Analysis of Semivolatiles Using High Efficiency Capillary GC Columns

Application Brief

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Introduction

U.S. EPA Method 8270 is broadly applicable for analysis of semivolatiles using capillary gas chromatography with mass spectral detection. EPA 8270 is widely used in both contract analytical and government environmental laboratories. The method is capable of concurrently measuring a mixture of 70 to 100 acidic, basic, and neutral species. Shifting these important analyses from 0.25-mm id to 0.18-mm id or high efficiency GC columns is a viable means of obtaining faster results and improving laboratory productivity.

In this example, 77 compounds of interest and six internal standards are resolved on a 0.18-mm id high efficiency GC column using 7 minutes of analysis time. The same compounds and internal standards were also resolved using a 0.25-mm id column where 25 minutes of analysis time was required. Analysis speed using the high efficiency column was 8 minutes faster, resulting in a 32% reduction in analysis time.

Experimental

Method translation software available from Agilent Technologies translates chromatographic parameters from an existing method to the new column format with a few simple keystrokes [1].

Column dimensions, flow, and temperature parameters from an existing method are entered into a table along with the desired new column dimensions. The software then generates flow and temperature setpoints for the new translated method. Often these new setpoints yield a successfully translated method with the same separation and elution order with no additional method development. In this example, one-to-one phase-ratio correspondence was maintained between the 0.25-mm and 0.18-mm id column formats, enhancing the reliability of the software's predicted conditions. Keeping the phase ratio constant helps maintain peak elution order on the new column.

Instrument conditions are described in Table 1, and Figures 1 and 2.

Highlights

- 0.18-mm id, also known as high-efficiency GC columns, deliver faster results for U.S. EPA 8270 analyses.
- 32% reduction in analysis time when translating 0.25-mm id column method to the 0.18-mm id format.
- Resolution of 77 peaks of interest is maintained for the faster 0.18-mm id separation.
- DB-5.625 column: Agilent DB-5.625 column in 0.18-mm id provides faster sample analysis without loss of resolution.

Table 1. Experimental Conditions

Column:	Figure 1. 30 m x 0.25 mm x 0.50 µm DB-5.625 column, Agilent Technologies part number 122-5632 Figure 2. DB-5.625 20 m x 0.18 mm x 0.36 µm column, Agilent Technologies part number 121-5622
Carrier:	He constant-flow mode 1.1 mL/min
Oven:	40 °C for 1.00 min, 25 °C/min to 320 °C 4.80 min hold
Injection:	Splitless 0.5 µL injected at 300 °C, QuickSwap pressure 5.0 psi during acquisition, 80.0 psi during backflush with inlet set to 1.0 psi during backflush
Detector:	Agilent Technologies 5975C Performance Turbo MSD equipped with a 6-mm large-aperture draw-out lens, Agilent Technologies part number G2589-20045

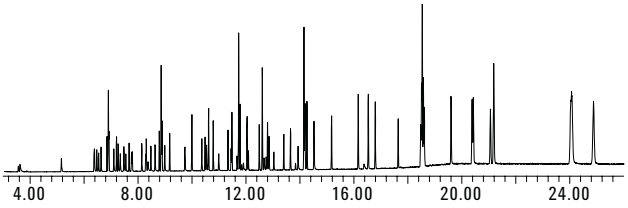

Figure 1. Figure 1. U. S. EPA Method 8270, 5 ng/mL System Performance Check Compounds Chromatogram using a 30-m x 0.25-mm x 0.50-µm DB-5.625 column, Agilent Technologies part number 122-5632. Please refer to Table 1 for instrument conditions.

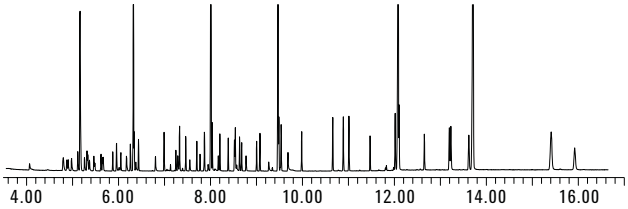

Figure 2. U.S. EPA Method 8270, 5 ng/mL System Performance Check Compounds Chromatogram using a 20-m x 0.18-mm x 0.36-µm DB-5.625 column Agilent Technologies part number 121-5622. Please refer to Table 1 for instrument conditions.

Discussion of Results

Figures 1 and 2 depict the resolution of 77 compounds of interest along with six internal standards first on a 30 m x 0.25 mm x 0.5 µm (Agilent part number 122-5632) standard-bore capillary column (Figure 1) and second on a 20 m x 0.18 mm x 0.36 µm (Agilent part number 121-5622) high efficiency column (Figure 2). Peak resolution and quantification are comparable, and in both cases meet EPA 8270 criteria for System Performance Check Compounds (SPCCs) and Continuous Calibration Compounds (CCCs) over a calibration range from 1 to 200 ppm; 5 ppm SPCC chromatograms were selected for visualization purposes.

Significant improvement in analysis time was achieved by shifting the column used from a 0.25-mm id standard-bore capillary to a 0.18-mm id high efficiency GC column example; the 0.25-mm id column required 25 minutes of run time, and the 0.18-mm id column required 17 minutes. In this semi-volatile analysis example, 25 minutes of run time were required for the 0.25-mm id column, and 17 minutes were required on the 0.18-mm id column. Moving the analysis to a 0.18-mm id column yielded 8 minutes in time savings or 32% faster sample analysis.

Typical run time for EPA 8270 analysis using 0.25-mm id or standard-bore capillary columns is 25 minutes, excluding post-analysis bakeout and system cooldown time often required for dirty samples. When bakeout and subsequent system cooldown periods are accounted for, the overall cycle time climbs to 57 minutes. As shown above, a time saving of 8 minutes was achieved by using a high efficiency column. Further improvements in the cycle time for EPA 8270 analysis are achieved through the use of several advanced features on the Agilent 7890A GC. A QuickSwap device installed in a 7890A can be used to backflush heavy material matrix contaminants back out of the inlet, dramatically reducing matrix bakeout time [2]. Faster cooldown and thermal isolation features available on the 7890A GC also reduce system cycle times for dirty samples. The combination of a high efficiency column and the unique features of the 7890A reduce sample analysis time from 57 minutes to 24.3, a 32.7-minute time saving per sample run.

Conclusions

High efficiency GC columns provide a straightforward way to obtain faster results for EPA 8270 analysis without compromising resolution.

References

1. Method translation software: free download of method translation software available at http://www.chem.agilent.com/cag/servsup/usersoft/files/GCTS.htm.
2. Mike Szelewski, “Significant Cycle Time Reduction Using the Agilent 7890A/6975C GC/MSD for EPA Method 8270,” Agilent Technologies publication 5989-6026EN

For More Information

For more information on our new line of high efficiency GC columns, please visit this link: http://www.chem.agilent.com/scripts/PDS.asp?lpage=60005.

For more information on our products and services, visit our Web site at www.agilent.com/chem.

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