Fast Screening of Pesticide and Endocrine Disrupters Using the Agilent 6890/5973N GC/MSD System, Part I

Application

Gas Chromatography
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Abstract

Agilent Technologies' new, fast GC/MSD method can significantly speed up the screening of pesticides. 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 the standard 42-min method. A 10 m x 0.1 mm x 0.1 µm HP-5 column was used to increase analysis speed up to fourfold. The time savings were implemented in increments (down to 10.5 minutes) to verify the predictability of scaling and the effect of scaling on the signal-to-noise ratio.

Key Words

RTL, pesticide, environmental, screening, fast GC, method translation, 5973, 6890, MTL

Introduction

Analysts want faster analyses to improve laboratory productivity. Often, when speeding up GC methods, an analyst will trade resolution for increased analysis speed. This loss of resolution can complicate peak identification, even with a mass selective detector (MSD).

Agilent Technologies has developed new techniques to solve the peak identification problem based on Agilent's retention time locking (RTL) software and a new mass spectral library that contains the locked retention times and characteristic ions for 567 of the most common pesticides and endocrine disrupters of concern worldwide. A GC/MSD method was developed based on the standard 42-min method[1] to screen for all 567 of the most common analytes. 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 analyzing extracts containing significant sample matrix content because of overlapping peaks and noisy baselines.

The new screening tool, integrated within Agilent's ChemStation for MSD, searches for all 567 compounds by first checking and integrating four characteristic ions within the expected time window, and second by printing 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 one application, the analysis time of the standard pesticide method was reduced by one half, two-thirds, and three-fourths. The faster methods were scaled exactly as predicted by using a combination of Agilent's method translation (MTL) and RTL software. Because scaling was exact, these faster methods can be used with precisely-scaled pesticide libraries, making the screening process even more powerful and adaptable to individual needs.

Experimental

The GC method translation software tool was used to find operating conditions for the faster methods. Figure 1 is a screen capture of MTL software data entry showing the original conditions and the new chromatographic conditions for a twofold speed gain. The column flow rate, which is helpful to avoid exceeding MSD pumping capacity[3], is also found in the table. A 16:1 split ratio was suggested in the table as a proportional scaling from the original column to the smaller i.d. column with corresponding lower capacity. 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.

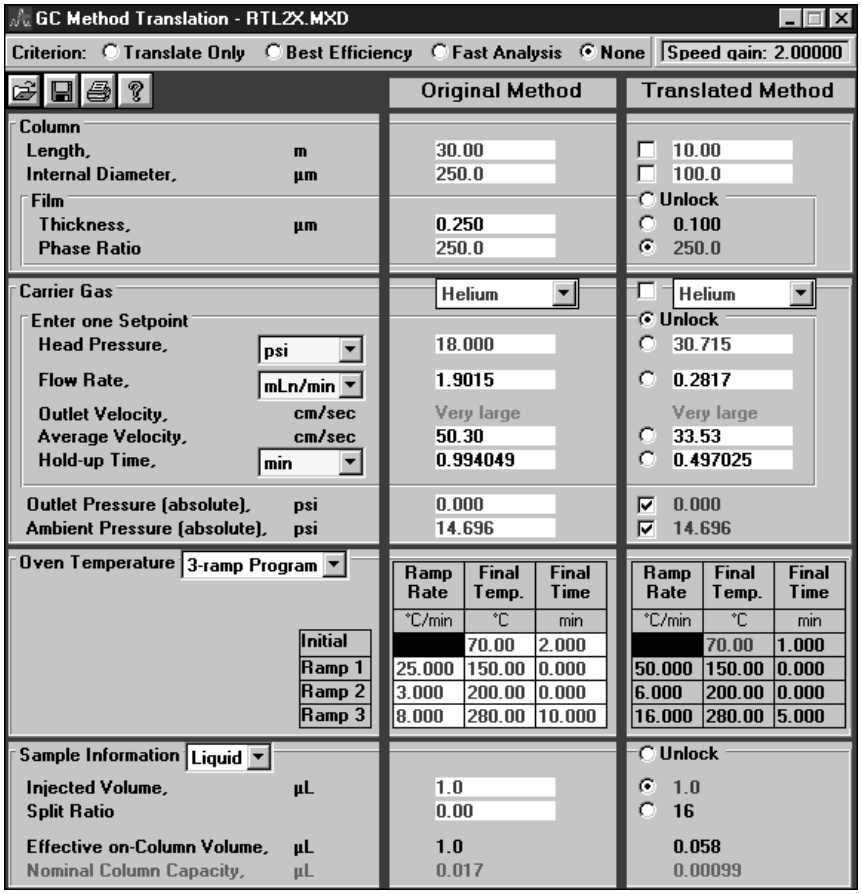

Figure 1. Screen capture showing the method translation (MTL) software data entry used in a twofold speed gain translation.

General chromatographic conditions are listed in table 1. The standard used was a mixture of 26 pesticides at 10 ppm. A 10 m x 0.1 mm x 0.1 µm HP-5 column (part number 19091J-141) was used. The head pressure determined by the method translation software (30.72 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 2) was determined using the automated RTL process integrated within the Agilent ChemStation for MSD. This process (first translate the method then lock the retention times) was repeated for the threefold and fourfold time reductions.

Table 1. Chromatographic Conditions

Speed	Onefold (1X)	Twofold (2X)	Threefold (3X)	Fourfold (4X)
GC	110 V	220/240 V		
Column	30 m x 0.25 mm x 0.25 µm HP5-MS (P/N 19091S-433)	10 m x 0.1 mm x 0.1 µm HP-5 (P/N 19091J-141)		
Injection mode	Splitless	16:1 split		
Column head pressure	18.0 psi	36.55 psi	63.17 psi	90.0 psi
Column flow (mL/min)	1.5	0.4	0.8	1.5
Inlet control mode	Constant pressure	Constant pressure		
Carrier gas	Helium	Helium		
Injector temperature	250 °C	250 °C		
Oven temperature	70 (2 min)	70 (1 min)	70 (0.67 min)	70 (0.5 min)
Ramp 1	25 °C/min	50	75	100
	150 (0 min)	150 (0 min)	150 (0 min)	150 (0 min)
Ramp 2	3 °C/min	6	9	12
	200 (0 min)	200 (0 min)	200 (0 min)	200 (0 min)
Ramp 3	8 °C/min	16	24	32
	280 (10 min)	280 (5 min)	280 (3.33 min)	280 (2.5 min)
Oven equilibration	2 min	2 min		
Injection volume	1 µL	1 µL		
Liner	5183-4647	5183-4647		
MS Conditions				
Solvent delay	3 min	1.8 min	1.2 min	0.9 min
Tune file	Atune.u	Atune.u		
Low mass	35 amu	35 amu		
High mass	500 amu	450 amu		
Threshold	150	250		
Sampling	2	2	1	1
Scans/sec	3.15	3.50	6.54	6.54
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 shows the results of the shortened analysis times. The three chromatograms look extremely similar, except that the time axis is scaled proportionally. 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). The peak heights from all the methods are very similar. Although the sample was split 16:1 for the smaller column, the small column i.d. and faster oven ramp combination made the peaks narrower and higher, so there was minimal loss in the signal to noise ratio.

Conclusion

The highly accurate and reproducible pressure and temperature control of the Agilent 6890 GC allows precise scaling of the standard 42-min GC/MSD pesticide method. Run time was shortened to 10.5 minutes using a fast oven ramp rate and a 10-meter 100-micron column. The combination of MTL and RTL facilitated scaling and yielded exact scaling. RTL libraries can accurately be scaled to correspond to the faster analyses.

References

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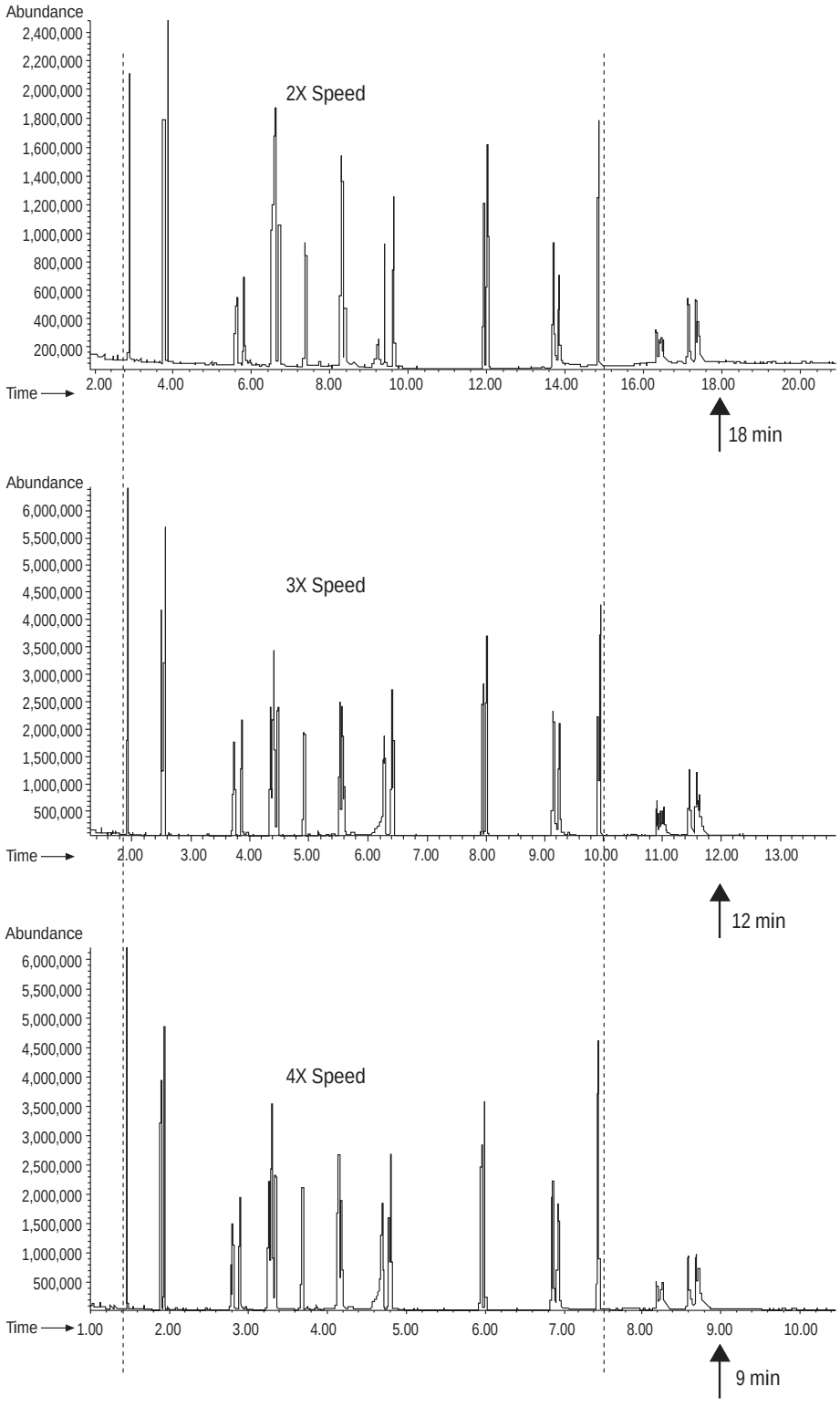

Figure 2. Three TICs of the 2X, 3X, and 4X speedups. The standard analysis (1X) was 42 minutes long. The two vertical lines on the figure are used as references to show the similarity of the TICs.

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