

Fast GC Analyses of Volatiles

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Introduction

The primary aim of Fast GC is to maintain (compared to conventional GC) sufficient resolving power in a shorter time. Basically, Fast GC is accomplished by using a short column (reduces analysis time) with a narrow I.D. (offsets the loss of efficiency of the shorter column) while manipulating specific operating parameters, such as linear velocity and oven temperature ramp rates (1). The use of Fast GC has previously been demonstrated for several applications (2-7). In this space, Fast GC will be demonstrated for the analysis of volatiles using purge and trap (P&T).

Options for Decreasing Analysis Time for P&T Methods

The overwhelming number of samples that must be analyzed for the presence of volatiles is due to the ease with which many of these compounds are able to migrate through the environment, because of their water-soluble nature. Many regulatory agencies require constant monitoring for volatiles, resulting in heavy sample loads with short turnaround times. Therefore, laboratories are constantly looking for ways to reduce analysis times. Several options currently exist for decreasing analysis time.

1. Use two P&Ts for each GC. Synchronize so while the GC is analyzing the sample from one P&T, that P&T is in bake mode and the other P&T is purging the next sample. When the GC is ready, a sample is also ready for desorption so that the GC is never idle.
2. Use a P&T model that employs super high flow rates (i.e. 400 mL/min.) during the bake mode so that it is ready to purge the next sample sooner.
3. Convert the existing conventional GC method to a Fast GC method. Note that this option can be used with the current equipment found in most laboratories, or in combination with either (or both) option(s) listed above.

Converting Conventional GC to Fast GC for Waste Water Volatiles

Converting a conventional GC method to a Fast GC method is not as simple as just changing to a smaller I.D. column. Column dimensions, linear velocity, and oven temperature ramp rates must be optimized together. Changing only one parameter may decrease analysis time (desirable), but will likely cause a loss of resolution (undesirable). It is also critical to account for the reduced sample capacity of the smaller I.D. column.

A method for the analysis of volatiles from waste water samples (US EPA Method 624, commonly performed in the United States) on the SPB™-624 column was selected to illustrate the change from conventional GC to Fast GC. The optimized chromatogram obtained using conventional GC is shown in Figure 1. The analysis time is <18 minutes, peak shapes are good, and the mass spectrometer (MS) is able to mass resolve all analytes.

The column dimensions were changed to Fast GC dimensions, and then linear velocity and oven temperature ramp rates were optimized to produce the chromatogram shown in Figure 2 (parameters which were changed are highlighted). While analysis time is reduced to <10 minutes and the MS is still able to mass resolve all analytes, peak shape overall is not good. Why the poor chromatography? The reduced sample capacity of the smaller I.D. column was not accounted for.

The primary aim of Fast GC is to maintain (compared to conventional GC) sufficient resolving power in a shorter time

The capacity of the 0.18 mm I.D. column is significantly less than the 0.25 mm I.D. column, leading to sample overload. To alleviate this problem, the mass of sample reaching the column must be reduced. Diluting the sample, decreasing the injection volume, or increasing the split ratio are options to achieve this. The first two options are not as compatible with P&T methods as the third. Therefore, the split ratio was increased from 30:1 to 100:1, and then the linear velocity was optimized to achieve the chromatogram shown in Figure 3 (parameters which were changed are highlighted). Now, the short analysis time (<10 minutes) and mass resolution of all analytes is accompanied with improved peak shape of all analytes. This represents a vast improvement in analysis time (from over 17 minutes to under 10 minutes) compared to the conventional GC chromatogram in Figure 1.

(continued on page 9)

Did you know...?

Supelco currently offers a total of **nineteen columns in Fast GC dimensions**, covering twelve popular phases (SPB-624, VOCOL™, SLB™-5ms, Equity®-1701, TCEP, SP™-2560, Omegawax™ 100, SUPELCOWAX™ 10, Equity-1, SPB-1, Equity-5, and SPB-5). If increasing sample throughput is your goal, consider a change to a Supelco Fast GC column.

Figure 1. Waste Water Volatiles on the SPB-624

sample/matrix: each analyte at 50 ppb in 5 mL water
 purge trap: VOCARB® 3000 "K" (24940-U)
 purge: 40 mL/min. at 25 °C for 11 min.
 dry purge: 2 min.
 desorption pre-heat: 205 °C
 desorption temp.: 210 °C for 2 min.
 desorption flow: 40 mL/min.
 bake: 260 °C for 10 min.
 transfer line/valve temp.: 110 °C
 column: SPB-624, 30 m x 0.25 mm I.D., 1.4 µm (24255)
 oven: 40 °C (2 min.), 7 °C/min. to 135 °C, 30 °C/min. to 230 °C (3 min.)
 inj.: 150 °C
 MSD interface: 200 °C
 scan range: m/z = 35-400
 carrier gas: helium, 1.1 mL/min.
 injection: 30:1 split
 liner: 0.75 mm I.D. SPME

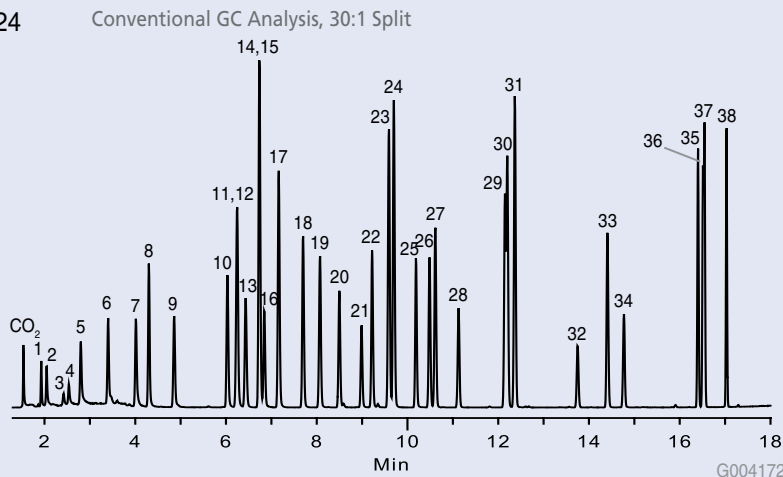


Figure 2. Waste Water Volatiles on the SPB-624

sample/matrix: each analyte at 50 ppb in 5 mL water
 purge trap: VOCARB® 3000 "K" (24940-U)
 purge: 40 mL/min. at 25 °C for 11 min.
 dry purge: 2 min.
 desorption pre-heat: 205 °C
 desorption temp.: 210 °C for 2 min.
 desorption flow: 40 mL/min.
 bake: 260 °C for 10 min.
 transfer line/valve temp.: 110 °C
 column: SPB-624, 20 m x 0.18 mm I.D., 1.0 µm (28662-U)
 oven: 40 °C (1 min.), 11 °C/min. to 125 °C, 35 °C/min. to 230 °C (2 min.)
 inj.: 150 °C
 MSD interface: 200 °C
 scan range: m/z = 35-400
 carrier gas: helium, 1.2 mL/min.
 injection: 30:1 split
 liner: 0.75 mm I.D. SPME

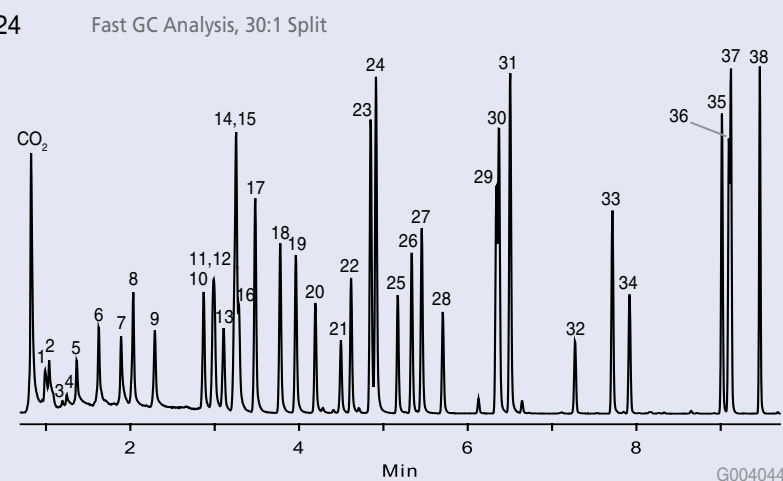
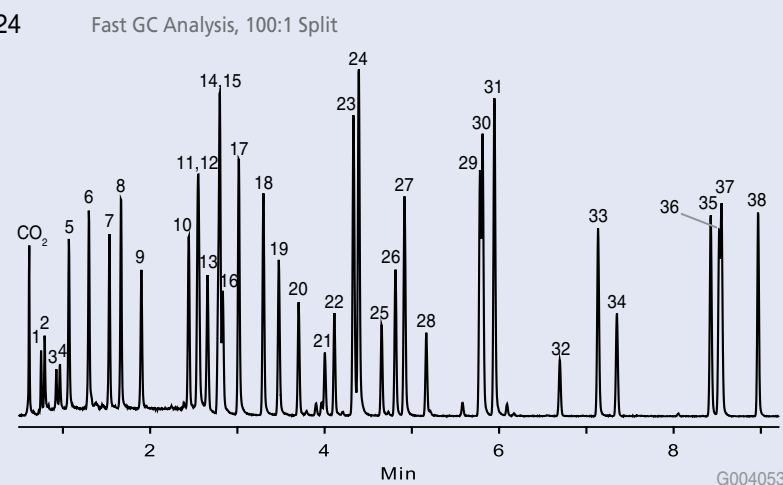


Figure 3. Waste Water Volatiles on the SPB-624

sample/matrix: each analyte at 50 ppb in 5 mL water
 purge trap: VOCARB 3000 "K" (24940-U)
 purge: 40 mL/min. at 25 °C for 11 min.
 dry purge: 2 min.
 desorption pre-heat: 205 °C
 desorption temp.: 210 °C for 2 min.
 desorption flow: 150 mL/min.
 bake: 260 °C for 10 min.
 transfer line/valve temp.: 110 °C
 column: SPB-624, 20 m x 0.18 mm I.D., 1.0 µm (28662-U)
 oven: 40 °C (1 min.), 11 °C/min. to 125 °C, 35 °C/min. to 230 °C (2 min.)
 inj.: 150 °C
 MSD interface: 200 °C
 scan range: m/z = 35-400
 carrier gas: helium, 1.5 mL/min.
 injection: 100:1 split
 liner: 0.75 mm I.D. SPME



Peak IDs for Figures 1-3

- | | | | |
|-----------------------------|---|---|---|
| 1. Chloromethane | 11. Dibromofluoromethane (surr.) | 21. 2-Chloroethyl vinyl ether | 31. Ethylbenzene |
| 2. Vinyl chloride | 12. 1,1,1-Trichloroethane | 22. cis-1,3-Dichloropropene | 32. Bromoform |
| 3. Bromomethane | 13. Carbon tetrachloride | 23. Toluene-d ₈ (surr.) | 33. 4-Bromofluorobenzene (surr.) |
| 4. Chloroethane | 14. 1,2-Dichloroethane-d ₄ (surr.) | 24. Toluene | 34. 1,1,2,2-Tetrachloroethane |
| 5. Trichlorofluoromethane | 15. Benzene | 25. trans-1,3-Dichloropropene | 35. 1,3-Dichlorobenzene |
| 6. 1,1-Dichloroethane | 16. 1,2-Dichloroethane | 26. 1,1,2-Trichloroethane | 36. 1,4-Dichlorobenzene-d ₄ (I.S.) |
| 7. Methylene chloride | 17. Fluorobenzene (I.S.) | 27. Tetrachloroethene | 37. 1,4-Dichlorobenzene |
| 8. trans-1,2-Dichloroethane | 18. Trichloroethene | 28. Dibromochloromethane | 38. 1,2-Dichlorobenzene |
| 9. 1,1-Dichloroethane | 19. 1,2-Dichloropropane | 29. Chlorobenzene-d ₅ (I.S.) | |
| 10. Chloroform | 20. Bromodichloromethane | 30. Chlorobenzene | |

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Figure 4. Solid Waste Volatiles on the VOCOL

sample/matrix: each analyte at 50 ppb in 5 mL water
 purge trap: VOCARB 3000 "K" (24940-U)
 purge: 40 mL/min. at 25 °C for 11 min.
 dry purge: 2 min.
 desorption pre-heat: 205 °C
 desorption temp.: 210 °C for 2 min.
 desorption flow: 40 mL/min.
 bake: 260 °C for 10 min.
 transfer line/valve temp.: 110 °C
 column: VOCOL, 30 m x 0.25 mm I.D., 1.5 µm (24205-U)
 oven: 40 °C (2 min.), 7 °C/min. to 125 °C,
 12 °C/min. to 220 °C (5 min.)
 inj.: 150 °C
 MSD interface: 200 °C
 scan range: m/z = 35-400
 carrier gas: helium, 0.7 mL/min.
 injection: 30:1 split
 liner: 0.75 mm I.D. SPME

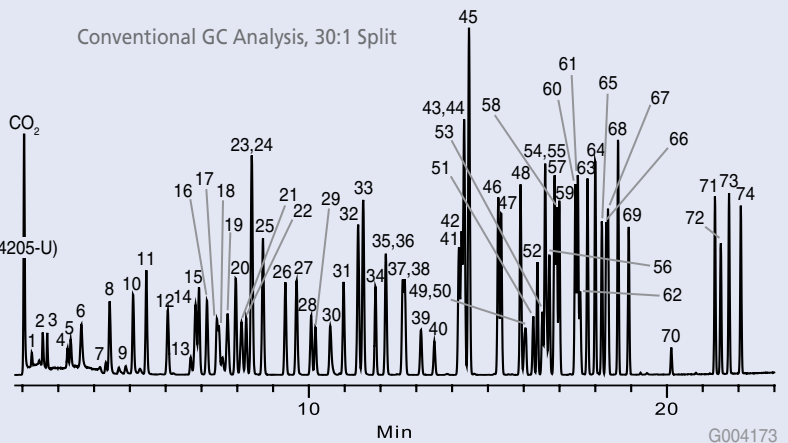


Figure 5. Solid Waste Volatiles on the VOCOL

sample/matrix: each analyte at 50 ppb in 5 mL water
 purge trap: VOCARB 3000 "K" (24940-U)
 purge: 40 mL/min. at 25 °C for 11 min.
 dry purge: **1 min.**
 desorption pre-heat: 205 °C
 desorption temp.: 210 °C for **1 min.**
 desorption flow: **46 mL/min.**
 bake: 260 °C for 10 min.
 transfer line/valve temp.: 110 °C
 column: VOCOL, **20 m x 0.18 mm I.D., 1.0 µm (28463-U)**
 oven: 40 °C (0.8 min.), 19 °C/min. to 125 °C,
 32 °C/min. to 220 °C (1 min.)
 inj.: 150 °C
 MSD interface: 220 °C
 scan range: m/z = 35-400
 carrier gas: helium, **1.4 mL/min.**
 injection: 30:1 split
 liner: 0.75 mm I.D. SPME

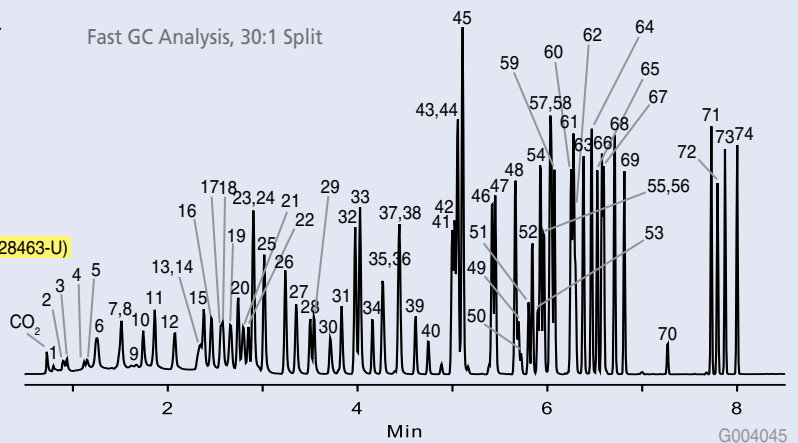
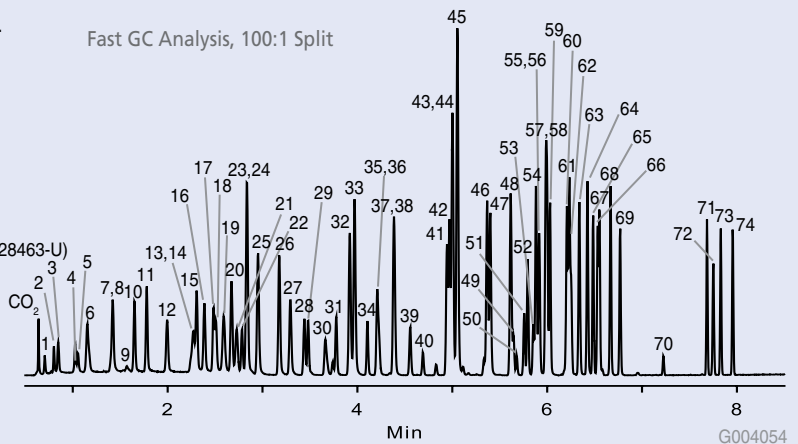


Figure 6. Solid Waste Volatiles on the VOCOL

sample/matrix: each analyte at 50 ppb in 5 mL water
 purge trap: VOCARB 3000 "K" (24940-U)
 purge: 40 mL/min. at 25 °C for 11 min.
 dry purge: 1 min.
 desorption pre-heat: 205 °C
 desorption temp.: 210 °C for 1 min.
 desorption flow: **150 mL/min.**
 bake: 260 °C for 10 min.
 transfer line/valve temp.: 110 °C
 column: VOCOL, 20 m x 0.18 mm I.D., 1.0 µm (28463-U)
 oven: 40 °C (0.8 min.), 19 °C/min. to 125 °C,
 32 °C/min. to 220 °C (1 min.)
 inj.: 150 °C
 MSD interface: 220 °C
 scan range: m/z = 35-400
 carrier gas: helium, **1.5 mL/min.**
 injection: **100:1 split**
 liner: 0.75 mm I.D. SPME



Peak IDs for Figures 4-6

- | | | | | |
|------------------------------|---|---|----------------------------------|---|
| 1. Dichlorofluoromethane | 16. Chloroform | 31. cis-1,3-Dichloropropene | 46. o-Xylene | 61. 1,2,4-Trimethylbenzene |
| 2. Chloromethane | 17. Bromochloromethane | 32. Toluene-d ₈ (surr.) | 47. Styrene | 62. Pentachloroethane |
| 3. Vinyl chloride | 18. Dibromofluoromethane (surr.) | 33. Toluene | 48. Isopropylbenzene | 63. sec-Butylbenzene |
| 4. Bromomethane | 19. 1,1,1-Trichloroethane | 34. trans-1,3-Dichloropropene | 49. Bromoform | 64. p-Isopropyltoluene |
| 5. Chloroethane | 20. 1,1-Dichloropropene | 35. 1,1,2-Trichloroethane | 50. cis-1,4-Dichloro-2-butene | 65. 1,3-Dichlorobenzene |
| 6. Trichlorofluoromethane | 21. Carbon tetrachloride | 36. 2-Hexanone | 51. 1,1,2,2-Tetrachloroethane | 66. 1,4-Dichlorobenzene-d ₄ (I.S.) |
| 7. Acetone | 22. 1,2-Dichloroethane-d ₄ (surr.) | 37. 1,3-Dichloropropane | 52. 4-Bromofluorobenzene (surr.) | 67. 1,4-Dichlorobenzene |
| 8. 1,1-Dichloroethane | 23. 1,2-Dichloroethane | 38. Tetrachloroethane | 53. 1,2,3-Trichloropropane | 68. Butylbenzene |
| 9. Iodomethane | 24. Benzene | 39. Dibromochloromethane | 54. n-Propylbenzene | 69. 1,2-Dichlorobenzene |
| 10. Methylene chloride | 25. Fluorobenzene (I.S.) | 40. 1,2-Dibromomethane | 55. Bromobenzene | 70. 1,2-Dibromo-3-chloropropane |
| 11. trans-1,2-Dichloroethane | 26. Trichloroethane | 41. Chlorobenzene-d ₆ (I.S.) | 56. trans-1,4-Dichloro-2-butene | 71. 1,2,4-Trichlorobenzene |
| 12. 1,1-Dichloroethane | 27. 1,2-Dichloropropane | 42. Chlorobenzene | 57. 1,3,5-Trimethylbenzene | 72. Hexachlorobutadiene |
| 13. 2-Butanone | 28. Bromodichloromethane | 43. Ethylbenzene | 58. o-Chlorotoluene | 73. Naphthalene |
| 14. 2,2-Dichloropropane | 29. Dibromomethane | 44. 1,1,1,2-Tetrachloroethane | 59. p-Chlorotoluene | 74. 1,2,3-Trichlorobenzene |
| 15. cis-1,2-Dichloroethane | 30. 4-methyl-2-pentanone | 45. m-Xylene & p-Xylene | 60. tert-Butylbenzene | |

(continued from page 6)

Converting Conventional GC to Fast GC for Solid Waste Volatiles

Fast GC is also compatible with more complex samples. A method for the analysis of volatiles from solid waste samples (US EPA Method 8260, also commonly performed in the United States) was selected as an example, this time using the VOCOL column. The optimized chromatogram obtained using conventional GC is shown in Figure 4. The analysis time is <23 minutes.

The column dimensions were changed to Fast GC dimensions, and then conditions were optimized to produce the chromatogram shown in Figure 5 (parameters which were changed are highlighted). While analysis time is reduced to <9 minutes, the shapes of the first several peaks are not good due to the lower capacity of the 0.18 mm I.D. column. Therefore, the split ratio was increased from 30:1 to 100:1, and then the linear velocity was optimized to achieve the chromatogram shown in Figure 6 (parameters which were changed are highlighted). Again, the short analysis time (<9 minutes) is accompanied with improved peak shapes. This represents a vast improvement in analysis time (from over 22 minutes to under 9 minutes) compared to the conventional GC chromatogram in Figure 4.

Fast GC of Hazardous Waste Site Volatiles

A method for the analysis of volatiles from hazardous waste site samples (US EPA Method OLM04.2 VOA) was selected to show the selectivity differences between the SPB-624 column and the VOCOL column. Optimized Fast GC chromatograms are shown in Figure 7 (SPB-624) and Figure 8 (VOCOL). Both show quick analysis times and good shapes of all peaks. Note the change in elution order for several peaks (10/11, 18/19, 31/32, 37/38, 47/48, and 49/50) due to the selectivity difference between the two columns.

Conclusion

Converting methods from conventional GC to Fast GC can result in decreased costs (less people and/or instruments are needed) and increased revenue (more samples can be processed). However, care must be taken to ensure that all Fast GC method parameters are optimized together. Changing only one may decrease analysis time (desirable), but will likely cause a loss of resolution (undesirable). With any Fast GC method, the reduced sample capacity of the smaller I.D. column must be accounted for so that unacceptable chromatography is not created.

Fast GC methods can be used with complex samples, and with any column, regardless of its selectivity. Furthermore, Fast GC is compatible with the current equipment found in most laboratories, and also with newer equipment that is designed for speed.

References

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(continued on page 10)

+ Featured Products

Description	Cat. No.
SPB-624 Fused Silica Columns	
20 m x 0.18 mm I.D., 1.0 µm	28662-U
30 m x 0.25 mm I.D., 1.4 µm	24255
VOCOL Fused Silica Columns	
20 m x 0.18 mm I.D., 1.0 µm	28463-U
30 m x 0.25 mm I.D., 1.5 µm	24205-U
VOCARB 3000 "K" Purge Traps	
Fits OI Analytical Eclipse 4660 / 4560	24940-U

+ Related Products

Description	Cat. No.
SPB-624 Fused Silica Columns	
60 m x 0.25 mm I.D., 1.4 µm	24256
30 m x 0.32 mm I.D., 1.8 µm	23323-U
60 m x 0.32 mm I.D., 1.8 µm	24251
30 m x 0.53 mm I.D., 3.0 µm	25430
60 m x 0.53 mm I.D., 3.0 µm	28663-U
75 m x 0.53 mm I.D., 3.0 µm	25432
105 m x 0.53 mm I.D., 3.0 µm	28664-U
VOCOL Fused Silica Columns	
10 m x 0.20 mm I.D., 1.2 µm	24129-U
60 m x 0.25 mm I.D., 1.5 µm	24154
30 m x 0.32 mm I.D., 1.8 µm	28464-U
60 m x 0.32 mm I.D., 1.8 µm	24217-U
60 m x 0.32 mm I.D., 3.0 µm	24157
30 m x 0.53 mm I.D., 3.0 µm	25320-U
60 m x 0.53 mm I.D., 3.0 µm	25381
105 m x 0.53 mm I.D., 3.0 µm	25358
60 m x 0.75 mm I.D., 1.5 µm	23313-U
VOCARB 3000 "K" Purge Traps	
Fits Tekmar Velocity XPT / 2000 / 4000	21066-U
Fits Tekmar 3000 / 3100	24920-U
Fits OI Analytical 4460	21131-U
Fits Dynatech "Dyna" Models	21085-U
Fits CDS Peak Master	21159

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Figure 7. Hazardous Waste Site Volatiles on the SPB-624

sample/matrix: each analyte at 50 ppb in 5 mL water
 purge trap: VOCARB 3000 "K" (24940-U)
 purge: 40 mL/min. at 25 °C for 11 min.
 dry purge: 2 min.
 desorption pre-heat: 205 °C
 desorption temp.: 210 °C for 2 min.
 desorption flow: 124 mL/min.
 bake.: 260 °C for 10 min.
 transfer line/valve temp.: 110 °C
 column: SPB-624, 20 m x 0.18 mm I.D., 1.0 µm (28662-U)
 oven: 40 °C (1 min.), 11 °C/min. to 125 °C,
 35 °C/min. to 230 °C (2 min.)
 inj.: 150 °C
 MSD interface: 200 °C
 scan range: m/z = 35-400
 carrier gas: helium, 1.2 mL/min.
 injection: 100:1 split
 liner: 0.75 mm I.D. SPME

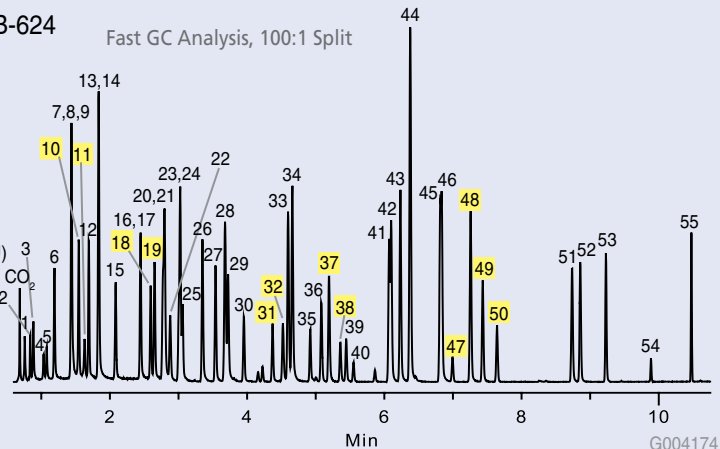
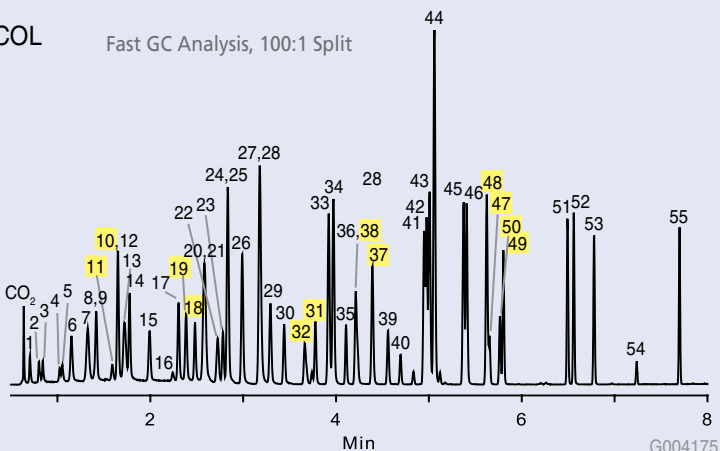


Figure 8. Hazardous Waste Site Volatiles on the VOCOL

sample/matrix: each analyte at 50 ppb in 5 mL water
 purge trap: VOCARB 3000 "K" (24940-U)
 purge: 40 mL/min. at 25 °C for 11 min.
 dry purge: 2 min.
 desorption pre-heat: 205 °C
 desorption temp.: 210 °C for 2 min.
 desorption flow: 150 mL/min.
 bake.: 260 °C for 10 min.
 transfer line/valve temp.: 110 °C
 column: VOCOL, 20 m x 0.18 mm I.D., 1.0 µm (28463-U)
 oven: 40 °C (0.8 min.), 19 °C/min. to 125 °C,
 32 °C/min. to 220 °C (1 min.)
 inj.: 150 °C
 MSD interface: 200 °C
 scan range: m/z = 35-400
 carrier gas: helium, 1.4 mL/min.
 injection: 100:1 split
 liner: 0.75 mm I.D. SPME



Peak IDs for Figures 7-8

- | | | | | |
|--|-------------------------------|---|---|----------------------------------|
| 1. Dichlorofluoromethane | 12. Methylene chloride | 23. 1,2-Dichloroethane-d ₂ (surr.) | 34. Toluene | 45. o-Xylene |
| 2. Chloromethane | 13. Methyl-tert-butyl ether | 24. Benzene | 35. trans-1,3-Dichloropropene | 46. Styrene |
| 3. Vinyl chloride | 14. trans-1,2-Dichloroethene | 25. 1,2-Dichloroethane | 36. 1,1,2-Trichloroethane | 47. Bromoform |
| 4. Bromomethane | 15. 1,1-Dichloroethane | 26. 1,4-Difluorobenzene (I.S.) | 37. Tetrachloroethene | 48. Isopropylbenzene |
| 5. Chloroethane | 16. 2-Butanone | 27. Trichloroethene | 38. 2-Hexanone | 49. 4-Bromofluorobenzene (surr.) |
| 6. Trichlorofluoromethane | 17. cis-1,2-Dichloroethene | 28. Methylcyclohexane | 39. Dibromochloromethane | 50. 1,1,2,2-Tetrachloroethane |
| 7. 1,1,2-Trichloro-1,2,2-trifluoroethane | 18. Bromochloromethane (I.S.) | 29. 1,2-Dichloropropane | 40. 1,2-Dibromomethane | 51. 1,3-Dichlorobenzene |
| 8. 1,1-Dichloroethene | 19. Chloroform | 30. Bromodichloromethane | 41. Chlorobenzene-d ₅ (I.S.) | 52. 1,4-Dichlorobenzene |
| 9. Acetone | 20. 1,1,1-Trichloroethane | 31. cis-1,3-Dichloropropene | 42. Chlorobenzene | 53. 1,2-Dichlorobenzene |
| 10. Carbon disulfide | 21. Cyclohexane | 32. 4-Methyl-2-pentanone | 43. Ethylbenzene | 54. 1,2-Dibromo-3-chloropropane |
| 11. Methyl acetate | 22. Carbon tetrachloride | 33. Toluene-d ₈ (surr.) | 44. m-Xylene & p-Xylene | 55. 1,2,4-Trichlorobenzene |

Did you know...?

The 2007 brochure "Fast GC: A Practical Guide for Increasing Sample Throughput without Sacrificing Quality" (T407096 JTW) contains valuable information concerning Fast GC principles that is not covered in this article. Included are practical considerations, theoretical discussions, a listing of columns in Fast GC dimensions, twenty-six chromatograms, a listing of related products designed to maximize performance, plus a list of literature for additional reading. Request a copy of this brochure on the attached postcard or contact Supelco Technical Service at 800-359-3041 (US and Canada only), 814-359-3041, or at techservice@sial.com



! Related Information

The Supelco "Purge-and-Trap System Guide" (T197916 BIN) contains both theory as well as troubleshooting information. Request a copy of this bulletin on the attached postcard or contact Supelco Technical Service at 800-359-3041 (US and Canada only), 814-359-3041, or at techservice@sial.com (Available in electronic form only. Please provide email address.)