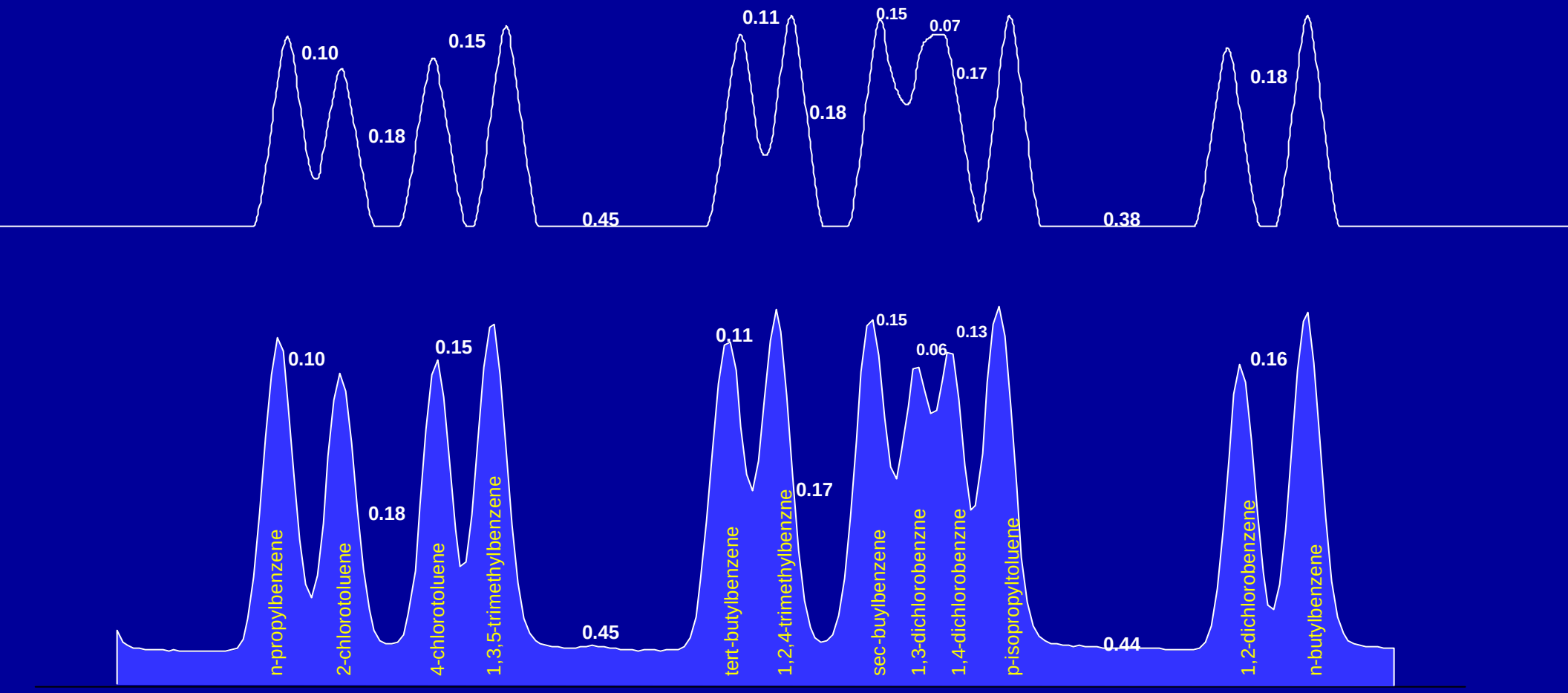
diphenyl
polysiloxane



cyanopropylphenyl
polysiloxane



Predicted vs. Actual Using a 4 Dimensional Phase

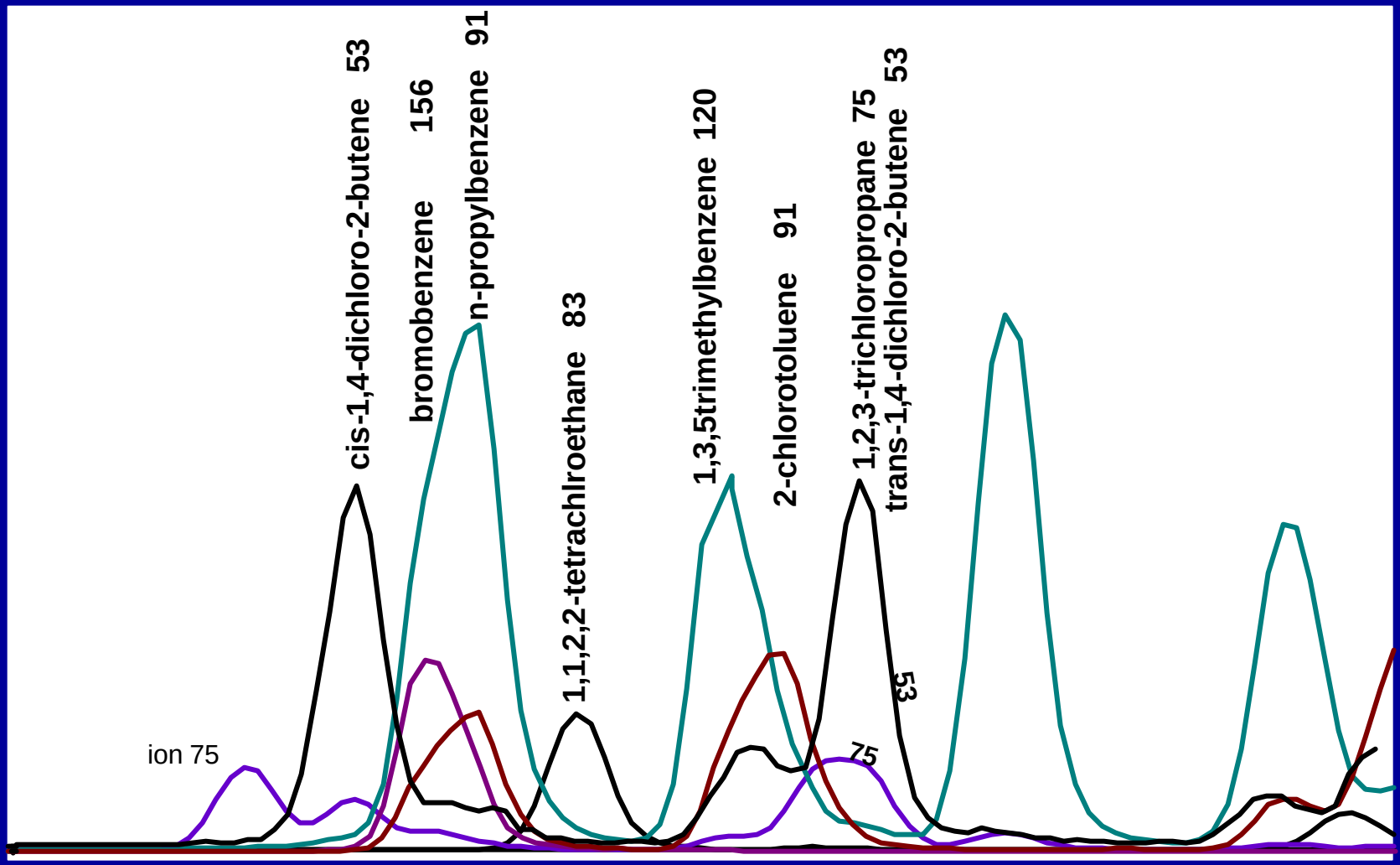


Rtx-VMS

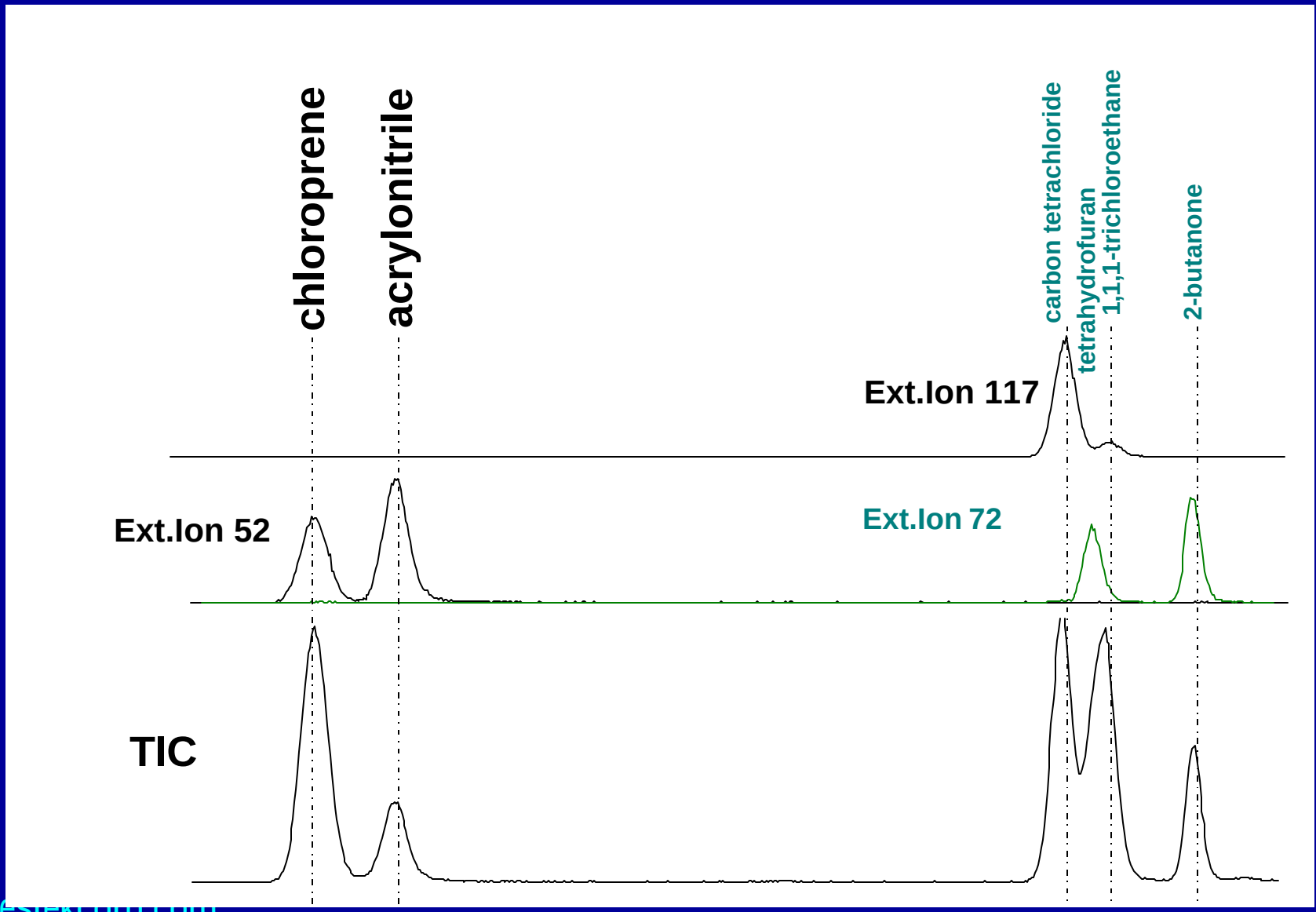
- Stationary Phase created using CASPD and optimized for GC/MS.

- Resolution and bleed levels were evaluated across a range of column dimensions and run conditions for maximum versatility.

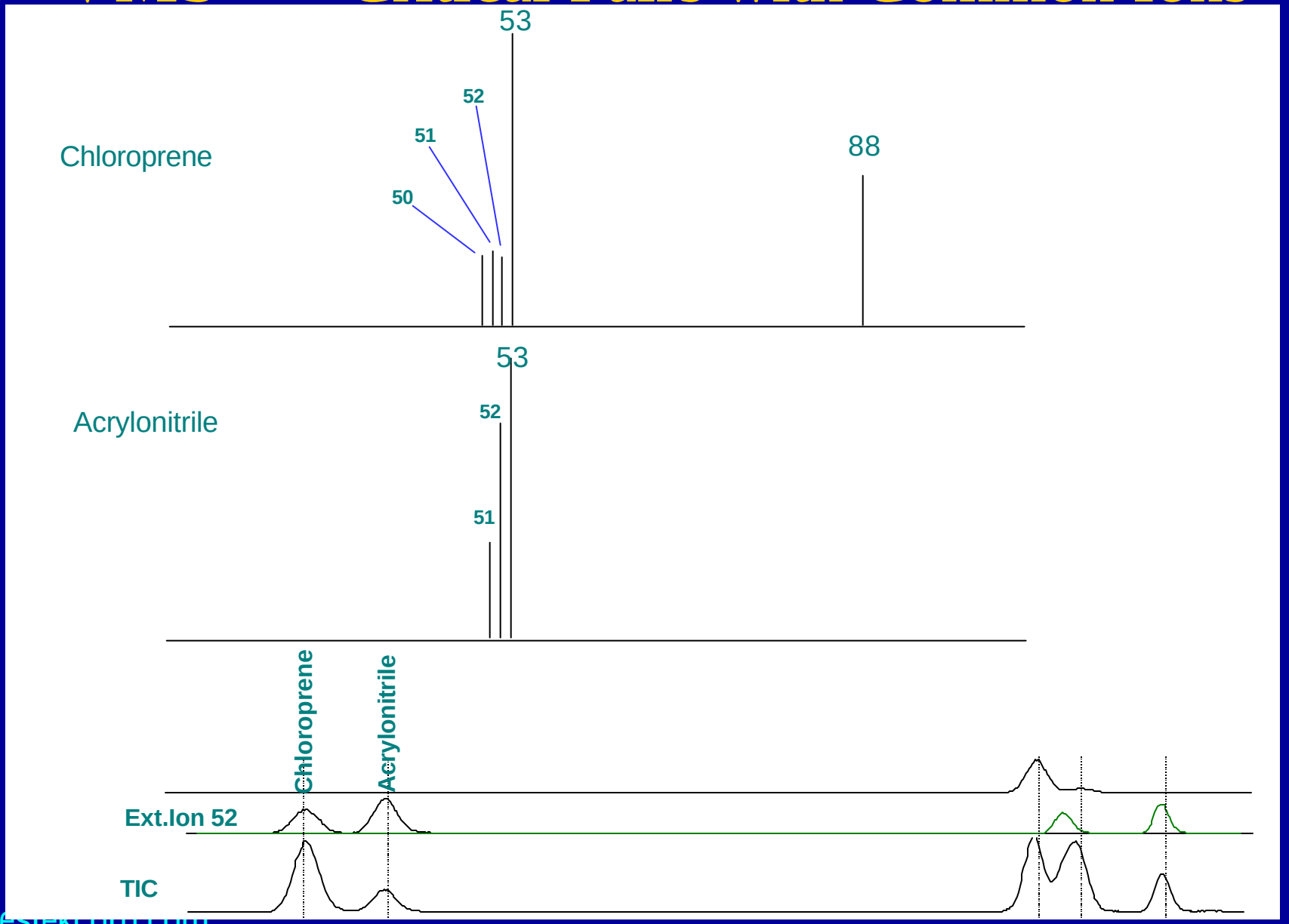
Rtx®-VMS Extracted Ion



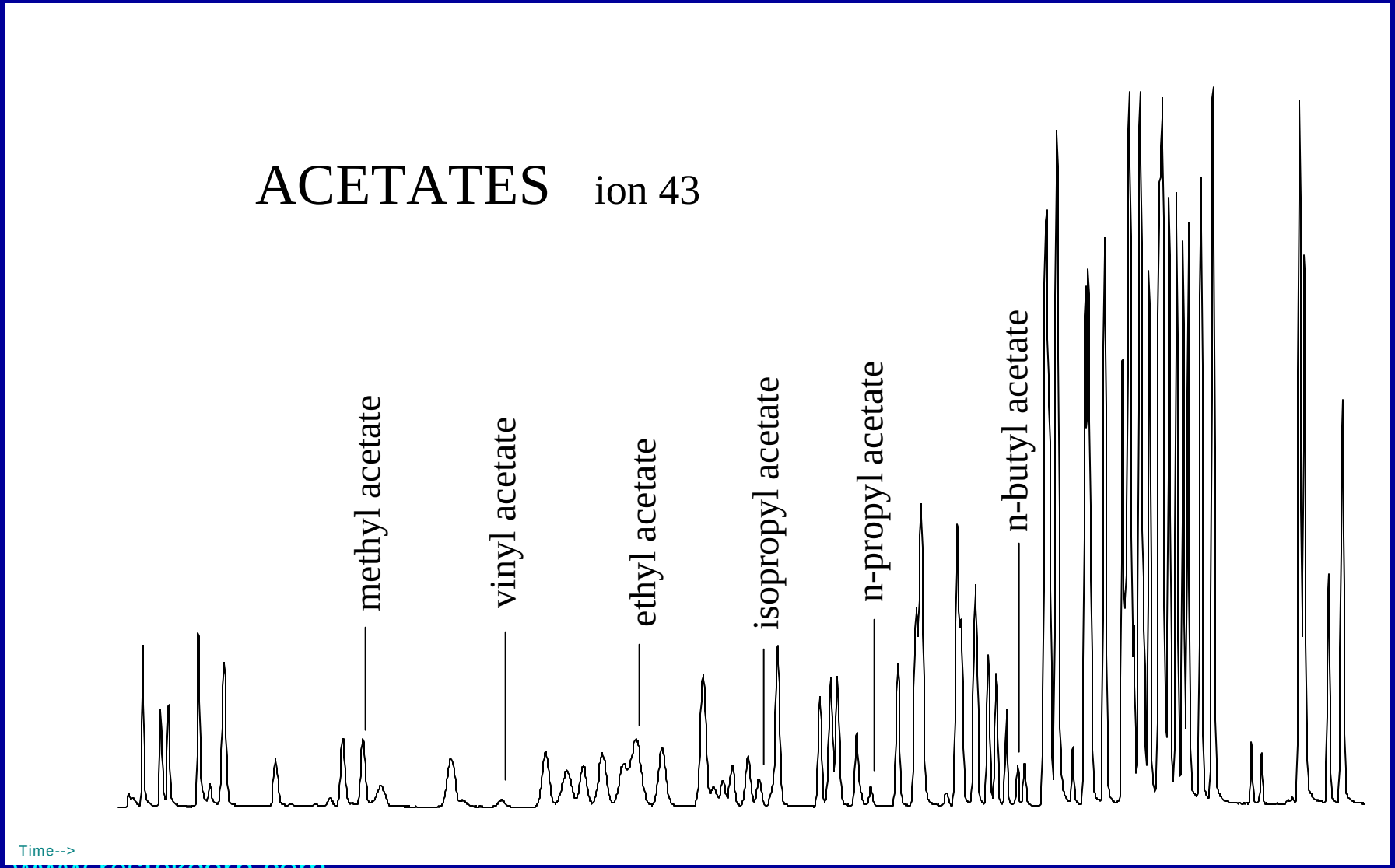
Rtx®-VMS Critical Pairs with Common Ions



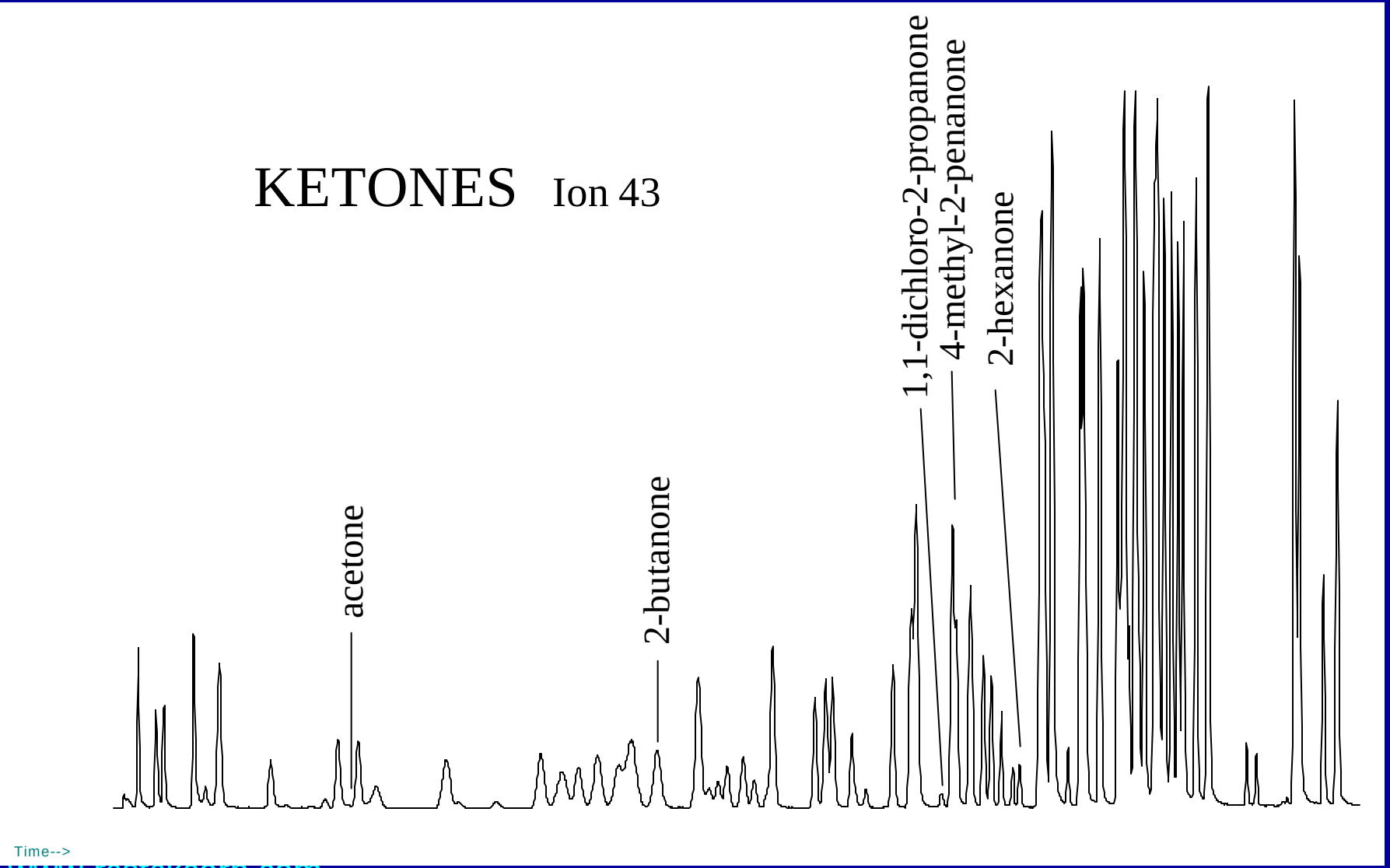
Rtx®-VMS Critical Pairs with Common Ions



Rtx®-VMS Volatiles GC/MS Column



Rtx®-VMS Volatiles GC/MS Column



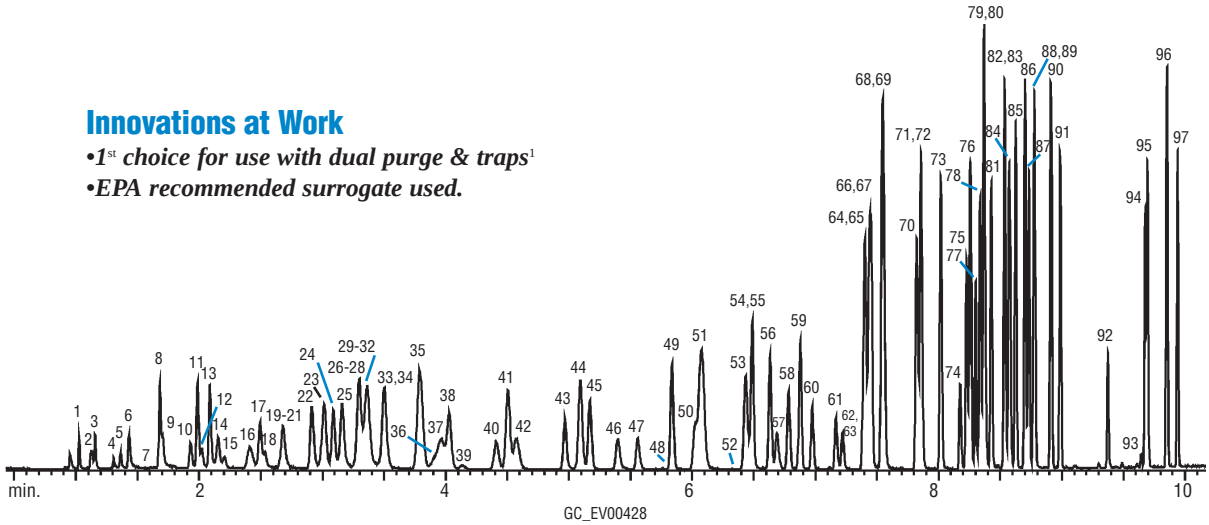
Column Variations for GC/MS – 0.18mm ID

- 0.18mm ID columns are attractive due to increased efficiency and resolution enhancements
- An Rtx-VMS 20m x 0.18mm x 1.0um column gives excellent resolution of EPA 8260B components in 10 minutes without cryogenic cooling
- Desorb flow rate set at 40ml/min for 1 min
- Column flow rate optimized at 1 ml/min

Volatile Organics
EPA Method 8260B
Rtx®-VMS

Innovations at Work

- *1st choice for use with dual purge & traps*[1]
- *EPA recommended surrogate used.*



20m, 0.18 mm ID, 1.00µm Rtx®-VMS (cat.# 49914)
Compounds in at 10ppb in 5mL of RO water
unless otherwise noted, ketones in at 2.5X

Concentrator:	Tekmar LSC-3100 Purge and Trap
Trap:	Vocarb 3000 (type K)
Purge:	11 min. @ 40mL/min. @ ambient temperature
Dry purge:	1 min. @ 40mL/min.
Desorb preheat:	245°C
Desorb:	250°C for 2 min., Flow 40mL/min.
Bake:	260°C for 8 min.
Interface:	transfer line 0.53mm ID Silcosteel® tubing 1:40 split at injection port. 1mm ID sleeve.
Oven temp.:	50°C (hold 4 min.) to 100°C @ 18°C/min. (hold 0 min.) to 230°C @ 40°C/min. (hold 3 min.)
Carrier gas:	helium @ ~1.0mL/min. constant flow

Adjust dichlorodifluoromethane to a retention time of 1.03 min. @ 50°C.

Detector:	HP 5973 MSD
Scan range:	35-300amu

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. methyl *tert*-butyl ether
15. *tert*-butyl alcohol (100ppb)
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. carbon tetrachloride
28. methyl acrylate
29. propargyl alcohol (500ppb)
30. dibromofluoromethane (SMC)
31. tetrahydrofuran
32. 1,1,1-trichloroethane
33. 2-butanone
34. 1,1-dichloropropene
35. benzene
36. pentafluorobenzene (IS)
37. *tert*-amyl-methyl ether
38. 1,2-dichloroethane
39. isobutyl alcohol (500ppb)
40. isopropyl acetate
41. trichloroethene
42. 1,4-difluorobenzene (SMC)
43. dibromomethane
44. 1,2-dichloropropane
45. bromodichloromethane
46. methyl methacrylate
47. *n*-propyl acetate
48. 2-chloroethanol (2500ppb)
49. *cis*-1,3-dichloropropene
50. toluene-d8(SMC)
51. toluene
52. pyridine (250ppb)
53. tetrachloroethene
54. 4-methyl-2-pentanone
55. *trans*-1,3-dichloropropene
56. 1,1,2-trichloroethane
57. ethyl methacrylate
58. dibromochloromethane
59. 1,3-dichloropropane
60. 1,2-dibromoethane
61. *n*-butyl acetate
62. 2-hexanone
63. 2-picoline (250ppb)
64. chlorobenzene-D5 (IS)
65. chlorobenzene
66. ethylbenzene
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. bromobenzene
76. *n*-propylbenzene
77. 1,1,2,2-tetrachloroethane
78. 2-chlorotoluene
79. 1,3,5-trimethylbenzene
80. 1,2,3-trichloropropane
81. 4-chlorotoluene
82. *tert*-butylbenzene
83. pentachloroethane
84. 1,2,4-trimethylbenzene
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. 1,2,4-trichlorobenzene
96. naphthalene
97. 1,2,3-trichlorobenzene

[1]*A.L. Hilling and G. Smith, Environmental Testing & Analysis, 10(3), 15-19, 2001.*

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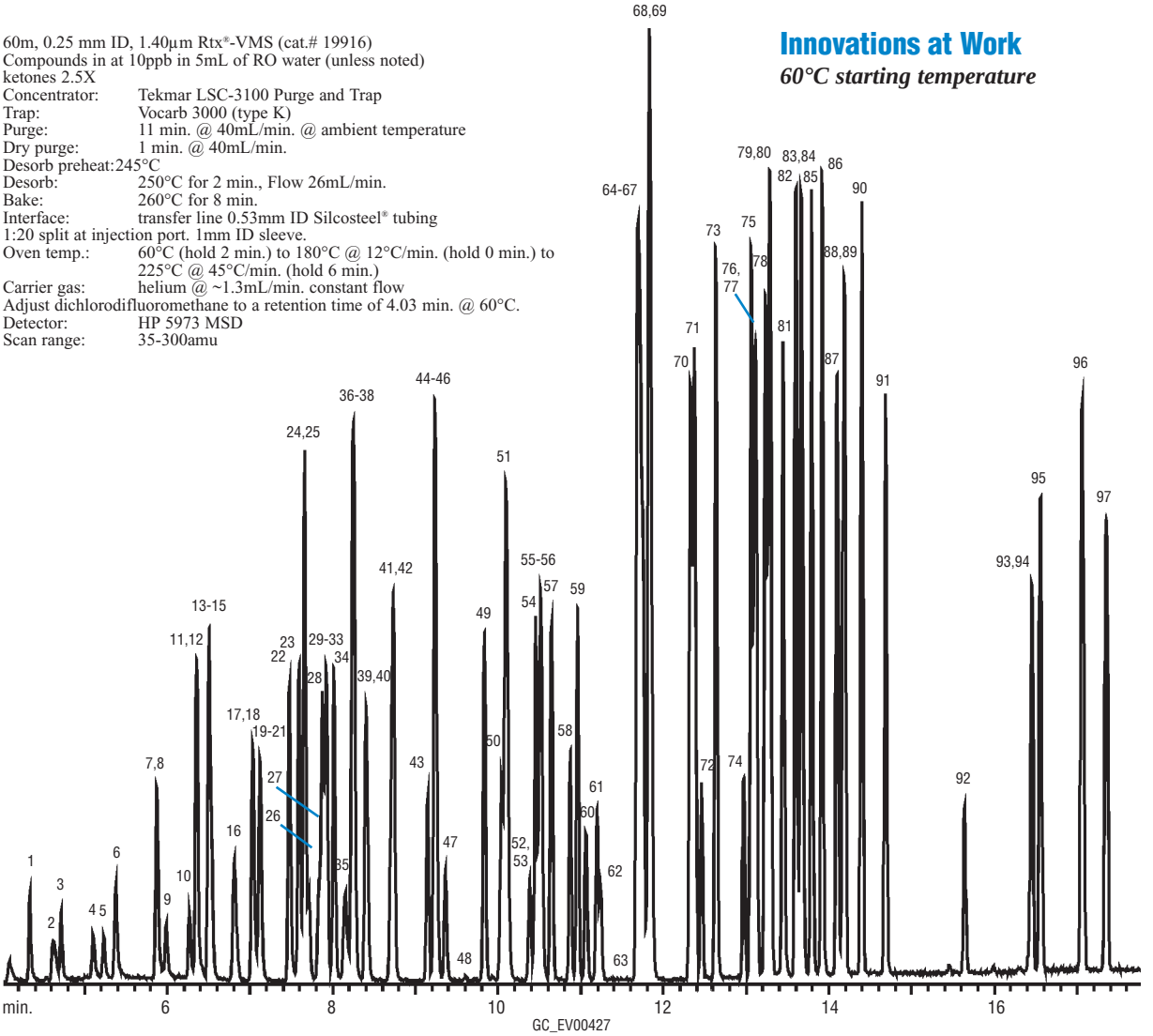
Column Variations – 0.25mm ID

- Rtx-VMS 60m x 0.25mm x 1.4um column
- 60°C starting temperature
- 20:1 split injection
- Constant Flow @ 1.3 ml/min.
- 97 components in EPA 8260B in 18 minutes

Volatile Organics
EPA Method 8260B
Rtx®-VMS

60m, 0.25 mm ID, 1.40µm Rtx®-VMS (cat.# 19916)
Compounds in at 10ppb 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. @ 40mL/min. @ ambient temperature
Dry purge: 1 min. @ 40mL/min.
Desorb preheat:245°C
Desorb: 250°C for 2 min., Flow 26mL/min.
Bake: 260°C for 8 min.
Interface: transfer line 0.53mm ID Silcosteel® tubing
1:20 split at injection port. 1mm ID sleeve.
Oven temp.: 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 @ ~1.3mL/min. constant flow
Adjust dichlorodifluoromethane to a retention time of 4.03 min. @ 60°C.
Detector: HP 5973 MSD
Scan range: 35-300amu

Innovations at Work
60°C starting temperature



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*-butyl 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-pentanone
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. styrene
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. 1,2,4-trichlorobenzene
96. naphthalene
97. 1,2,3-trichlorobenzene

**These compounds can be resolved using a lower starting temperature.*

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EPA Method 8240

- Developed to monitor 73 compounds in hazardous waste
- Rtx-VMS 30m x 0.25mm x 1.4um is a good choice for this shorter list
- Analysis times of 14 minutes

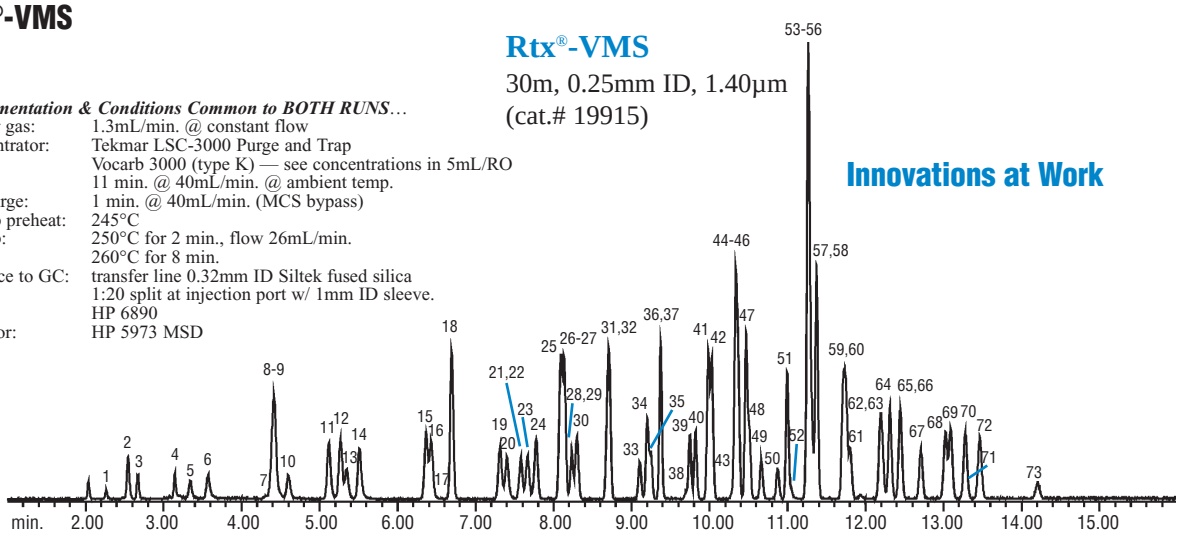
Volatile Organics
EPA Method 8240 (8260 Short List)
Rtx®-VMS

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

Innovations at Work

Instrumentation & Conditions Common to BOTH RUNS…

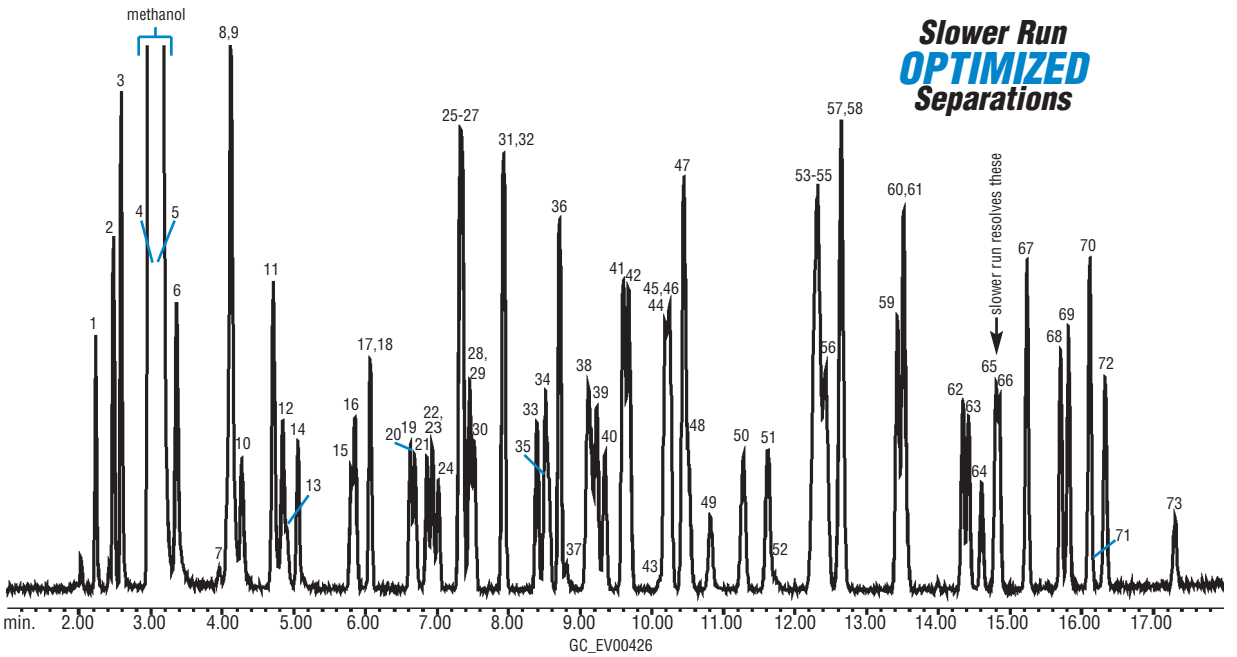
Carrier gas:	1.3mL/min. @ constant flow
Concentrator:	Tekmar LSC-3000 Purge and Trap
Trap:	Vocarb 3000 (type K) — see concentrations in 5mL/RO
Purge:	11 min. @ 40mL/min. @ ambient temp.
Dry purge:	1 min. @ 40mL/min. (MCS bypass)
Desorb preheat:	245°C
Desorb:	250°C for 2 min., flow 26mL/min.
Bake:	260°C 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:	HP 6890
Detector:	HP 5973 MSD



Top chromatogram:
Oven temp.: 40°C (hold 4 min.) to 90°C @ 16°C/min. (no hold) to 210°C @ 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 at 100ppb (cat.# 30213, 30004, 30006, 30011, 30042)
Alcohols at 1ppm (cat.# 30214) except 2Cl ethanol at 10ppm.
vinyl acetate at 500ppb (cat.#30216)
8240 Nitrile Mix at 200ppb (cat.# 30215)
8240 Mix 1A at 300ppb (cat.# 30217)
8240 Mix 2A at 500ppb (cat.# 30218)

Bottom chromatogram:
Oven temp.: 45°C (hold 4 min.) to 110°C @ 19°C/min. (hold 5 min.) to 220°C @ 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 at 100ppb (cat.# 30213, 30004, 30006, 30011, 30042)
Alcohols at 1ppm (cat.# 30214) (see MS scan)
vinyl acetate at 100ppb (cat.# 30216)
8240 Nitrile Mix at 400ppb (cat.# 30215)
8240 Mix 1A at 300ppb (cat.# 30217)
8240 Mix 2A at 500ppb (cat.# 30218)

Slower Run OPTIMIZED Separations



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. styrene
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

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EPA Method 624

- 37 aromatic and halogenated compounds in wastewater
- Previously used packed columns, but can be performed with many different capillary column dimensions
- Rtx-VMS 30m x 0.25mm x 1.4um gives separation in less than 15 minutes, starting at 40°C

EPA Method 624
Rtx®-VMS

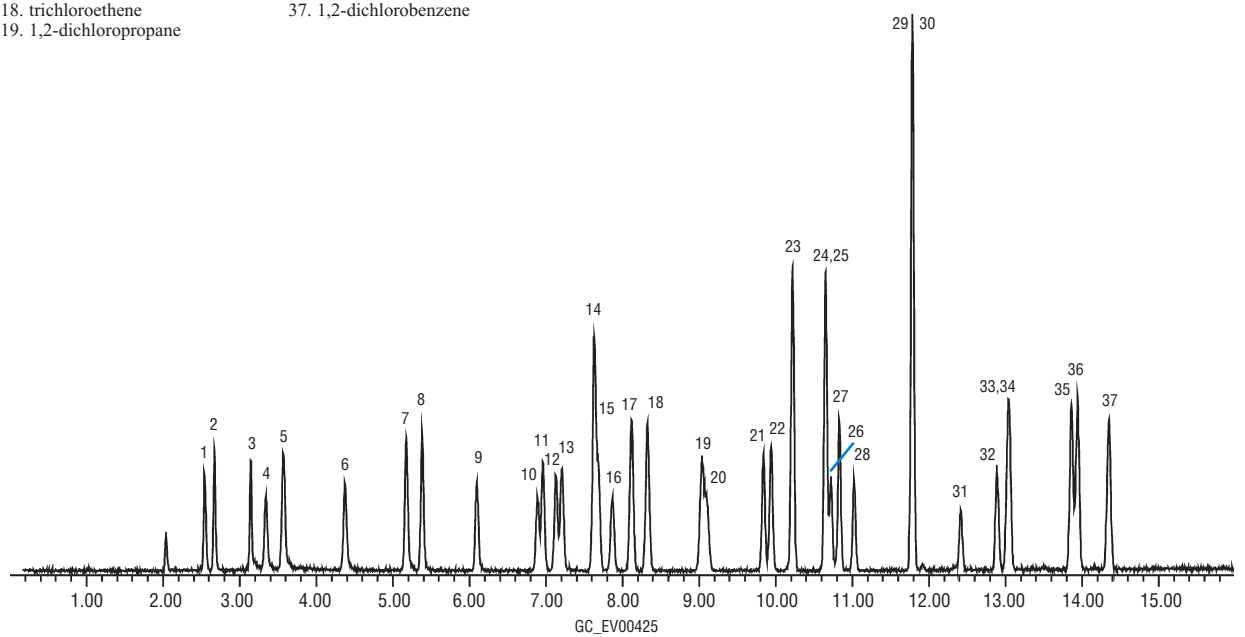
1. chloromethane
2. vinyl chloride
3. bromomethane
4. chloroethane
5. trichlorofluoromethane
6. 1,1-dichloroethene
7. methylene chloride
8. *trans*-1,2-dichloroethene
9. 1,1-dichloroethane
10. bromochloromethane
11. chloroform
12. carbon tetrachloride
13. 1,1,1-trichloroethane
14. benzene
15. pentafluorobenzene
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

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

Conc.:	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.
GC Interface:	1:10 split at injection port. 1mm ID sleeve.
GC:	HP 6890
Oven temp.:	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 @ ~1mL/min. constant flow

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

Detector:	HP 5973 MSD
Scan range:	25-300amu



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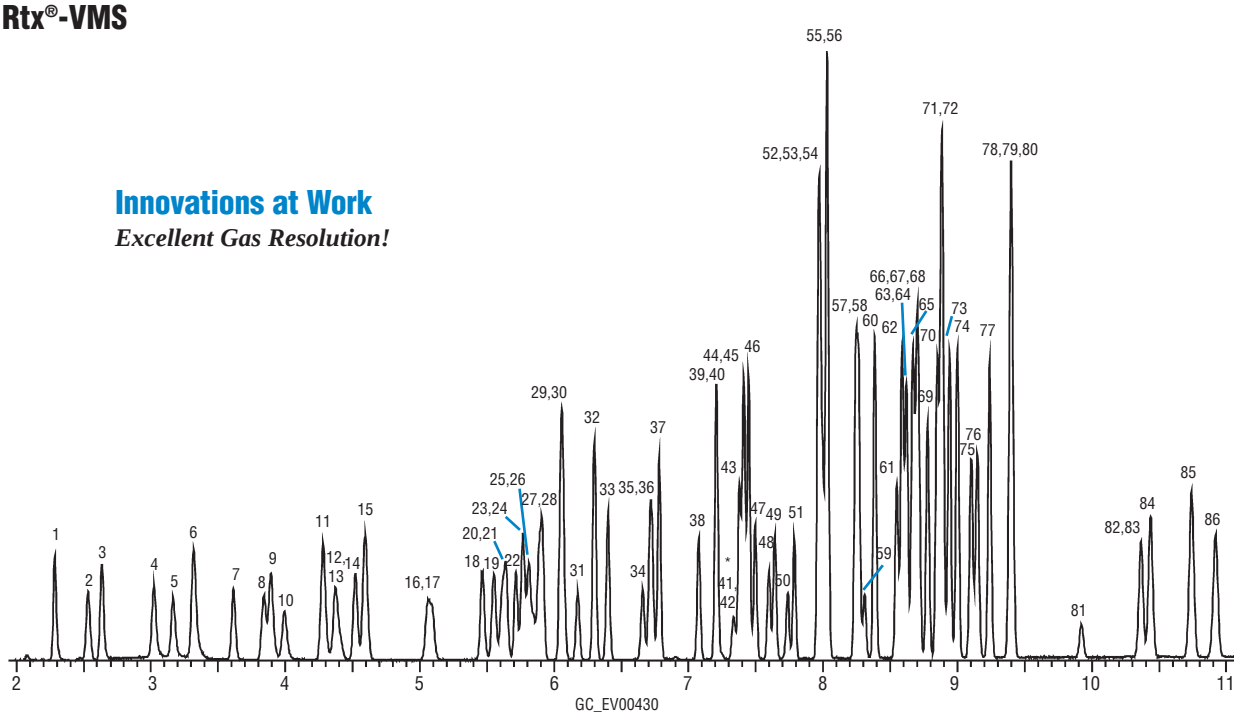
EPA Method 524.2 revision IV

- Monitors 84 compounds in drinking water
- Rtx-VMS overcomes closely eluting pairs found on other phases (ex. “624”) such as 1,1-dichloro-2-propanone/4-methyl-2-pentanone and methyl acrylate/propionitrile
- Rtx-VMS 30m x 0.25mm x 1.4um performs the analysis in less than 11 minutes
- Only difficult pair on the Rtx-VMS is 2-nitropropane/ 1,1-dichloro-2-propanone which share ion 43

EPA Method 524.2, Revision 4
Rtx®-VMS

Innovations at Work

Excellent Gas Resolution!



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°
Concentrator: Tekmar LSC-3000 Purge and Trap
Oven temp.: 45°C (hold 2 min.) to 85°C @ 14°C/min. to 210°C @ 40°C/min. (hold 4 min.)
GC: HP 6890 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: HP 5973 MSD
Scan range: 35-300amu

Standards:
20ppb in 5mL of RO water (unless otherwise noted); ketones at 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)

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-pentanone
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. styrene
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

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

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Conclusions

- Computer modeling can be used to optimize phase development, column dimensions and run conditions
- Column dimensions to interface with the GC/MS is a matter of customer preference
- Analysis of 100 + compounds typically use longer columns for better resolution and higher starting temperatures
- Rtx-VMS has excellent selectivity and rapid cycle time for all commonly used EPA GC/MS volatile methods

For More Information...

Author e-mail: cenglish@restekcorp.com

Restek Corporation
110 Benner Circle
Bellefonte, PA 16823
(800) 356-1688