Application Note

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Reducing Analysis Time of 8270D with the Intuvo 9000 GC

Technology Advantage: Agilent Intuvo 9000 GC with an Agilent 5977A MSD

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Abstract

Requirements and methods for the quantitation of semivolatile organic compounds (SVOCs) are described in method 8270D produced by the Environmental Protection Agency (EPA). This Application Note presents GC method translation from one developed for EPA 8270D, using a 30 m × 0.25 mm column, to a faster method using a conductively heated 20 m × 0.18 mm column on an Agilent Intuvo 9000 GC.

Introduction

EPA method 8270D provides procedures and requirements for the quantitation of SVOCs extracted from solid waste, soil, water, or air by GC/MS[1]. Typically, 30 m capillary columns with 0.25 mm internal diameters (30 m × 0.25 mm) are used with standard convective column heaters. With careful optimization, run times of less than 20 minutes can be achieved. Efforts to further reduce run time have spurred some laboratories to investigate the use of columns with smaller internal diameters to improve separation kinetics and conductive column heating to provide increased temperature programing rates and faster cooling. This Application Note demonstrates translation of a GC method developed for EPA 8270D on a 30 m × 0.25 mm column to a fast method using a conductively heated 20 m × 0.18 mm column on an Intuvo 9000 GC.

Experimental

Sample

The sample was a mixture of 110 acids, bases, and neutrals at 50 µg/mL, and six internal standards at 40 µg/mL in dichloromethane.

Instrument methods

Table 1. Instrument methods.

	Original method	Translated method
GC	Intuvo 9000 GC with simple MS flowpath and split/splitless Guard Chip (G4587-60565)	Intuvo 9000 GC with simple MS flowpath and split/splitless Guard Chip (G4587-60565)
MS	Agilent 5977A MSD with EI inert source and 6 mm drawout plate	5977A MSD with EI inert source and 6 mm drawout plate
Column	Agilent J&W DB-UI 8270D Intuvo GC column 30 m × 0.25 mm, 0.25 µm (p/n 122-9732-INT)	J&W DB-5ms UI Intuvo GC column 20 m × 0.18 mm, 0.18 µm (p/n 121-5522UI-INT)
Liner	Ultra Inert, splitless, single-taper liner with glass wool (p/n 5190-2293)	Ultra Inert, split, low pressure drop with glass wool (p/n 5190-2295)
Injection volume	1 µL	1 µL
Inlet	Splitless 250 °C Purge 50 mL/min at 0.2 minutes Septum purge switched flow mode 3 mL/min	Split 250 °C Split ratio 10:1 Septum purge 3 mL/min
Column pressure/flow	7 psi for 0 minutes 90 psi/min to 30 psi for 0.1 minutes 99 psi/min to 13 psi for 2.6 minutes 1.5 psi/min to 34 psi	1.2 mL/min
Guard Chip temperature program	45 °C for 0.5 minutes 20 °C/min to 100 °C 25 °C/min to 270 °C 10 °C/min to 310 °C	200 °C for 0.4 minutes 25 °C/min to 100 °C 32 °C/min to 270 °C 12.5 °C/min to 310 °C
Column temperature program	45 °C for 0.5 minutes 20 °C/min to 100 °C 25 °C/min to 270 °C 10 °C/min to 310 °C for 2.45 minutes	45 °C for 0.4 minutes 25 °C/min to 100 °C 32 °C/min to 270 °C 12.5 °C/min to 310 °C for 1.95 minutes
Bus temperature	310 °C	310 °C
Transfer line temperature	300 °C	320 °C
Ion source temperature	300 °C	350 °C
Quadrupole temperature	150 °C	200 °C

Results and Discussion

The original method was optimized on a 30 m column with a 0.25 mm id with a 0.25 µm film thickness. To achieve the desired resolution of all the target compounds, the method relied on four temperature and four pressure-programing ramps (Table 1). To increase the speed of analysis, the method was translated to a narrow bore column (20 m × 0.18 mm, 0.18 µm) with the same phase ratio (β = 250) using the Agilent method translator software. As a first pass, the flow rate in the original method was set to a constant 1.5 mL/min, and translated to the narrow bore dimensions with the best efficiency radio button selected. This resulted in the translated temperature program listed in Table 1 and a translated flow rate of 0.72 mL/min. However, to further reduce the run time while meeting the resolution requirements of EPA 8270D, a flow rate of 1.2 mL/min was applied in the final translated method.

Since the loading capacity of the narrow column was lower, compared to the original column, the inlet was operated in split mode with a ratio of 10:1 to reduce the amount of material on-column. This allowed the same standard set used in the original method to be used in the translated method. For most compounds, the calibration ranged from 0.5 to 120 µg/mL.

Figure 1 shows the separation of SVOCs using the original method and translated method. Based upon the elution time of the last target, benzo[ghi]perylene, a speed enhancement of an approximate factor of 1.4 was achieved with the narrow column.

According to EPA 8270D, isomers must be resolved to a maximum value of 50 % as calculated by the ratio of the valley height between the isomers to the average of the two height maxima. Figure 2 shows an extracted ion chromatogram (*m/z* 252) of benzo[b]fluoranthene and benzo[k]fluoranthene. The calculated resolution between the isomers was 33.5 %, which was within method specification.

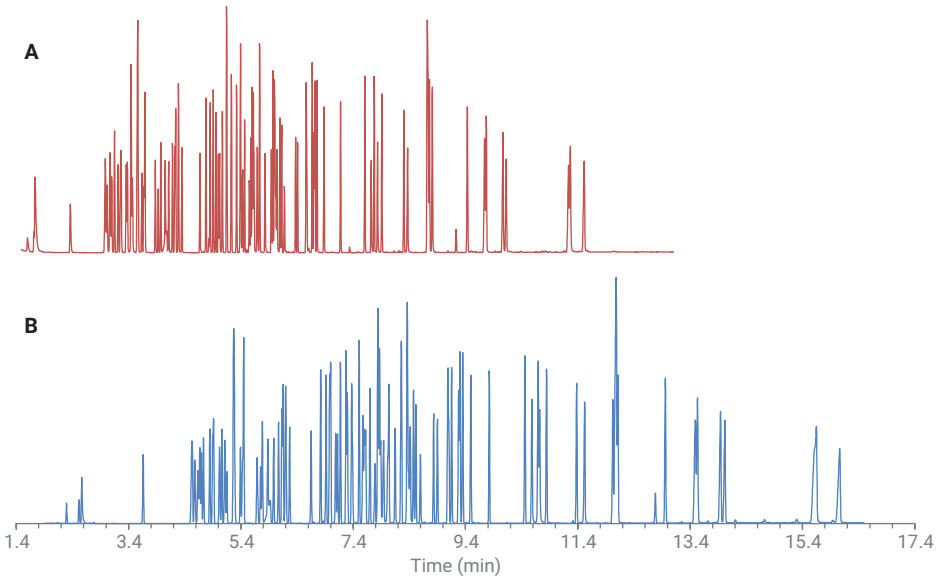

Figure 1. Separation of SVOCs using fast (A) and original (B) methods.

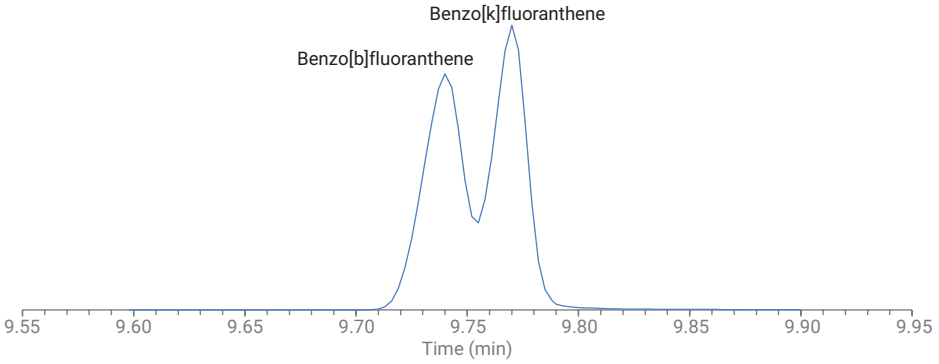

Figure 2. Extracted ion chromatogram (*m/z* 252) of benzo[b]fluoranthene and benzo[k]fluoranthene using the fast method.

In addition to the resolution requirement for benzo[b]fluoranthene and benzo[k]fluoranthene, some laboratories apply a resolution limit to the separation of indenopyrene and dibenzoanthracene. Although these targets are not isomers, dibenzoanthracene has a qualifier ion of the same mass-to-charge of the indenopyrene target ion (*m/z* 276). Without deconvolution, coelution of these compounds could lead to errors in quantitation of indenopyrene. Figure 3 shows overlays of the extracted ions *m/z* 276 and 278. The calculated resolution of the extracted ion chromatogram *m/z* 276 was 34.3 %, which met the 50 % requirement.

One difference between the Intuvo 9000 and a traditional GC is the incorporation of a microfluidic temperature programmable retention gap (that is, Guard Chip). In this method, the Guard Chip acts primarily as a sacrificial trap to prevent low volatility matrix from reaching the column. Depending on the temperature programing applied to the Guard Chip and the volatility of the solutes, some broadening may be observed for compounds that are not well focused by the analytical column. This was observed for the first three eluters, 1,4-dioxane, N-nitrosodimethylamine, and pyridine, with the Guard Chip running in track oven mode (Figure 4A). The track oven mode applied a positive 25 °C offset to the Guard Chip heater compared to the column temperature program, which resulted in a starting temperature of 70 °C. By increasing the Guard Chip starting temperature to 200 °C (Table 1), peak shape can be improved by allowing the compounds to stay in the vapor phase until they reach the column, as shown in Figure 4B and Table 2. This enabled reliable quantitation for these compounds down to 5 µg/mL.

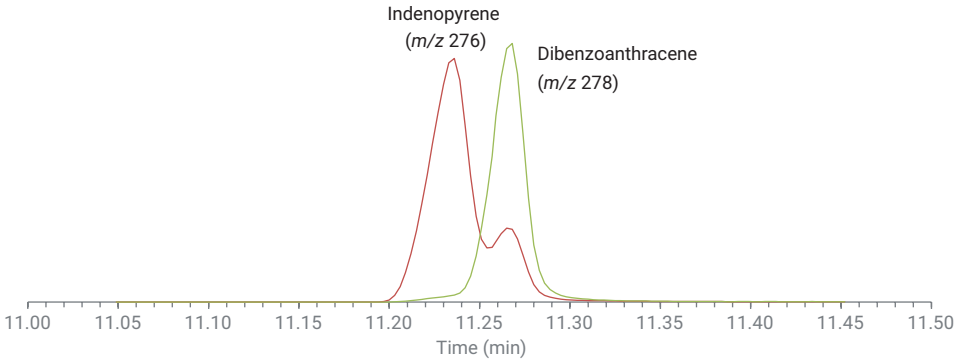

Figure 3. Overlay of extracted ion chromatograms of indenopyrene (*m/z* 276) and dibenzoanthracene (*m/z* 278).

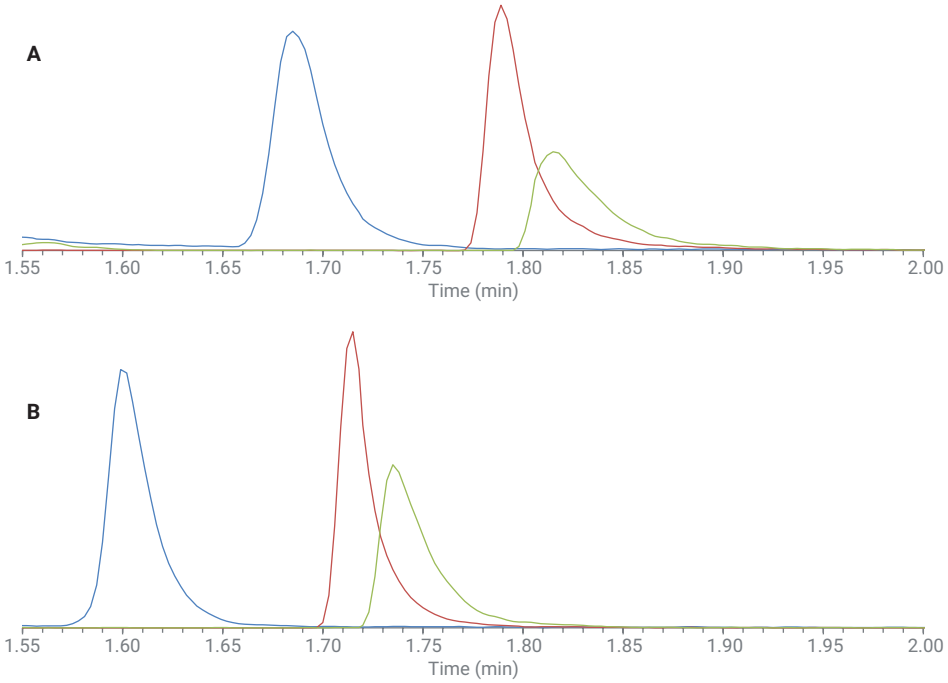

Figure 4. Overlay of extracted ion chromatograms of 1,4-dioxane in blue (*m/z* 88, N-nitrosodimethylamine in red (*m/z* 74) and pyridine in green (*m/z* 78) for the Guard Chip in track oven mode (A) and with the Guard Chip starting at 200 °C.

Table 2. Tailing factors at 5 % peak height.

	1,4-Dioxane	N-nitrosodimethylamine	Pyridine
70 °C (Track oven)	1.8	2.4	3.0
200 °C (Programed)	1.7	1.9	2.4

Conclusion

The GC method optimized for the separation of SVOCs for EPA 8270D on a 30 m × 0.25 mm column was translated to a 20 m × 0.18 mm column on the Intuvo 9000 GC. The run time was reduced by a factor of approximately 1.4 while maintaining the resolution requirements of the method.

Reference

1. Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS); Method 8270D; *United States Environmental Protection Agency, Revision 4*, February **2007**.

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