Fast GC Using 100µm ID Capillary Columns

L.M. Sidisky, K. K. Stenerson, J.L. Desorcie, and G. A. Baney
Supelco, 595 North Harrison Road, Bellefonte, PA 16823, USA

Abstract

Speed of analysis has become an important parameter in many modern laboratories, as analysts look for faster methods to improve sample throughput. Approaches to improve the speed of analysis include shorter capillary columns, decreased column internal diameter, thinner stationary phase films, higher carrier gas linear velocities, and faster oven temperature programming rates. Many times, analysts change only one of these parameters and the change produces a loss of sample resolution. Our focus is on using 100µm ID capillary columns, in combination with higher carrier gas linear velocities and fast temperature programming rates. This reduces the analysis time, while maintaining sample resolution. Theoretical aspects of the performance of 100µm ID columns is presented, with a series of complex petrochemical and food & beverage applications. Applications show use of 100µm ID columns with changes in operating parameters, reducing analysis times, and maintaining sample resolution compared to standard dimension capillary columns.

Introduction

Narrow bore, high speed capillary columns offer important advantages over traditional 0.25mm ID columns, in terms of efficiency and analysis speed. The increased efficiency is an inherent advantage of the narrower bore capillary tubing. Table 1 shows typical plate numbers generated by capillary columns of various dimensions.

Table 1. Efficiency of Capillary Columns of Various ID

Column ID (mm)	Plate Number (N_{eff})	Plates/meter (N_{eff}/m)
0.10	219000	7300
0.18	121500	4050
0.20	109500	3650
0.25	87750	2925
0.32	69000	2300

Theoretical Values, calculated @ a k'=6.00 and 85% CE for a 30 meter long column.

As is the case in all chromatographic analyses, however there are tradeoffs. The drawbacks to using a 100µm ID column is reduced sample capacity and a limitation on maximum column length due to pressure requirements for operating the column. Sample capacities for 100µm ID columns are approximately 1 nanogram or less per component. Table 2 lists the typical head pressures required to operate 100µm ID columns of various lengths at various helium and hydrogen carrier gas linear velocities. Columns longer than 20 meters require fairly high carrier gas pressures which limit their applicability, especially for operating at fairly high carrier gas linear velocities.

Table 2. Carrier Gas Head Pressure for 100µm ID Columns

	Carrier Gas Linear Velocity							
	Helium				Hydrogen			
Column	20	30	40	50	40	50	60	70
Length	Head Pressure Required (psig)							
40 meters	115	177	239	302	111	141	171	201
20 meters	54	84	115	146	52	66	81	96
10 meters	25	39	54	69	25	31	38	45
5 meters	12	19	25	32	12	15	18	21

Values calculated @ 160ºC

Column length and internal diameter are only two of the analytical parameters that can be modified to provide fast GC. Other parameters to modify include the stationary phase, stationary phase film thickness, carrier gas, carrier gas linear velocity, analysis temperature and temperature programming rate. The effects of modifying these parameter are as follows:

Stationary phase - Changing the phase can result in a decrease in analysis time. Selectivity differences will affect resolution.

Stationary phase film thickness - Decreasing the film thickness will decrease the analysis time.

Carrier Gas - Nitrogen, helium and hydrogen are the typical carrier gases for GC. Hydrogen is the best choice for fast GC due to hydrogen's high diffusivity and high optimal carrier gas linear velocity.

Carrier Gas Linear Velocity - Increasing the carrier gas linear velocity will decrease the speed of analysis. Loss of resolution can occur if the speed is increased much higher than the optimal velocity for the carrier gas.

Analysis Temperature - Increasing the analysis temperature for an isothermal analysis will decrease analysis time and may result in a loss of resolution if the temperature increase is too high.

Temperature Program Rate - A faster temperature program rate will decrease the analysis time and may result in a loss of resolution. 100µm ID capillary columns typically require a program rate of 1.5 to 2 times faster than a wider bore column to retain their inherent efficiency.

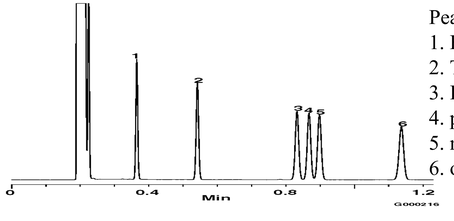
Applications

To demonstrate that the high efficiency of the 100µm ID columns provides good resolution of sample components, with a significant decrease in analysis time relative to traditional 250µm ID columns, we performed two petrochemical applications using 100µm capillary columns.

Two analyses demonstrate the capability of the 100µm ID columns. BTEX analysis typically requires a 30 meter x 0.25mm ID column to achieve baseline resolution of p- and m-xylene with an analysis time of about 15 minutes. The 5 meter SUPELCOWAX 10 column baseline resolves the para- and meta-xylene isomers in about 1.2 minutes.

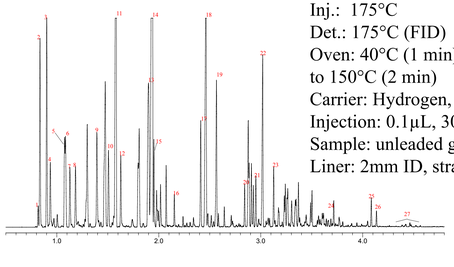
The analysis of gasoline is typically performed on long 100 meter columns and requires analysis times up to 2 hours. Similar results can be obtained using a 15 meter x 0.10mm ID x 0.10µm Equity-1 capillary column coupled with hydrogen carrier gas and a rapid temperature program rate of 45ºC/ minute.

BTEX Analysis on 5m x 0.10mm ID x 0.1µm SUPELCOWAX 10, 60°C Isothermal, Helium, 50cm/second



Peak List
1. Benzene
2. Toluene
3. Ethylbenzene
4. p-xylene
5. m-xylene
6. o-xylene

Petrochemical - Unleaded Gasoline



Equity-1, 15m x 0.10mm ID x 0.1µm
Cat. No.: 28039-U
Inj.: 175°C
Det.: 175°C (FID)
Oven: 40°C (1 min), 45°C/min to 150°C (2 min)
Carrier: Hydrogen, 45cm/sec, constant
Injection: 0.1µL, 300:1 split
Sample: unleaded gasoline neat
Liner: 2mm ID, straight

Unleaded Gasoline Peak List

1. Isobutane
2. Butane
3. Isopentane
4. Pentane
5. 2,3-Dimethylbutane
6. 2-Methylpentane
7. 3-Methylpentane
8. Hexane
9. Benzene
10. 3-Methylhexane
11. Isooctane
12. Heptane
13. 2,3,4-Trimethylpentane
14. Toluene
15. 2,3-Dimethylhexane
16. Octane
17. Ethylbenzene
18. m&p-Xylenes
19. o-Xylene
20. Propylbenzene
21. 1-Methyl-2-ethylbenzene
22. 1,2,4-Trimethylbenzene
23. 1,2,3-Trimethylbenzene
24. Naphthalene
25. 2-Methylnaphthalene
26. 1-Methylnaphthalene
27. Dimethylnaphthalenes

Summary

Fast GC can be performed using short 100µm ID capillary columns without sacrificing resolution compared to longer, wider bore capillary columns. The 100µm ID columns offer increased efficiency, which allows shorter columns to be used to perform many analyses. Shorter columns also typically require an increased carrier gas linear velocity to operate optimally, which reduces analysis times. The smaller columns do have a limited sample capacity, however, and require higher operating pressures, which can limit the column length for an analysis.

Other physical factors to modify in order to perform Fast GC include optimizing the stationary phase and stationary phase film thickness, carrier gas choice, and the analysis temperature and temperature programming rate.

Applications demonstrating the use of the 100µm ID columns for two petrochemical samples have been demonstrated. Additional applications have been performed with environmental and food & beverage samples and are available upon request.

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