

Volatile Organic Compounds by GC/MS

Columns and Reference Mixes for US EPA 524.2 Revision IV.

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- All 84 compounds listed in Method 524.2 resolved in 12 minutes, using an Rtx[®]-VMS column.
- MegaMix[™] reference mix includes 73 compounds in stable solution.
- Three reference mixes include all 84 compounds.

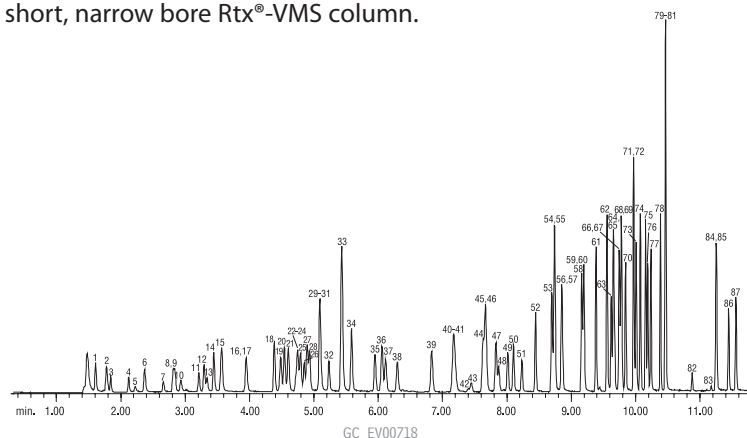
Initially, US Environmental Protection Agency Method 524.2, a purge and trap, capillary GC/MS method, was used to identify 60 volatile aromatic and halogenated hydrocarbons in municipal drinking water. Revision 4.0 (1992) added 24 polar compounds and, in 2003, California allowed the addition of *tert*-amyl methyl ether (TAME), *tert*-butyl alcohol (TBA), ethyl-*tert*-butyl ether (ETBE), and 1,1,2-trichlorotrifluoroethane (Freon[®] 113) to the list of target compounds.

Of these 84 compounds, only the six gases and five ketones are not included in Drinking Water VOA MegaMix[™], 524.2 Rev 4.1. To help ensure long-term stability of the mixes, we offer the ketones separately, in purge & trap methanol/water (90:10, v/v). This solvent system protects the keto groups and prevents acetal formation, which is more likely to occur in 100% methanol.

Table 1 Method 524.2 analytes and internal standards.

| | | |
|--------------------------------------|---------------------------------------|---|
| 1. dichlorodifluoromethane | 30. methacrylonitrile | 59. styrene |
| 2. chloromethane | 31. benzene | 60. bromoform |
| 3. vinyl chloride | 32. 1,2-dichloroethane | 61. isopropylbenzene |
| 4. bromomethane | 33. fluorobenzene (IS) | 62. 4-bromofluorobenzene (IS) |
| 5. chloroethane | 34. trichloroethene | 63. bromobenzene |
| 6. trichlorofluoromethane | 35. dibromomethane | 64. <i>n</i> -propylbenzene |
| 7. diethyl ether | 36. 1,2-dichloropropane | 65. 1,1,2,2-tetrachloroethane |
| 8. 1,1-dichloroethene | 37. bromodichloromethane | 66. 2-chlorotoluene |
| 9. carbon disulfide | 38. methyl methacrylate | 67. 1,2,3-trichloropropane |
| 10. iodomethane | 39. <i>cis</i> -1,3-dichloropropene | 68. 1,3,5-trimethylbenzene |
| 11. allyl chloride | 40. toluene | 69. <i>trans</i> -1,4-dichloro-2-butene |
| 12. methylene chloride | 41. chloroacetonitrile | 70. 4-chlorotoluene |
| 13. acetone | 42. 2-nitropropane | 71. <i>tert</i> -butylbenzene |
| 14. <i>trans</i> -1,2-dichloroethene | 43. 1,1-dichloro-2-propanone | 72. pentachloroethane |
| 15. methyl <i>tert</i> -butyl ether | 44. 4-methyl-2-pentanone | 73. 1,2,4-trimethylbenzene |
| 16. 1,1-dichloroethane | 45. tetrachloroethene | 74. <i>sec</i> -butylbenzene |
| 17. acrylonitrile | 46. <i>trans</i> -1,3-dichloropropene | 75. <i>p</i> -isopropyltoluene |
| 18. <i>cis</i> -1,2-dichloroethene | 47. 1,1,2-trichloroethane | 76. 1,3-dichlorobenzene |
| 19. 2,2-dichloropropane | 48. ethyl methacrylate | 77. 1,4-dichlorobenzene |
| 20. bromochloromethane | 49. dibromochloromethane | 78. <i>n</i> -butylbenzene |
| 21. chloroform | 50. 1,3-dichloropropane | 79. hexachloroethane |
| 22. methyl acrylate | 51. 1,2-dibromoethane | 80. 1,2-dichlorobenzene-d4 (IS) |
| 23. carbon tetrachloride | 52. 2-hexanone | 81. 1,2-dichlorobenzene |
| 24. tetrahydrofuran | 53. chlorobenzene | 82. 1,2-dibromo-3-chloropropane |
| 25. 1,1,1-trichloroethane | 54. ethylbenzene | 83. nitrobenzene |
| 26. 2-butanone | 55. 1,1,1,2-tetrachloroethane | 84. hexachlorobutadiene |
| 27. 1,1-dichloropropene | 56. <i>m</i> -xylene | 85. 1,2,4-trichlorobenzene |
| 28. 1-chlorobutane | 57. <i>p</i> -xylene | 86. naphthalene |
| 29. propionitrile | 58. <i>o</i> -xylene | 87. 1,2,3-trichlorobenzene |

Peaks 1-6 are components of cat. # 30439; peaks 13,26,43,44,52 are components of cat. # 30602; all other analytes except IS are components of cat. # 30601.

Figure 1 12-minute analysis of 84 volatile compounds, using a short, narrow bore Rtx®-VMS column.**Purge and Trap Conditions:**

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. (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: Silcosteel® transfer line
 1:30 split at injection port. 1mm ID split injection sleeve (cat.# 20972).
 Column: Rtx®-VMS, 30m, 0.25mm ID, 1.4µm (cat.# 19915)
 Sample: 502.2 Calibration Mix #1 (cat.# 30042)
 Drinking Water VOA MegaMix™, 524.2 Rev 4 (cat.# 30601)
 524 Internal Standard/Surrogate Mix (cat.# 30201)
 Ketone Mix, EPA Method 524.2 Rev 4.1 (cat.# 30602)
 Compounds at 20 ppb each in 5mL RO water
 (ketones at 50ppb; internal standards at 40ppb)
 Inj. temp.: 250°C
 Carrier gas: helium, constant flow
 Flow rate: 1.1mL/min.
 Dead time: 1.48 min. @ 40°C
 Oven temp.: 40°C (hold 2 min.) to 85°C @ 14°C/min. (hold 2 min.)
 to 220°C @ 30°C/min. (hold 4 min.).
 Det: Agilent 5971A GC/MS
 Transfer line temp.: 280°C
 Scan range: 35-300 amu
 Tune: PFTBA/BFB
 Ionization: EI

A 30m, 0.25mm ID, 1.4µm Rtx®-VMS capillary column (cat.# 19915) is an excellent choice for analyzing the 84 target compounds (Figure 1). This narrow bore column improves resolution of traditionally coeluting compounds, such as carbon tetrachloride / 1,1,1-trichloroethane, while shortening the analysis time. Analysis time is less than 12 minutes, and the cycle time is 16 minutes, which is well below the cycle time of a standard purge and trap system. This allows the fastest run-time attainable using a Tekmar 3100 purge and trap unit coupled to a single GC. A slower initial temperature ramp rate makes additional resolution possible.

We recommend using the 30m, 0.25mm ID column for best resolution of the target gases. At 20ppb in 5mL water, the gases are better than 90% resolved, using an initial temperature of 40°C (Figure 1). We encourage laboratories using either dual purge and trap technology or newer purge and trap systems with rapid cycle times to use a 20m, 0.18mm ID, 1.0µm Rtx®-VMS column for sub-10 minute runtimes.^{1,2} Whatever your system for analyzing volatiles, we offer the columns, analytical standards, GC accessories, and technical knowledge to get your laboratory running these analyses quickly and accurately.

References

- Butler J.C., E. Phillips, and M. Conoley Application Note AN9197, Thermo Electron Corporation, 2215 Grand Avenue Parkway, Austin, TX., 2003.
- A.L. Hilling and G. Smith, Environmental Testing & Analysis, 10 (3),15-19, 2001.

Rtx®-VMS (fused silica)

| ID | df (µm) | temp. limits | length | cat. # |
|--------|---------|------------------|----------|--------|
| 0.18mm | 1.00 | -40 to 240/260°C | 20-Meter | 49914 |
| 0.25mm | 1.40 | -40 to 240/260°C | 30-Meter | 19915 |

Drinking Water VOA MegaMix™, 524.2 Rev. 4.1

(73 components—see Table 1)

2,000µg/mL each in P&T methanol, 1mL/ampul
 cat. # 30601 (ea.)

Ketones Mix, 524.2 Rev. 4.1 (5 components)

acetone 2-hexanone
 2-butanone (MEK) 4-methyl-2-pentanone (MIBK)
 1,1-dichloro-2-propanone
 5,000µg/mL each in 90% P&T methanol:10% water, 1mL/ampul
 cat. # 30602 (ea.)

502.2 Calibration Mix #1 (gases)

bromomethane dichlorodifluoromethane
 chloroethane trichlorofluoromethane
 chloromethane vinyl chloride
 200µg/mL each in P&T methanol, 1mL/ampul
 cat. # 30439 (ea.)
 2,000µg/mL each in P&T methanol, 1mL/ampul
 cat. # 30042 (ea.)

For individual solutions of *tert*-amyl methyl ether, *tert*-butyl alcohol, ethyl-*tert*-butyl ether, and 1,1,1-trichlorotrifluoroethane (Freon® 113), and for internal and surrogate standards, please see our catalog, or visit our website.