Faster Gas Chromatographic Applications with 0.18 mm ID High-Efficiency Capillary GC Columns

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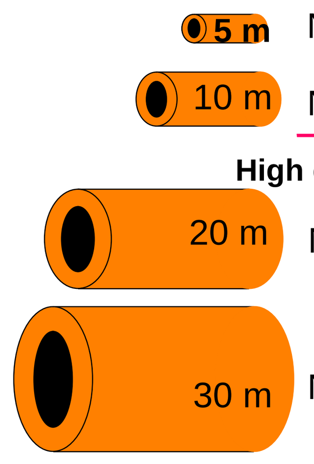
Introduction

The term high-efficiency GC refers to columns with 0.18 mm internal diameters. These columns have more theoretical plates per meter (N/m) than standard bore (0.25 mm ID) capillary columns. The typical N/m value for 0.18 mm ID columns is 6600 or more and 4700 or less for 0.25 mm ID columns at a retention 5 times k (void time). By convention the term high-efficiency GC indicates columns in the more efficient 0.18 mm ID format.

Column Diameter - Theoretical Efficiency



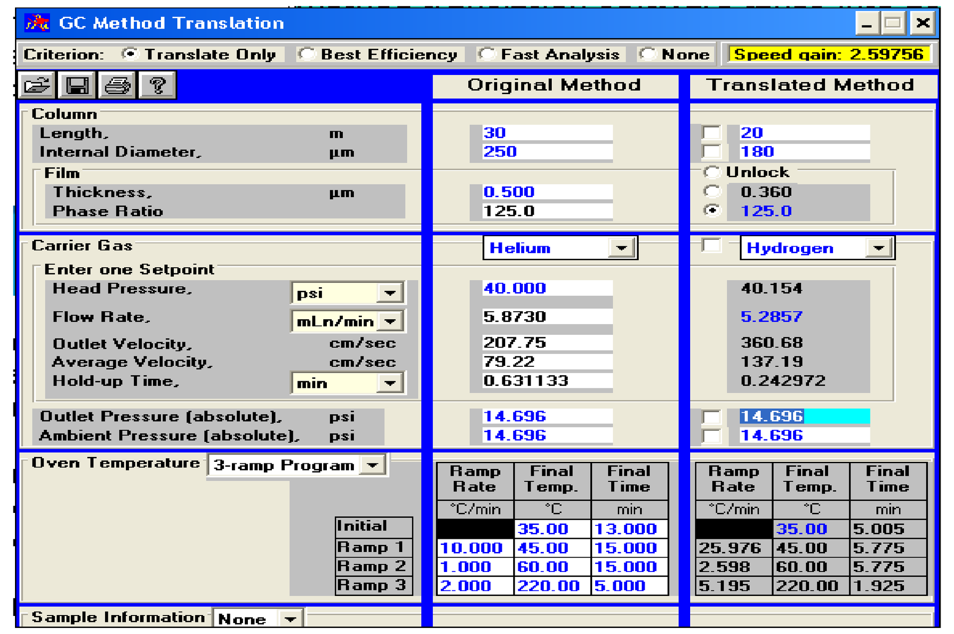
	Total Plates	I.D. (mm)	N/m
5 m	N ~ 112,000	0.05	23,160
10 m	N ~ 112,000	0.10	11,580
High efficiency GC		0.18	6,660
20 m	N ~ 112,000	0.20	5830
		0.25	4630
30 m	N ~ 112,000	0.32	3660
		0.45	2840
		0.53	2060

k = 5

There are many misconceptions about what it means to perform fast gas chromatography (GC) and what the term fast GC implies. Fast GC is often associated with the use of hydrogen as a carrier gas, and although this is certainly a good approach, it is not always necessary in order to shorten the analysis time. A second misconception is that changing column dimensions results in time consuming method development. Utilizing high-efficiency GC columns can greatly reduce the analysis time, and when coupled with the Method Translation software, the time spent on method development can be kept to a bare minimum.

High-efficiency columns are designed to maintain the same phase ratio as the more commonly used 0.25 mm columns, making for easy method translation, as will be illustrated.

Method Translation



Method Translation Software Input Screen

Easy Options with Method Development Software

-Different Column Dimensions

-Same Column & Gas Type but Faster Velocities

-Switch He to H_2 Carrier Gas and Try Faster Velocities

-Combination of all of the above

- **Four translation modes**

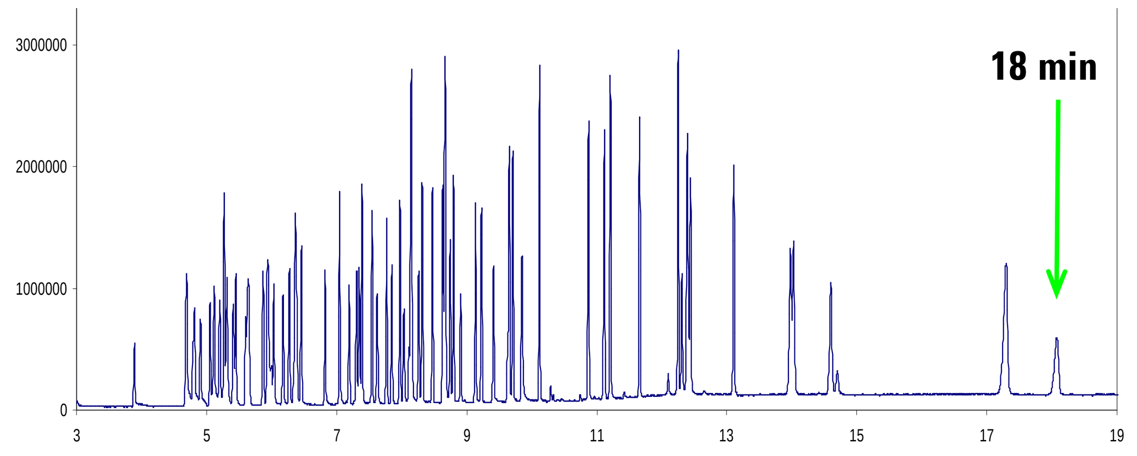
  – Translation only
  – Best efficiency
  – Fast analysis

  } Lock all carrier gas parameters, making the flow rate an independent parameter.

  – None (unlock all carrier gas parameters)

•If translating to a different ID column, phase ratio should be maintained for the most reliable results.
•If there are significant differences in phase ratio, Method translations Software can still be used but elution order should be confirmed.
•Stationary phase of a new column must be the same as the original – the Method Translation software cannot account for differences in selectivity.

Semi-volatiles



DB-5.625
20 m x 0.18 mm I.D. x 0.36 µm
Part no: 121-5622

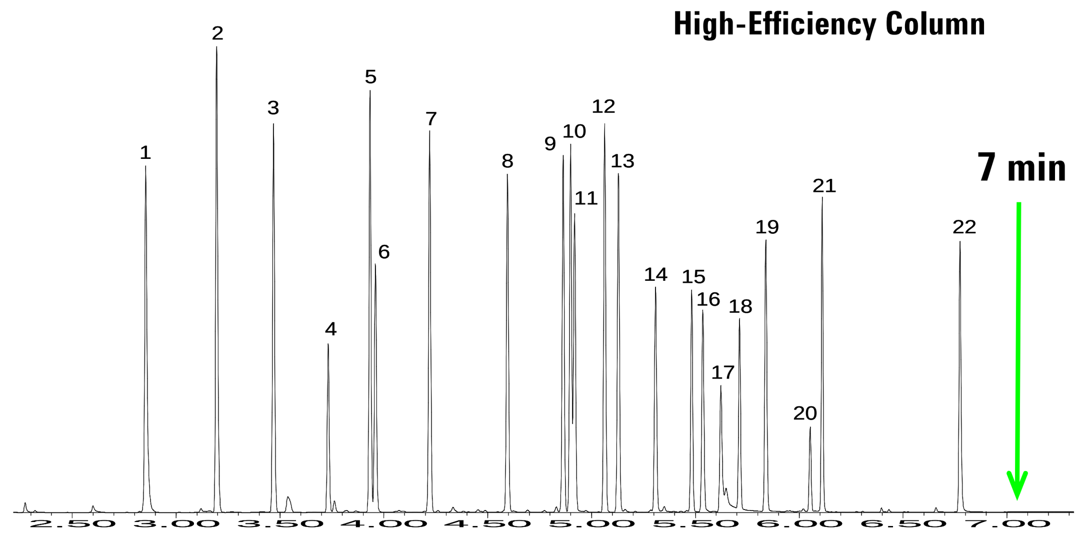
Carrier:	He@ 1.0 mL/min
Oven:	55°C for 1.00 minutes 55 – 320 @ 25 °C
Injector:	Pulsed Splitless, 250°C Pressure pulse 40.0 psi for 0.20 min 0.5 µl in DCM
Detector:	MSD

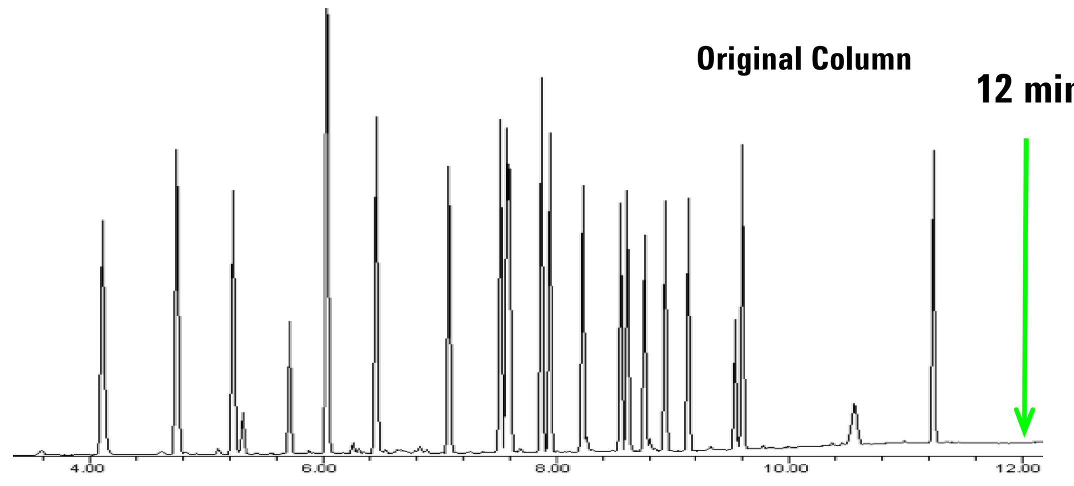
The analysis of semi-volatile organic compounds according to the criterion established in the US EPA Method 8270 is demanding, both on the instrumentation and the laboratory. Finding ways to improve sample throughput without sacrificing the analysis integrity is a significant challenge. The analysis consists of anywhere from 70 – 200 or more acids, bases, and neutrals, many exhibiting high activity. Sample matrices are typically very dirty.

A high-efficiency DB-5.625 column is employed in this study. This column undergoes a unique deactivation process which produces superior chromatography for even the most active components. Along with a direct connect deactivated liner, the splitless GC method uses a pulsed injection technique which aids in a quick sample transfer onto the column thereby shortening the residence time of the sample in the inlet. The shorter residence time has the effect of increasing the recovery of labile constituents and reducing tailing.

Demonstrated here is a set of method parameters wherein a laboratory following current EPA Method 8270 protocol, can reasonably increase sample throughput by one-third using a highly inert quadrapole MSD. This speed increase was accomplished by using high-efficiency GC columns, optimizing temperature conditions/sample introduction parameters and implementing key maintenance steps. This method is robust and yields more billable samples per hour than a standard bore capillary approach.

CLP Pesticides





High-Efficiency Column

Column:	DB-XLB 20m x 0.18mm i.d., 0.18µm
Carrier:	H2, constant flow, 77.3cm/s at 120 °C
Injector:	Pulsed Splittless, 220 °C Pulse pressure & time: 35psi for 0.5min Flow ramp at 6.25min of 99mL/min^2 to 3mL/min 2mm i.d. liner 0.5µL, 50ppb
Oven:	120°C for 0.49min 120°C to 160°C at 59.4°/min 160°C to 260°C at 23.7°/min 260°C to 300°C (1.69min) at 35.6°/min
Detector:	µ-ECD, 320°C Ar/CH4 (P5) makeup gas at 60mL/min

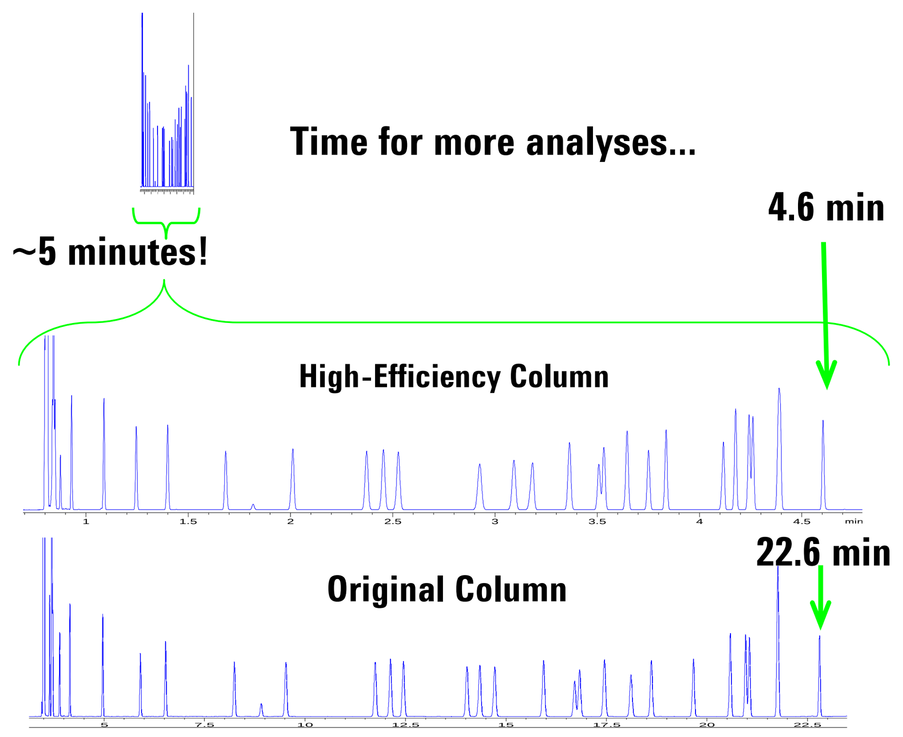
Original Column

Column:	DB-XLB 30m x 0.32mm i.d., 0.25µm
Carrier:	H_2, constant flow, 38 cm/s at 120°C
Injector:	Pulsed Splittless, 220 °C Pulse pressure & time: 35psi for 1.15min 2µL, 50ppb
Oven:	120°C for 1.17min 120°C to 160°C at 25°/min 160°C to 260°C at 10°/min 260°C to 300°C (4min) at 15°/min
Detector:	µ-ECD, 320°C Ar/CH4 (P5) makeup gas at 60mL/min

CLP Pesticide Standard Key List

1. TCMX	12. 4,4' DDE
2. Alpha BHC	13. Dieldrin
3. Gamma BHC	14. Endrin
4. Beta BHC	15. 4,4' DDD
5. Delta BHC	16. Endosulfan II
6. Heptachlor	17. 4,4' DDT
7. Aldrin	18. Endrin Aldehyde
8. Heptachlor Epoxide	19. Endosulfan Sulfate
9. Gamma Chlordane	20. Methoxychlor
10. Alpha Chlordane	21. Endrin Ketone
11. Endosulfan I	22. DCB

Aromatic Solvents



High-Efficiency Column

GC:	Agilent 7890
Oven:	70° (3 min),45° C/min to 145° C (1 min)
Injection:	0.2 to 1 µl, 100:1 to 600:1 split 250°C,
Carrier:	He 33 psi constant pressure mode
Column:	HP-INNOWax 20 m x 0.18 mm x0.18 µm
Detection:	FID 250°C

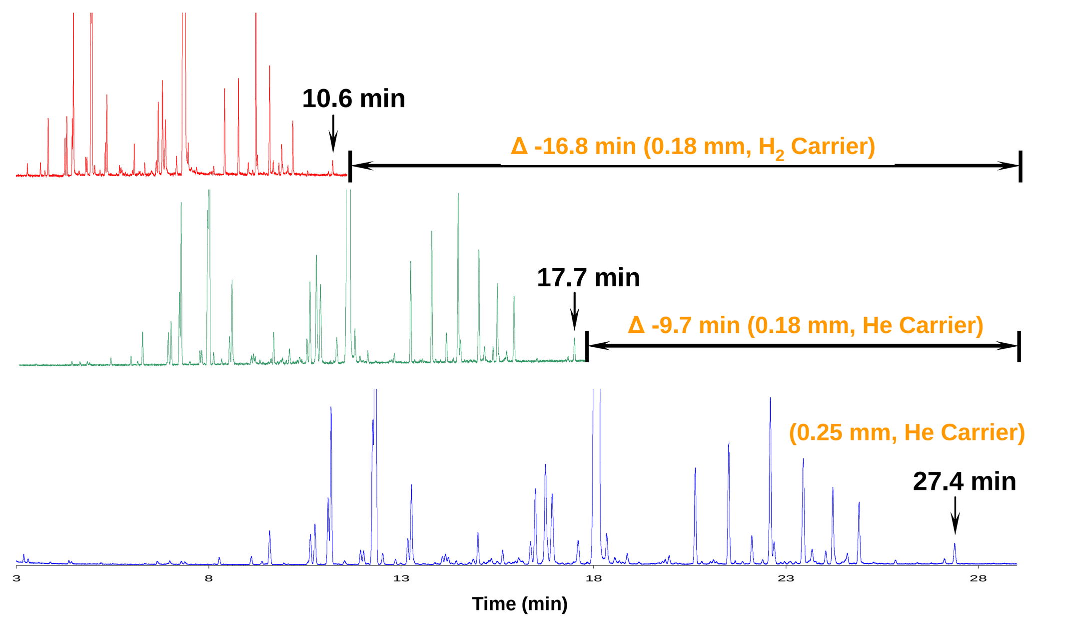
Original Column

GC:	Agilent 7890
Oven:	75° (10.0 min),3° C/min to 100° C, then 10° C/min to 145° C
Injection:	1 µl 100:1 split 250°C,
Carrier:	He 20 psi constant pressure mode
Column:	HP-INNOWax 60 m x 0.32 mm x 0.50 µm
Detection:	FID 250°C

Purity determination of aromatic hydrocarbons is critical for many QA and QC laboratories in the chemical and petrochemical industries. In an effort to standardize analysis procedures, the American Society of Testing and Materials (Such as ASTM D-5060 and D-3760) has developed and published a number of GC methods specifically for an aromatic compound or a class of aromatic compounds such as styrene, *o*-Xylene, *p*-Xylene, and ethylbenzene.

A high-efficiency HP-INNOWax column and Method Translation software in "fast analysis" mode were used to speed up this aromatic solvent analysis.

Food and Fragrances



Speed gain demonstrated for Spearmint Oil with use of high-efficiency GC column and hydrogen carrier.

Compound Resolution			
Compounds	**0.25 mm Helium**	**0.18 mm Helium**	**0.18 mm Hydrogen**
Sabinene **β-Pinene**	**1.52**	**1.59**	**1.56**
α-Terpinene ***p*-Cymene**	**1.61**	**1.73**	**1.86**
Speed Gain	**N/A**	**35%**	**61%**

Conclusions

The use of high efficiency columns have many benefits as illustrated here. Shorter analysis times were achieved without significant loss of resolution. Time spent in method development was kept to a minimum through the use of the GC Method Translation Software and phase ratio matching. While there are additional benefits to using hydrogen as the carrier gas, significant speed gain can be realized by simply using high efficiency columns while maintaining helium as the carrier.

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