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.

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
2. Vinyl chloride
3. Bromomethane
4. Chloroethane
5. Trichlorofluoromethane
6. 1,1-Dichloroethane
7. Methylene chloride
8. trans-1,2-Dichloroethene
9. 1,1-Dichloroethane
10. Chloroform
11. 2-Chloroethanol vinyl ether
12. cis-1,3-Dichloropropene
13. Toluene-d₆ (surr.)
14. Toluene
15. trans-1,3-Dichloropropene
16. 1,1,2-Trichloroethane
17. Tetrachloroethene
18. Dichloromethane
19. Chloroform-d₆ (I.S.)
20. Chloroform
21. Ethylbenzene
22. Bromodichloromethane
23. 1,1,1-Trichloroethane
24. Carbon tetrachloride
25. 1,1,2,2-Tetrachloroethane
26. 1,2-Dichlorobenzene-d₆ (I.S.)
27. 1,3-Dichlorobenzene
28. 1,2-Dichloroethane-d₄ (surr.)
29. 1,1,1-Trichloroethane-d₂
30. Chlorobenzene
**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.:** 280 °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.)
- **Injection:** 30:1 split
- **Carrier gas:** helium, 0.7 mL/min.
- **Liner:** 0.75 mm I.D. SPME

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**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.:** 280 °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.), 18 °C/min. to 125 °C,
  32 °C/min. to 220 °C (1 min.)
- **Injection:** 30:1 split
- **Carrier gas:** helium, 1.4 mL/min.
- **Liner:** 0.75 mm I.D. SPME

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**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.:** 280 °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.), 18 °C/min. to 125 °C,
  32 °C/min. to 220 °C (1 min.)
- **Injection:** 30:1 split
- **Carrier gas:** helium, 1.5 mL/min.
- **Liner:** 0.75 mm I.D. SPME

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**Peak IDs for Figures 4-6**

1. Dichlorofluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethene
6. Trichlorofluoromethane
7. Acetone
8. 1,1-Dichloroethene
9. 1,2-Dichloroethene
10. Methylene chloride
11. trans-1,2-Dichloroethene
12. 1,1-Dichloroethane
13. 2-Butanone
14. 2,2-Dichloropropane
15. cis-1,2-Dichloroethene
16. Chloroform
17. Bromochloromethane
18. Dibromofluoromethane (surr.)
19. 1,1,1-Trimethylchloroethane
20. 1,1-Dichloroethane
21. Carbon tetrachloride
22. 1,2-Dichloroethene (d.), (surr.)
23. 2,2-Dichloroethene
24. Benzene
25. Fluorobenzene (I.S.)
26. Trichloroethene
27. 1,2-Dichloropropane
28. Bromodichloromethane
29. Dibromomethane
30. cis-1,2-Dichloroethene
31. cis-1,3-Dichloropropene
32. Toluene-d1 (surr.)
33. Toluene
34. trans-1,3-Dichloropropene
35. 1,1,2-Trichloroethane
36. 2-Hexanone
37. 1,3-Dichloropropene
38. Tetrachloroethene
39. Dibromochloromethane
40. 1,2-Dibromoethane
41. Chloroform-d1 (I.S.)
42. Chlorobenzene
43. Ethylbenzene
44. 1,1,1,2-Tetrachloroethane
45. m-Xylene & p-Xylene
46. o-Xylene
47. Styrene
48. Isopropylbenzene
49. Bromoform
50. cis-1,4-Dichloro-2-butoene
51. 1,1,1,2-Tetrachloroethane
52. 4-Bromofluorobenzene (surr.)
53. 1,2,3-Tribromoethane
54. n-Propylbenzene
55. 1,2-Dichloroethane
56. Methylene chloride
57. 1,3-Dichlorobenzene
58. p-Chlorotoluene
59. 1,1-Dichloroethane
60. tert-Butylbenzene
61. 1,2,4-Trimethylbenzene
62. Pentachloroethene
63. sec-Butylbenzene
64. p-Isopropyltoluene
65. 1,3-Dichlorobenzene
66. 1,1-Dichloroethane
67. Butylbenzene
68. 1,2,3-Tribromoethane
69. 1,2,4-Trimethylbenzene
70. 1,2-Dibromo-3-chloropropane
71. 1,2,4-Trichlorobenzene
72. Hexachlorobutadiene
73. Naphthalene
74. 1,2,3-Trichlorobenzene
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

6. M.D. Buchanan, Fast GC Analysis of Detailed cis/trans Fatty Acid Methyl Esters (FAMEs) on the 75 m SP-2560 Capillary Column, Supelco The Reporter, Aug 2007; Vol. 25.4: 3-4.

Related Information

The complete list of our analytical standards can be viewed at sigma-aldrich.com/standards

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

- Oven: 40 °C (0.8 min.), 19 °C/min. to 125 °C,
- Oven: 40 °C (1 min.), 11 °C/min. to 125 °C,
- Sample/matrix: each analyte at 50 ppb in 5 mL water
- Column: VOCOL, 20 m x 0.18 mm I.D., 1.0 μm (28463-U)
- Transfer line/valve temp.: 110 °C
- Bake.: 260 °C for 10 min.
- Desorption flow: 150 mL/min.
- Desorption temp.: 210 °C for 2 min.
- Desorption pre-heat: 205 °C
- Dry purge: 2 min.
- Purge: 40 mL/min. at 25 °C for 11 min.
- Purge trap: VOCARB 3000 “K” (24940-U)
- Linner: 0.75 mm I.D. SPME
- Injection: 100:1 split
- Carrier gas: helium, 1.4 mL/min.
- Scan range: m/z = 35-400
- MSD interface: 200 °C
- Inj.: 150 °C

Peak IDs for Figures 7-8

1. Dichlorofluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethane
6. Trichlorofluoromethane
7. 1,1,2-Trichloro-1,2,2-trifluoroethane
8. 1,1-Dichloroethene
9. Acetone
10. Carbon disulfide
11. Methyl acetate
12. Methylene chloride
13. Methyl tert-butyl ether
14. trans-1,2-Dichloroethene
15. 1,1-Dichloroethane
16. 2-Butanone
17. cis-1,2-Dichloroethene
18. Bromochloromethane (I.S.)
19. Chloroform
20. 1,1,1-Trichloroethane
21. Cyclohexane
22. Carbon tetrachloride
23. 1,2-Dichloroethane-d₄ (surr.)
24. Benzene
25. 1,2-Dichloroethane
26. 1,4-Difluorobenzene (I.S.)
27. Trichloroethene
28. Methylcyclohexane
29. Trichloroethene
30. Bromodichloromethane
31. cis-1,3-Dichloropropene
32. Methylcyclohexane
33. Toluene-d₄ (surr.)
34. Toluene
35. trans-1,3-Dichloropropene
36. 1,1,2-Trichloroethane
37. Tetrachloroethene
38. 2-Hexanone
39. Dibromochloromethane
40. 1,2-Dibromoethene
41. Chlorobenzene-d₆ (I.S.)
42. Chlorobenzene
43. Ethylbenzene
44. m-Xylene & p-Xylene
45. o-Xylene
46. Styrene
47. Bromofluorobenzene (surr.)
48. Isopropylbenzene (I.S.)
49. Isobutylbenzene
50. 1,1,2,2-Tetrachloroethane
51. 1,3-Dichlorobenzene
52. 1,4-Dichlorobenzene
53. 1,2-Dichlorobenzene
54. 1,2-Dibromo-3-chloropropane
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.)