METHOD TRANSLATION OF HJ679-2013 FOR INTUVO

Translating a 530 µm id Column Method to 250 µm or 320 µm column Methods for the Agilent Intuvo 9000 GC

Introduction

HJ method 679-2013 describes the determination of acrolein, acrylonitrile, and acetonitrile from soil and sediments by headspace-gas chromatography. The method specifies a 30 m × 530 µm column with a polyethylene glycol phase. This method has historically been run on conventional gas chromatographs with traditional nut and ferrule connections. To apply this to the technology included in the Agilent Intuvo 9000 GC, such as direct heating and click-and-run connections, the method was translated to a 250 µm column. By maintaining the same phase ratio between the initial method and the translated method, similar chromatograms, retention times, and elution order were achieved.

Experimental

The original HJ-679 method specifies a 30 m column with an internal diameter of 530 µm. An Agilent DB-Wax UI (p/n 125-7032UI) was used for the initial evaluation on an Agilent 7890 GC equipped with an FID and Agilent 7697 headspace samples [1]. A DB-Wax UI column with a 250 µm phase and 0.50 µm film thickness (p/n 122-7033UI-INT) provided the same phase ratio, and was used initially with an Intuvo 9000 GC to demonstrate the translated method (Tables 1 and 2). The Method Translator calculator was used to determine the column flow rate for the translated method (Figure 1).

Taking advantage of the high heating rates the Intuvo system can achieve, a third method was developed using the Method Translator with a thinner film column (Figure 2).

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Table 1. GC Method conditions.

Parameter	Agilent 7890 value	Agilent Intuvo 9000 value	Agilent Intuvo 9000 fast value
Inlet	150 °C Split 5:1		
Column	Agilent DB WaxUI, 30 m × 530 µm, 1.0 µm	Agilent DB WaxUI, 30 m × 250 µm, 0.50 µm	Agilent DB Wax UI, 30 m × 320 µm, 0.25 µm
Column flow rate	4.5 mL/min	1.54 mL/min	3.5 mL/min
Oven	40 °C (5 minutes) then 5 °C/min to 60 °C then 30 °C/min to 150 °C (5 minutes)	40 °C (5 minutes) then 5 °C/min to 60 °C then 30 °C/min to 150 °C (5 minutes)	40 °C (1.34 minutes) then 18.6 °C/min to 60 °C then 112 °C/min to 150 °C (1.34 minutes)
FID	250°C		
Jumper chip	150 °C		
Bus temperature	ON (Default)		

Table 2. Instrument conditions for the Agilent 7697 Headspace Sampler. These conditions were used with each instrument/method permutation.

Parameter	Value
Oven	75 °C
Loop	105 °C
Transfer line	150 °C
Vial equilibration	30 minutes
Injection duration	0.1 minutes
Vial	20 mL
Shaking	On, level 1
Vial fill flow	50 mL/min
Vial fill pressure	8 psi
Vial pressure equilibration time	2 minutes
Loop fill ramp rate	20 psi/min
Final loop pressure	1.2 psi
Loop equilibration	0.2 minutes

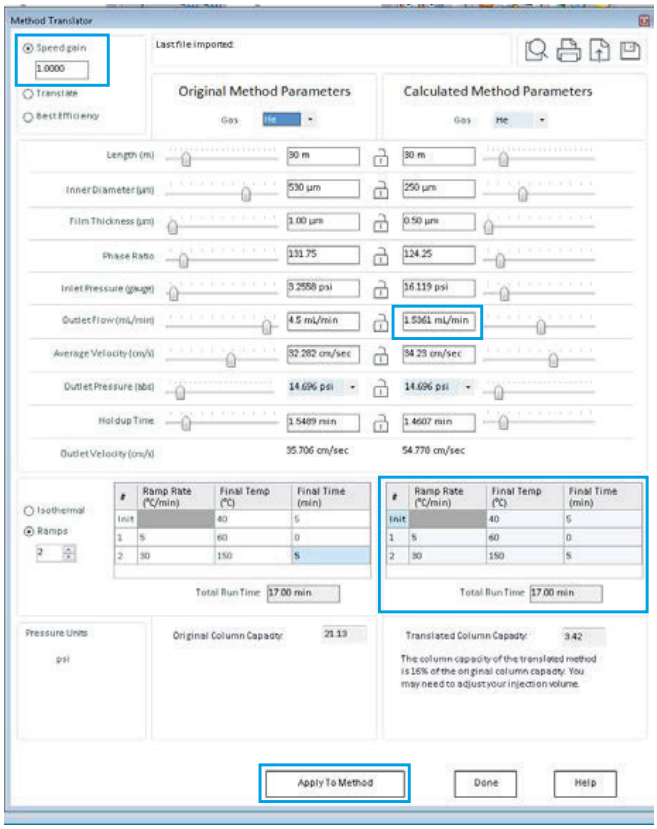

Figure 1. The Method Translator, one of the available GC calculators, enables the application of an Agilent 7890 method configured with a 530 µm id column to an Agilent Intuvo 9000 configured with a 250 µm id column.

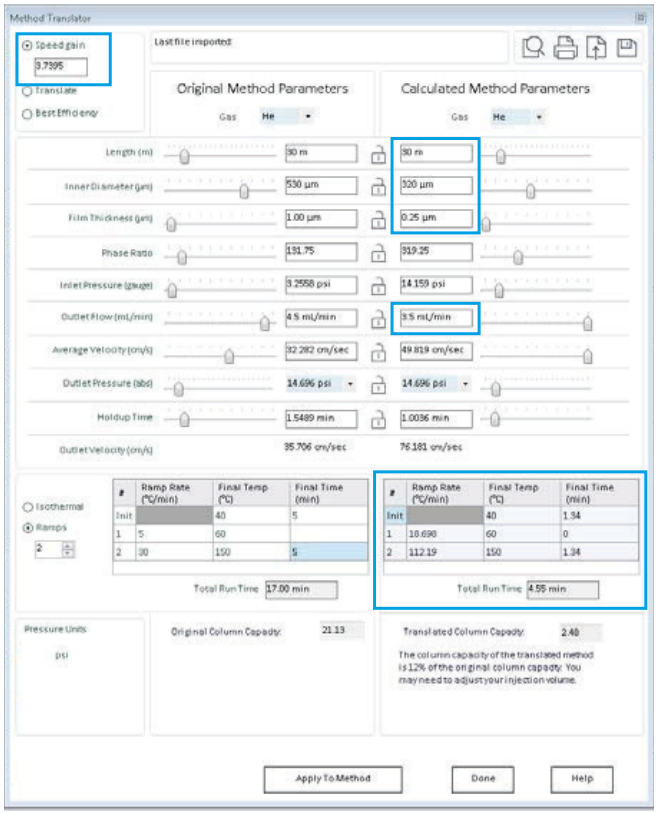

Figure 2. A translated method with thinner film thickness provides a faster method with a speed gain of 3.7.

Results and Discussion

The Method Translator calculator makes translating methods simple and straight forward. The original column and method parameters were entered in the left column: **Original Method Parameters**. Because Intuvo columns feature a smart key, which preconfigures the instrument and method with the correct column information, the column parameters in the right column: **Calculated Method Parameter** were prepopulated. A new flow rate and oven program was calculated based on the original column parameters and the new column parameters. By selecting columns with the similar phase ratios and selecting **Speed gain 1**, a new flow rate for the Calculated Method was determined, but the oven program remained unchanged. Using the **Apply to Method** button, the new method parameters were added to the current method.

Figure 3 shows the original chromatogram obtained on the Agilent 7890 GC system. The chromatogram in Figure 4 was obtained by applying the translated method parameters from Figure 1 to the Intuvo 9000 GC system. The resolution between the three compounds was maintained with the same oven program as the original 7890 method. The retention times obtained for acrolein, acrylonitrile, and acetonitrile with the translated method were 4.1 minutes, 8.4 minutes, and 8.8 minutes, respectively. Because the column film thickness phase ratio was similar, but not identical, between the 7890 and Intuvo systems, the observed retention time shift was expected. However, the resolution between the peaks was maintained. For the 7890 system, the resolution between acrolein and acrylonitrile was calculated to be 58, and the resolution between acrylonitirile and acetonitrile was calculated to be 5.3. For the Intuvo chromatogram, the resolution between acrolein and acrylonitrile was also calculated to be 56, and the resolution between acrylonitrile and acetonitrile was calculated to be 5.8. Retention time locking can be used to further match the retention times between the two GC systems.

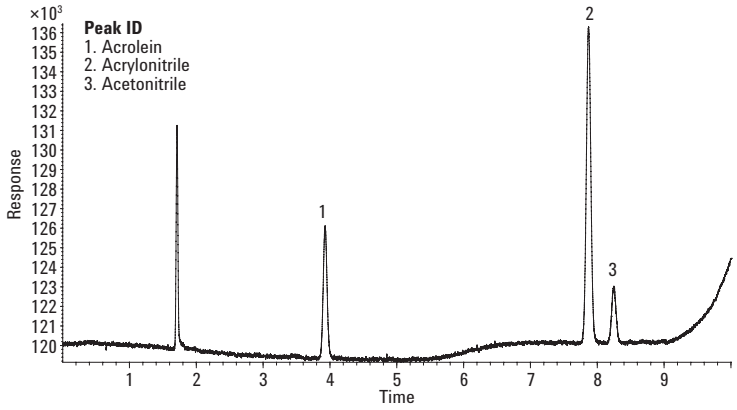

Figure 3. The original chromatogram, obtained on an Agilent 7890 GC system with a 530 µm id column, demonstrates the separation of acrolein (1), acrylonitrile (2), and acetonitrile (3) at 3.9 minutes, 7.9 minutes, and 8.3 minutes, respectively.

Because a different column was required when translating the original HJ679-2013 method to Intuvo, a new method was developed to take advantage of the additional features of Intuvo, such as the direct heating capability, which allows temperature programming rates up to 250 °C/min. Using a thinner column phase, the analysis time was reduced, and throughput was increased. Using the column dimensions shown in the Method Translator in Figure 2, a new flow rate and oven programming rate was obtained and applied to the Intuvo method. In Figure 5, the same three analytes were separated in under 3 minutes. The elution order remained unchanged because the same column phase was used. The phase ratio changed, which decreased the resolution (peak spacing), but still exceeded baseline resolution requirements at 2.5 or better.

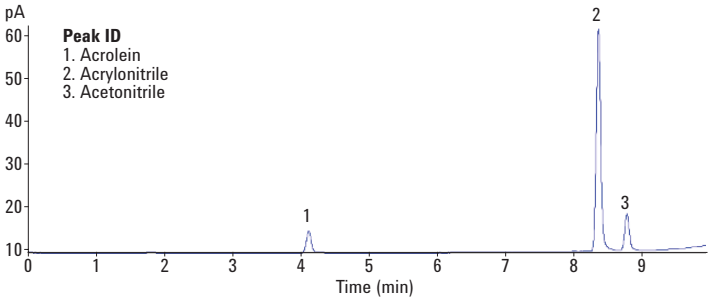

Figure 4. Agilent Intuvo 9000 GC chromatogram with the translated method, using a 250 µm id column, showing the same elution order, resolution, and similar retention times as the original method.

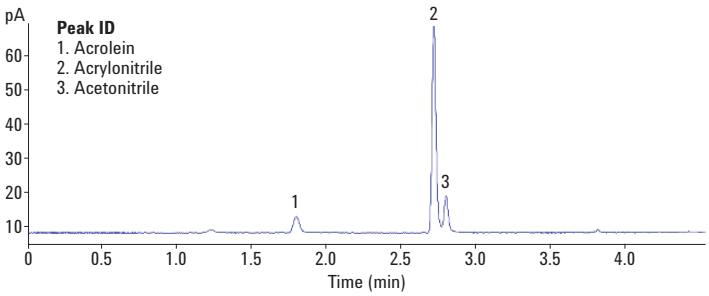

Figure 5. A translated method, taking advantage of the fast heating rates achievable with the Agilent Intuvo 9000 GC system shows acrolein (1), acrylonitrile (2), and acetonitrile (3) at 1.796 minutes, 2.718 minutes, and 2.798 minutes, respectively.

Conclusions

The tools available in Agilent chromatography software provide an easy way to translate methods from one GC platform to another. In the case of method HJ 679-2013, the column used on the Agilent 7890 GC system was not available for the Agilent Intuvo 9000 GC so a column of similar phase ratio was chosen. The Method Translator calculator was also used to determine the column flow rate needed to obtain similar chromatography with the same oven program as the orginal method. The elution order and resolution was maintained. The Method Translator calculator was also used to determine new column flow and oven program for a speed-optimized method. Because the same column phase (DB-Wax UI) was used in all three cases, the elution order did not change. However, because the film thickness was changed, the resolution decreased for the third, fast method. Both methods yielded excellent chromatographic results, and demonstrated the ease with which analytical methods from various gas chromatography platforms can be applied and optimized for the Intuvo 9000 GC system.

Reference

1. Acrolein, Acrylonitrile, and Acetonitrile by HS-GC, *Agilent Technologies*, publication number 5991-8096EN (**2017**).

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Information, descriptions and specifications in this publication are subject to change without notice.

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