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Fast GC Analysis Using the 0.10mm ID Equity-5

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A current trend in gas chromatography has been decreased analysis times and increased sample throughput. Decreased run time can be achieved by decreasing column length, inner diameter (ID), stationary phase film thickness, increasing carrier gas linear velocity and oven ramp rate. This type of GC analysis, which is often termed "fast GC," utilizes narrow bore columns (<0.25mm ID) combined with thin films. Narrow bore columns offer a greater number of theoretical plates per meter than wider bore columns, and thus shorter lengths can be used while maintaining or improving the theoretical efficiency of the system. For example, comparing a 0.25mm ID column to a 0.10mm ID column with similar retention (k') and coating efficiency, the approximate plate numbers per meter are 2925 and 7300 respectively. For this reason, 0.10mm ID columns are a good choice for fast GC analysis.

Comparing 0.10mm ID with 0.25mm ID columns, the Van Deemter plots (plate height, H vs. linear velocity, μ) of 0.10mm ID columns have a higher μopt and a more shallow increase in H with increasing μ than 0.25mm ID columns. Consequently, a higher linear velocity can usually be used on these columns to shorten analysis time, without significantly affecting resolution. However, these columns require higher head pressures than 0.25mm ID columns to establish the same linear velocity. This limits the length of column that is practical for use in a conventional GC system. Helium, the most commonly used carrier gas, requires a head pressure of 55psi to achieve a linear velocity of 30cm/sec at 100°C on a 15m x 0.10mm ID x 0.10μm column. In comparison, the same length column with a 0.25mm ID requires only a 7.8psi head pressure to achieve the same linear velocity.

Hydrogen, an alternative carrier gas choice, has a lower viscosity than helium and will not require as high of a head pressure to achieve the same linear velocity. A 15m x 0.10mm ID x 0.10μm column run with hydrogen carrier would only require a head pressure of 25psi to achieve 30cm/sec at 100°C.

Another benefit of hydrogen carrier gas over helium is its higher diffusivity. The Golay theory for open tubular columns predicts that optimum gas velocity is proportional to diffusivity. This means that the optimum linear velocity for hydrogen will be higher than helium. In addition, the Van Deemter plot for hydrogen is flatter than that of helium, with the minimum H for hydrogen achieved at a higher average gas velocity than helium. The result is that using hydrogen carrier at a linear velocity near or just above its μopt will result in a faster run time without a significant loss in resolution.

Fast oven ramp rates are also essential in decreasing run time, and all hold times during the temperature program should be as short as possible. The various GC systems available have different capabilities with regards to oven ramping. Before developing a fast GC method, it is advisable to check with the manufacturer of your GC to find out the temperature ramping capability of your system.

In this work, we compared the use of a 30m x 0.25mm ID x 0.25μm to a 15m x 0.10mm ID x

0.10µm Equity-5 for two common applications, organochlorine pesticides and PCBs. The run conditions established using the 0.10mm ID Equity-5 reduced the run time by 75%. **Figure A** illustrates the separation of 20 organochlorine pesticides and 2 surrogates on the 30m x 0.25mm ID x 0.25µm Equity-5. **Figure B** illustrates the same compound list on a 15m x 0.10mm ID x 0.10µm Equity-5 column. The total run time decreased from 32 to 6.5 minutes. Hydrogen carrier in constant flow mode and rapid oven ramp rates with no hold at the initial oven temperature were necessary to decrease the run time to this level. A 4mm ID liner was used to accommodate the expansion volume of the 2µL injection. One coelution was noted, endosulfan I and a-chlordane. Labs typically do this application as a dual column analysis with a second column of different selectivity. Since this secondary column would probably resolve this pair, the tradeoff in resolution in this case could be acceptable for a 75% decrease in run time. After converting a conventional method to a "fast" method, the elution order of the analytes should always be verified. In this case, there was a change in elution order between endosulfan sulfate/DDT and endrin ketone/methoxychlor.

Column:	Equity-5, 30m x 0.25mm ID, 0.25µm
Cat. No.:	28089-U
Oven:	100°C (2 min), 15°C/min to 160°C, 5°C/min to 300°C (10 min)
Inj.:	225°C
Det.:	ECD, 310°C
Carrier Gas:	Helium, 30cm/sec @ 100°C
Injection:	2µL, splitless (0.5 min)
Liner:	4mm ID double taper
Sample:	50ppb of a 22 component chlorinated pesticide standard (Cat. No. 46845-U)

Compounds:

1. 2,4,5,6-Tetrachloro-m-xylene (surr.)
2. a-BHC
3. b-BHC
4. g-BHC
5. d-BHC
6. Heptachlor
7. Aldrin
8. Heptachlor epoxide
9. g-BHC
10. Endosulfan I
11. a-Chlordane
12. 4,4'-DDE
13. Dieldrin
14. Endrin
15. Endosulfan II
16. 4,4'-DDD
17. Endrin aldehyde
18. Endosulfan sulfate
19. 4,4'-DDT
20. Endrin ketone
21. Methoxychlor
22. Decachorobiphenyl (surr.)

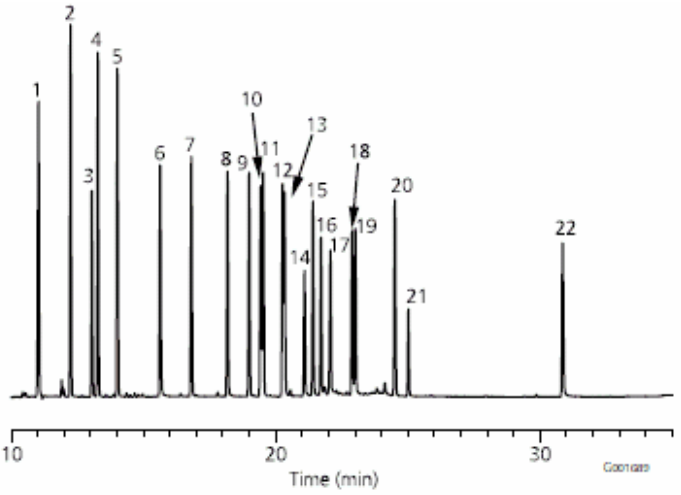

Figure A. Organochlorine Pesticides on the Equity-5, 30m x 0.25mm ID, 0.25µm

Column:	Equity-5, 15m x 0.10mm ID, 0.10µm
Cat. No.:	28083-U
Oven:	100°C (0 min), 50°C/min to 200°C (0 min), 35°C/min to 360°C (1 min)
Inj.:	225°C
Det.:	ECD, 360°C
Carrier Gas:	Hydrogen, 30cm/sec constant
Injection:	2µL, splitless (0.75 min)
Liner:	4mm ID single taper
Sample:	50ppb of a 22 component chlorinated pesticide standard (Cat. No. 46845-U)

Compounds:

1. 2,4,5,6-Tetrachloro-m-xylene (surr.)
2. a-BHC
3. b-BHC
4. g-BHC
5. d-BHC
6. Heptachlor
7. Aldrin
8. Heptachlor epoxide
9. g-Chlordane
10. Endosulfan I
11. a-Chlordane
12. 4,4'-DDE
13. Dieldrin
14. Endrin
15. Endosulfan II
16. 4,4'-DDD
17. Endrin aldehyde
18. Endosulfan sulfate
19. 4,4'-DDT
20. Endrin ketone
21. Methoxychlor
22. Decachorobiphenyl (surr.)

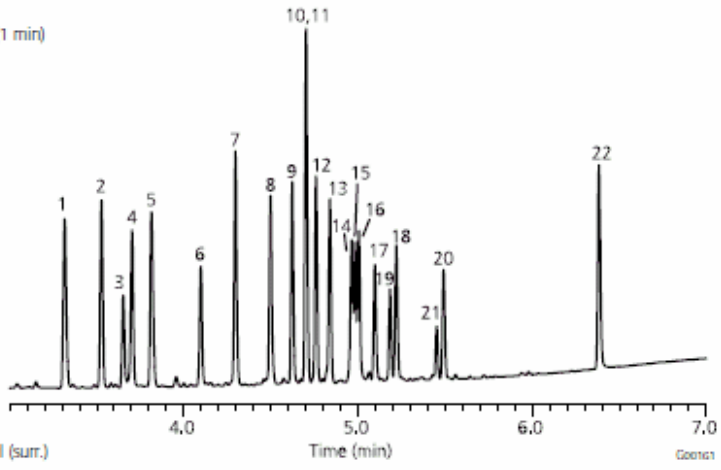

Figure B. Organochlorine Pesticides on the Equity-5, 15m x 0.10mm ID, 0.10µm

In the case of the PCB analysis, the time savings were similar to the pesticides. A chromatogram of a mixture of Aroclors® 1016 and 1260 on a 30m x 0.25mm ID x 0.25µm Equity-5 column is presented in **Figure C**. Analysis times of 38-45 minutes are not uncommon for this application. If the same mix is rerun on a 15m x 0.10mm ID x 0.10µm Equity-5 with the same run conditions used for the pesticide analysis (**Figure D**), the analysis can be done in under 8 minutes. Even with the significantly reduced run time; resolution is sufficient to provide excellent pattern recognition of both Aroclors.

Column:	Equity-5, 30m x 0.25mm ID, 0.25µm
Cat. No.:	28089-U
Oven:	100°C (2 min), 15°C/min to 160°C, 5°C/min to 300°C (10 min)
Inj.:	225°C
Det.:	ECD, 310°C
Carrier Gas:	Helium, 30cm/sec @ 100°C
Injection:	2.0µL, splitless (0.5 min)
Liner:	4mm ID double taper
Sample:	Aroclor Mix 1 standard at 75ppb with surrogates at 7.5ppb Cat. No. 46846-U
Compounds:	1. 2,4,5,6-Tetrachloro-m-xylene (surr.)
	2. Aroclor 1016
	3. Aroclor 1260
	4. Decachlorobiphenyl (surr.)

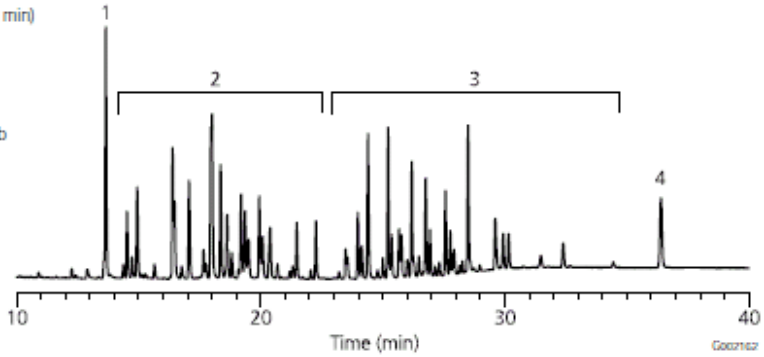

Figure C. Aroclors 1016 and 1260 on the Equity-5, 30m x 0.25mm ID, 0.25µm

Figure D. Aroclors 1016 and 1260 on the Equity-5, 15m x 0.10mm ID, 0.10µm

Column:	Equity-5, 30m x 0.25mm ID, 0.25µm
Cat. No.:	28083-U
Oven:	100°C (0 min), 50°C/min to 200°C (0 min), 35°C/min to 360°C (1 min)
Inj.:	225°C
Det.:	ECD, 360°C
Carrier Gas:	Hydrogen, 30cm/sec constant
Injection:	2µL, splitless (0.75 min)
Liner:	4mm ID single taper
Sample:	200ppb of Aroclors 1016 & 1260 with surrogates at 20ppb (Cat. No. 46846-U)
Compounds:	1. 2,4,5,6-Tetrachloro-m-xylene (surr.)
	2. Aroclor 1016
	3. Aroclor 1260
	4. Decachlorobiphenyl (surr.)

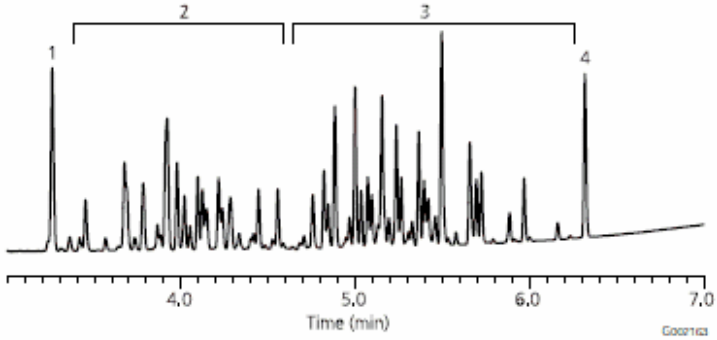

Figure D. Aroclors 1016 and 1260 on the Equity-5, 15m x 0.10mm ID, 0.10µm

We have seen here that reducing column length and ID, increasing oven ramp rates and using hydrogen as a carrier gas can significantly reduce GC run time. The 15m x 0.10mm ID x 0.10µm Equity-5 can be used for such a purpose. If you are in need of increasing GC sample throughput in your laboratory, consider letting us help you convert your current method to a faster version.

For further assistance, please contact technical service at uktechsv@europe.sial.com or call 0800 424342 / 814-359-3041.