Analysis of Essential Oils by Fast Capillary GC Using The Agilent 6890 Series GC

Application

Gas Chromatography

November 1995

Authors

F. David
Research Institute for Chromatography
Kennedypark 20, B-8500
Kortrijk, Belgium

P. Sandra
University of Gent, Krijgslaan 281, S4, B-9000 Gent, Belgium,
Eindhoven University of Technology, The Netherlands

Philip Wylie
Agilent Technologies, Inc.
Wilmington, DE 19808-1610
USA

Abstract

By reducing the internal diameter of the capillary column, a higher efficiency per unit of column length is obtained in capillary GC (CGC). The application of narrow bore columns, therefore, results in faster analyses compared to conventional columns while maintaining the resolution.

The use of narrow bore columns in routine analysis, however, make high demands on instrumentation. The 6890 Series GC offers electronic control of column flow and split flow, faster oven heating and faster electronics, making fast capillary GC accessible for routine applications. Method translation software is an additional tool to translate an existing operating procedure into a method using a narrow bore column. The performance of the 6890 Series GC in fast CGC is demonstrated by the analysis of some essential oils.

Introduction

The ultimate goal of a gas chromatographic method is to obtain complete resolution of a given mixture in the shortest possible analysis time. For trace analysis, sensitivity and sample loadability are additional requirements. In order to meet these goals, capillary columns with a length of 25 to 30 m and an internal diameter of 0.25 to 0.32 mm are most frequently used in routine applications. These capillary columns offer high efficiency (100,000 plates) and sufficient sample capacity and loadability. Decreasing the internal diameter results in an increase of the column efficiency and, therefore, the column length can be reduced while keeping the resolution constant[1-4]. A 10 m x 0.1 mm i.d. column, for instance, offers the same resolution as a 25 m x 0.25 mm i.d. column. Because the column is 2.5 times shorter, the analysis time is reduced drastically. Moreover, because the van Deemter curves are flatter for narrow bore columns and the optimum gas velocities are higher, the speed of analysis can further be increased without reducing the resolution. The price to pay for the gain in speed of analysis is the lower sample capacity, resulting in the practical consequence that only small volumes can be injected. In practice, split injection with a high split ratio is used. Many applications in chemical, petrochemical, food and, flavor and fragrance analysis, however, do not need ultimate sensitivity and for these applications the use of narrow bore columns can offer an important reduction in analysis time and, consequently a higher sample throughput, while maintaining resolution.

Until now, the major obstacle for the implementation of narrow bore columns in routine CGC were instrumental requirements. The use of narrow bore columns requires high inlet pressures, better split flow control, fast oven temperature heating rates and fast electronics. With the introduction of the 6890 Series GC a major step was made in meeting these GC requirements.

The 6890 Series GC indeed offers:

High inlet pressures: The maximum inlet pressure is 150 psi (1030 kPa).

This pressure is high enough for 0.1 mm i.d. columns up to 50 m long and for 0.05 mm i.d. columns up to 15 m long when using hydrogen carrier gas.

Electronic pneumatic control of carrier and detector gases: Both the column head pressure and the split flow are controlled electronically from the keyboard or the ChemStation. This results in excellent repeatibility of flow settings.

Gas saver mode: Due to the high split ratio used, a very high flow of carrier gas exits the split vent. Because this high split vent flow is only needed during injection, the gas saver mode can be activated after injection to reduce the split flow for reducing gas consumption and for safety reasons when hydrogen is used as the carrier gas.

Fast oven heating: The 6890 Series GC allows oven heating rates up to 120°C/min. Heating rates of 50°C/min or more are sometimes necessary in order to fully exploit the speed gain that can be obtained using narrow bore columns.

Fast electronics: A data sampling rate up to 200 Hz makes signal acquisition of very sharp and fast eluting peaks possible.

Using narrow bore columns, different operational conditions are used. Because little information is yet available on the use of narrow bore columns, the transfer of standard operating procedures for conventional capillary columns into operating procedures for narrow bore columns might be difficult. In this respect, the development of method translation software[5] is very helpful for translating a standard operating procedure developed for a standard column to an operating procedure for a narrow bore column. After performing the analyses on the standard column, the optimized conditions are introduced in the program and all operational conditions for the narrow bore column are calculated in order to obtain the same resolution. The gain in analysis time is also predicted.

In this application note, the performance of the 6890 Series GC for fast CGC of essential oils is illustrated.

Experimental

Samples

Two samples were analyzed. Sample A is an artificial mixture of flavor compounds used for perfuming soaps and detergents. The sample was diluted to 1% in dichloromethane. Sample B is a natural lavender oil, also diluted to 1% in dichloromethane.

Analytical Conditions

The analyses were performed on a 6890 Series GC. Automated split injection was done using a 7673 autosampler. The instrumental configuration and analytical conditions are summarized in Table 1. First the analyses were performed on a 30 m x 0.25 mm i.d. x 0.25 µm HP-5 standard capillary column.

The conditions applied with this column are listed in experimental conditions A. The same analyses were then performed on a 15 m x 0.1 mm i.d. x 0.1 µm HP-5 narrow bore column. The conditions applied with this column were calculated by the method translation software and are listed in experimental conditions B. From the method translation software, a speed gain by a factor 2.4 was predicted.

Table 1. Instrumentation

Chromatographic System	
Gas chromatograph	6890 Series
Inlet	Split/splitless
Detector	FID
Automatic sampler	7673
Liner	Split/splitless liner, 4 mm i.d. with glass wool plug, (part no. 19251-60540)
Data handling	ChemStation (DOS Series)
Column A	30 m x 0.32 mm i.d. x 0.25 µm H-5
Column B	15 m x 0.10 mm i.d. x 0.1 µm H-5
Experimental Conditions A	
Inlet temperature	250°C
Injection volume	1 µL
Split ratio	1/50
Carrier gas	Hydrogen
Head pressure	6 psi
Flow/velocity	1 mL/min-35 cm/s
Split flow	49 mL/min
Gas saver	OFF
Oven temperature	40°C, 5°C/min to 190°C
Detector temperature	250°C
Detector gases.	Hydrogen: 35 mL/min; Air: 400 mL/min; Helium: 30 mL/min
Experimental Conditions B	
Inlet temperature	250°C
Injection volume	1 µL
Split ratio	1/530
Carrier gas	Hydrogen
Head pressure	29 psi
Flow/velocity	1.8 mL/min-88 cm/s
Split flow	192 mL/min
Gas saver	ON at 1 min
Oven temperature	40°C, 12°C/min to 190°C
Detector temperature	250°C
Detector gases	Hydrogen: 35 mL/min; Air: 400 mL/min; Helium: 30 mL/min

Results and Conditions

The analysis of the artificial flavor sample is shown in Figure 1. A good separation is obtained in an analysis time of 30 minutes.

The same analysis was then performed on the narrow bore column, applying the operation conditions calculated by the method translation software. The resulting chromatogram is shown in Figure 2. By comparing both figures, it is obvious that practically the same resolution is obtained. Using the narrow bore column, the analysis time is only 12.5 minutes, perfectly corresponding with the predicted speed gain by a factor 2.4.

Figure 3. Analysis of lavender oil on a 15 m x 0.10 mm i.d. x 0.1 µm HP-5 column.

Using the same analytical conditions, a solution of a natural lavender oil was analyzed. The chromatogram is shown in Figure 3. The main peaks in this essential oil correspond to linalool, camphor, linalyl acetate and geranyl acetate. For these four compounds, the retention time and peak area reproducibility was calculated for 10 runs. The results are given in Tables 2 and 3. The standard deviation on the retention times was smaller than 0.002 minutes (RSD < 0.03) The relative standard deviation on peak areas was better than 1 %.

Conclusion

The Agilent 6890 Series GC offers excellent potentials for fast capillary GC. The electronic pneumatic control of the column head pressure and the split flow, the gas saving option, the fast and reproducible oven heating and the fast electronics, result in high reproducibility of retention times and peak areas. Using the method translation software, an existing operating procedure applying a standard capillary column can be translated into an operating procedure for a narrow bore column, resulting in a faster analysis while maintaining resolution.

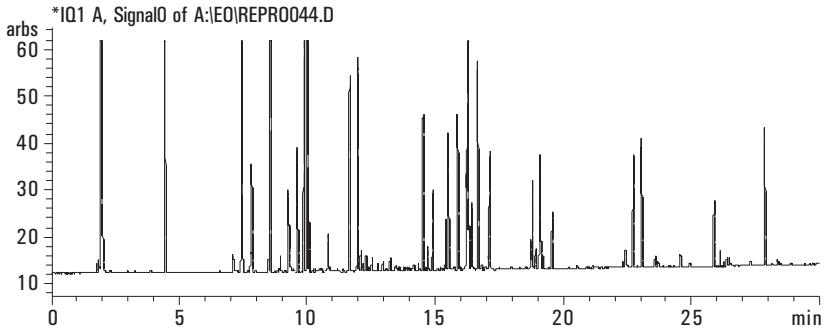

Figure 1. Analysis of an artificial flavor sample on a 30 m x 0.25 mm i.d. x 0.25 µm HP-5 column.

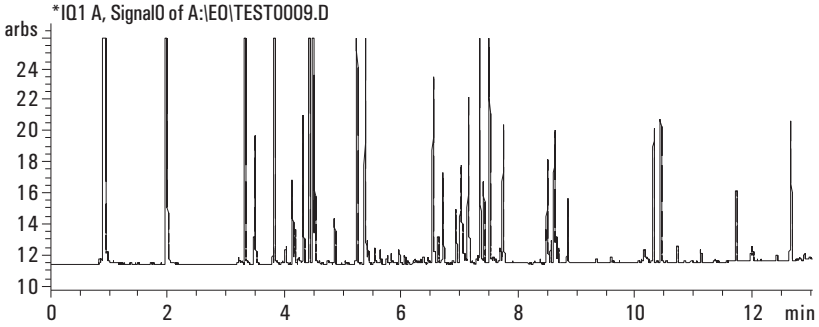

Figure 2. Analysis of an artificial flavor sample on a 15 m x 0.10 mm i.d. x 0.1 µm HP-5 column.

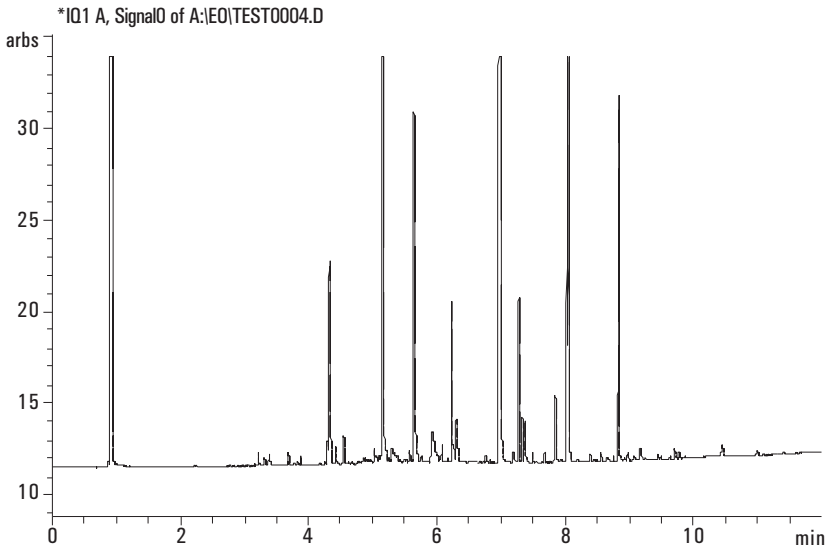

Figure 3. Analysis of lavender oil on a 15 m x 0.10 mm i.d. x 0.1 µm HP-5 column.

Table 2. Peak Area Precision for the Analysis of Lavender Oil Using a 15 m x 0.10 mm i.d. x 0.1 µm HP-5 Column

Run	Peak Linalool	Camphor	Linalylacetate	Geranylacetate
1		23.80851	103.47838	87.62983
2	115.93026	23.98765	103.49316	87.55368
3	117.01113	24.19413	104.4487	88.41127
4	116.82664	24.10895	104.15176	88.263
5	117.13489	23.91479	104.39146	88.3009
6	117.0449	24.00706	104.41306	88.30447
7	118.30569	24.23741	105.43989	89.23877
8	117.31309	24.34547	104.68652	88.69226
9	118.18525	24.18469	105.25503	89.21329
10	118.06036	24.06571	105.05595	89.24325
MEAN	117.16897	24.085437	104.481391	88.485072
S	0.849920602	0.161532422	0.666212109	0.617913328
RSD (%)	0.725	0.671	0.638	0.698

Table 3. Retention Time Precision for the Analysis of Lavender Oil Using a 15 m x 0.10 mm i.d. x 0.1 µm HP-5 Column

Run	Peak Linalool	Camphor	Linalylacetate	Geranylacetate
1	5.112	5.592	6.925	7.96
2	5.113	5.592	6.924	7.959
3	5.114	5.591	6.925	7.96
4	5.114	5.593	6.924	7.96
5	5.114	5.593	6.924	7.959
6	5.112	5.591	6.924	7.959
7	5.114	5.593	6.925	7.96
8	5.114	5.593	6.924	7.961
9	5.114	5.593	6.927	7.963
10	5.114	5.593	6.927	7.965
MEAN	5.1135	5.5924	6.9249	7.9606
S	0.000849837	0.000843274	0.001197219	0.00195505
RSD (%)	0.0166	0.0151	0.0173	0.0246

References

1. P. Sandra and M. Proot, HRC 9 (1986) 618.

2. P. Sandra, LC.GC Magazine 5 (1987) 236.

3. P. Sandra, M. Proot, G. Diricks and F. David, in P. Sandra and C. Bicchi (Eds), Capillary Gas Chromatography in Essential Oil Analysis, Huthig Verlag, Heidelberg, 1987, pp. 29-83.

4. F. Lancas, F. David and P. Sandra, HRC 11 (1988) 73.

5. W. D. Snyder and L. Blumberg, Proc. 14th Int. Symp. on Cap. Chrom., P. Sandra and M. L. Lee (Eds.), Baltimore, USA, May 1992.

Printed in the USA 3/2000
5964-3523E

Agilent Technologies
Innovating the HP Way