Fast Screening of PCB Congeners Using the Agilent 6890/5973N GC/MSD System

Application

Gas Chromatography

May 2000

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Abstract

Agilent Technologies' fast GC/MSD method can significantly speed up the screening of PCB congeners. Agilent's GC method translation software (available free from the Agilent Technologies Web site, http://www.chem.agilent.com/cag/servsup/usersoft/main.html#mxlator) was used in developing the new method based on a standard 42-min method. A 15-m × 0.25-mm × 0.25-µm Agilent HP-5MS column was used to increase analysis speed up to four-fold. The time savings were implemented in increments (down to 10.5 minutes) to verify the predictability of scaling and the affect of scaling on the signal-to-noise ratio.

Key Words

RTL, PCB, polychlorinated biphenyls, congeners, environmental, screening, fast GC, method translation, 5973, 6890, MTL

Introduction

Polychlorinated biphenyls (PCBs) are a group of 209 individual compounds (known as congeners) with varying harmful effects. Chronic (long term) exposure to some PCB formulations by inhalation in humans results in respiratory tract symptoms, gastrointestinal effects, mild liver effects, and effects on the skin and eyes such as chloracne, skin rashes, and eye irritation.

PCBs are no longer produced in the United States and are no longer used in the manufacture of new products. Smaller amounts of PCBs may be released to the air from disposal sites containing transformers, capacitors, and other PCB wastes, incineration of PCB-containing wastes, and improper disposal of the compounds to open areas. Today, PCBs are still detected in water and soil due to the environmental recycling of the compound. PCBs have been detected in foods and they bio-accumulate through the food chain, with some of the highest concentrations found in fish.

The analysis of PCBs normally is accomplished using GC with an electron-capture detector (ECD). Because of the drastically different toxicity of the different congeners, it is of great interest to identify the individual congeners using a mass spectrometer (MS).

Agilent Technologies has developed techniques to solve the peak identification problem based on Agilent's retention time locking (RTL) and a mass spectral library that contains the locked retention times and characteristic ions for all 209 PCB congeners. A GC/MSD method was developed based on a standard 42-min method[1] to screen for all congeners. A specific combination of column stationary phase, carrier-gas flow rate, and oven temperature programming is required to lock all the compounds to an expected retention timetable[2]. Compound identification based only on spectral searching alone is difficult when the isomers have the same mass spectra.

The screening tool, integrated within Agilent's ChemStation for MSD software, searches for all 209 congeners by first checking and integrating the

expected target ion within the expected time window. If the target ion is found, the software will then search and integrate the three qualifier ions within the expected time window. Last, the software will print out a report showing "hits" and "possible hits" (ratios of characteristic ions that do not match the expected values in the library within specified limits).

In order to improve laboratory productivity, we scaled the method for four-fold speed-up. While a 30-m × 0.25-mm × 0.25-µm Agilent HP-5MS column is used for standard speed, a 15-m × 0.25-mm × 0.25-µm Agilent HP-5MS column is used for the four-fold speed. These faster methods were able to be scaled exactly as predicted by using a combination of Agilent's method translation (MTL) and RTL software.

Often, when speeding up GC methods, an analyst trades resolution for increased analysis speed. This loss of resolution can complicate peak identification, even with a mass selective detector (MSD). However, because scaling was exact, the faster methods can be used with precisely scaled congener libraries, making the screening process even more powerful and adaptable to individual needs.

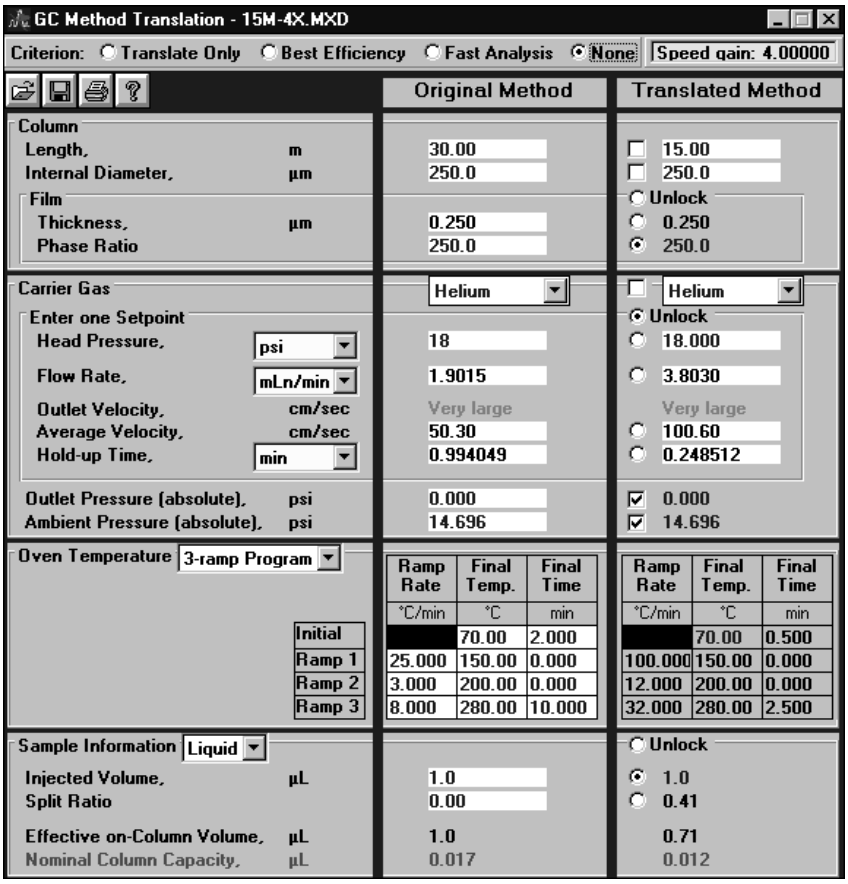

Figure 1. Screen capture showing the method translation (MTL) software data entry used in a 4X speed-gain translation.

Experimental

The GC method translation software tool was used to find operating conditions for the faster methods. Figure 1 is a screen capture of the MTL software data entry showing the original conditions and the new chromatographic conditions for a four-fold speed gain. The column flow rate, which is helpful to avoid exceeding MSD pumping capacity[3], also is found in the table. In this study, a turbo pump that could handle the 3.8 mL/min carrier flow was used. The program also determined the required column head pressure and corresponding oven ramp. The Agilent 6890 GC fast oven option (220/240V in the U.S.) was required for the faster oven ramp used in this study.

General chromatographic conditions are listed in Table 1. The RTL standard used was a mixture of pesticides and PCB congeners at 10 ppm. A 15-m × 0.25-mm × 0.25-µm Agilent HP-5MS column (part number 19091S-431) was used. The head pressure determined by the method translation software (18 psi) was used as the starting point for retention time locking. The column head pressure required to lock retention times of the compounds to the library (the original retention time divided by four) was determined using the automated RTL process integrated within the Agilent ChemStation for MSD.

A very important modification to the MS method is changing the default values of "Use mass range from" to **–0.50** to **+0.50** amu (the default values are –0.3 to +0.7). The changes can be made from the "Extracted Ion Chromatograms…" dialog box selected from the "Chromatogram" on the menu bar.

Figure 2 shows the results of the shortened analysis times. The two chromatograms look extremely similar, except that the time axis is scaled proportionally. It is interesting to note that the last peak in the 4X analysis came out *before the first peak of the 1X analysis.* Because MTL followed by RTL scales methods very precisely, scaled screening libraries for corresponding time

reductions can be obtained by dividing the retention times in the library by the speed gain (which does not have to be an integer).

Conclusion

The highly accurate and reproducible pressure and temperature control of the Agilent 6890 GC allows precise scaling of a standard 42-min GC/MSD method. The run time was shortened to 10.5 minutes using a fast oven ramp rate and a 15-meter 250-micron column. The combination of MTL and RTL facilitated scaling and yielded exact scaling. RTL libraries can be scaled accurately to correspond to the faster analyses. The GC/MSD conditions used are the same as the fast pesticide method[4], which allows for screening pesticides and PCB congeners in a single analysis.

References

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Table 1. Chromatographic Conditions

Speed	Standard	Four-fold
GC	110 V	220/240 V
Column	30-m × 0.25-mm × 0.25-µm Agilent HP-5MS (part number 19091S-433)	15-m × 0.25-mm × 0.25-µm Agilent HP-5MS (part number 19091S-431)
Injection mode	Splitless	Splitless
Column head pressure	18.0 psi	18.0 psi
Column flow (mL/min)	1.9	3.8
Inlet control mode	Constant pressure	Constant pressure
Carrier gas	Helium	Helium
Injector Temperature	250 °C	250 °C
Oven Temperature	70 (2 min)	70 (0.5 min)
Ramp 1	25 °C/min	100
	150 (0 min)	150 (0 min)
Ramp 2	3 °C/min	12
	200 (0 min)	200 (0 min)
Ramp 3	8 °C/min	32
	280 (10 min)	280 (2.5 min)
Oven equilibration	2 min	2 min
Injection volume	1 µL	1 µL
Liner	5183-4647	5183-4647
MS Conditions (Turbo pump)		
Solvent delay	3 min	0.9 min
Tune file	Atune.u	Atune.u
Low mass	50 amu	50 amu
High mass	550 amu	550 amu
Threshold	200	200
Sampling	3	1
Scans/sec	1.52	5.56
Quad Temperature	150 °C	150 °C
Source Temperature	230 °C	230 °C
Transfer line Temperature	280 °C	280 °C
Acquisition mode	Scan (EI)	Scan (EI)

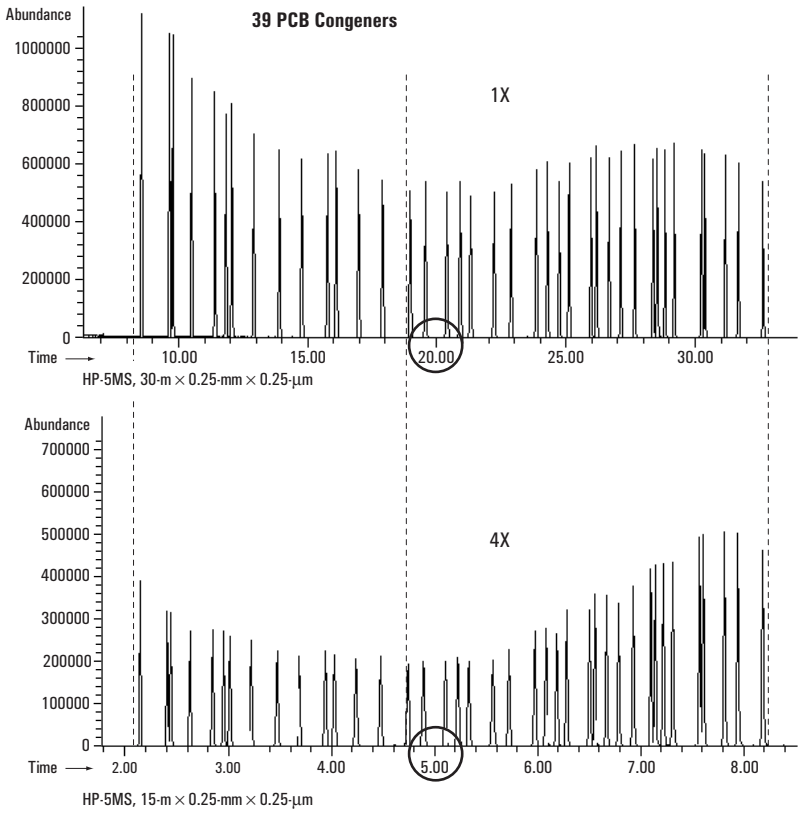

Figure 2. The TICs of the standard speed and fast (4X) analyses. The standard analysis (1X) was 42 minutes long.

Printed in the USA 5/2000
5980-1472E

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