Demonstrating Method Equivalency with a new GC Platform

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Introduction

Transferring established chromatographic methods to a new platform can be troublesome if it requires significant changes or re-optimization of method parameters. This is most commonly experienced when changing the inlet type or analytical column. The Intuvo GC system leverages the same inlet and column technology as conventional Agilent GC systems in order to provide seamless method transfer. The same inlet parameters, column stationary phase, oven programming rate, and detector setpoints can be used with the Intuvo GC system as in the previously established method. The uniqueness of the Intuvo flow path, i.e. the guard chip and modular flow chips, only result in two additional set points that are added to the Intuvo GC system method.

Three examples of method transfer from an Agilent 7890 GC system to an Agilent Intuvo 9000 system are given. Additional examples are given in the references[1,2].

Method Transfer Basics

In general, conventional methods transfer seamlessly from air bath oven systems to Intuvo GC systems. Whenever transferring methods between systems, it is important to ensure that the two systems are configured in the same way. If the original system is configured with a split/splitless inlet and a flame ionization detector, for example, the Intuvo should be configured the same way. Figure 1 shows the components of Intuvo. Intuvo flow settings are equivalent to method flow settings in conventional air bath ovens and can be ported over directly. However, Intuvo greatly simplifies backflush and complex configurations through the use of click-and-run technology.

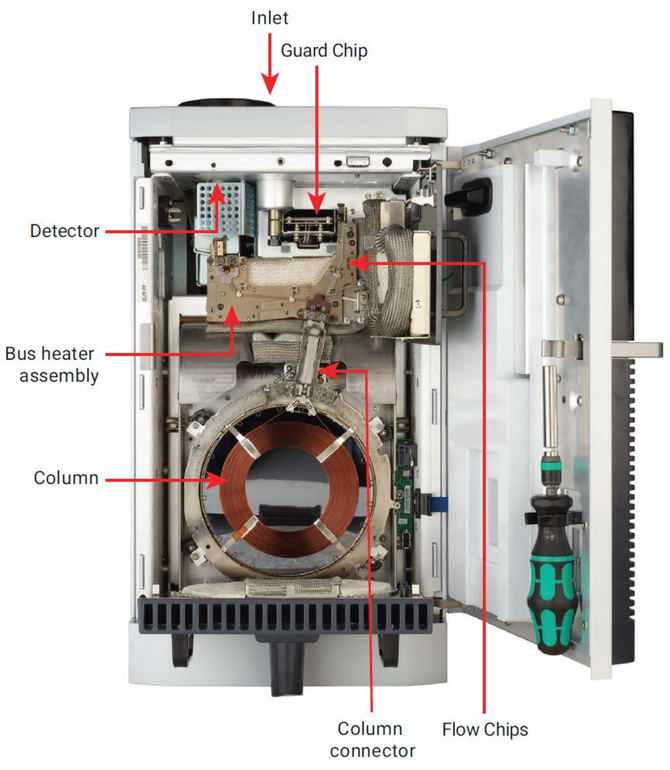

Figure 1. Basic Agilent Intuvo 9000 GC flowpath components.

Retention Time Considerations

It is normal for column length to vary slightly from one column to another. If a conventional column had been clipped at all, it is likely that its length will vary from the Intuvo column. This could shift retention times slightly. It is good practice to always check retention times and adjust accordingly.

Intuvo has a built-in retention gap therefore, when transferring from a conventional systems using a similar retention gap (approximately 0.75 m path length), retention times should compare very well. If the original conventional system does not use a retention gap, there is likely a flowpath length difference from Intuvo. This difference could lead to a slight shift when transferring to Intuvo, especially for early eluting compounds. This is to be expected, and is due to the slight increase in path length of the retention gap[3].

Table 1. In most method transfers, temperature settings do not require user intervention; they are either the same as conventional, or set automatically by the Agilent Intuvo 9000 GC.

Component	Function	Temperature Setting
Inlet	Equivalent to conventional	Same as conventional
Guard Chip	Retention gap	Default = automatically set to track oven temperature Optional = user variable
Bus heater block	Oven function	Default = automatically set by oven temperature Optional = user variable
Column connector		Automatically set based on oven temperature
Column	Equivalent to conventional	Same as conventional
Detector		

Semivolatile Organic Compounds by EPA 8270D

As certain semivolatile organic compounds are considered environmental contaminants, there are established methods by world regulatory agencies, such as the United States Environmental Protection Agency (EPA). These methods have set performance criteria that must be met before reporting this type of contamination. The method established on a conventional GC system was successfully transferred to Intuvo[4]. Only the Guard Chip temperature (track oven) and bus temperature (default) were added (Table 2). In track oven mode, the Guard Chip is programmed at the same rate as the column, with a 25 °C offset. The default bus temperature is the final temperature of the oven program. Nearly identical performance, including retention times, was achieved on the Intuvo GC system when compared to the results achieved with the established 7890 GC system (Figure 2). In fact, the average relative retention time difference was only 0.0006 (Figure 3).

Semivolatile Organic Compounds by EPA 8270D

Table 2. Instrument parameters for a semivolatile organic compound analysis on an Agilent 7890 GC system and an Agilent Intuvo 9000 GC system.

Parameter	Agilent 7890 Value	Agilent Intuvo Value
Injection volume	1 µL	Same
Split/Splitless inlet	300 °C	Same
Pulsed splitless	60 psi until 0.5 minutes	Same
Purge flow	50 mL/min at 0.50 minutes	Same
Guard Chip temperature		Track oven
Bus temperature		Default
Column	Agilent DB-5MSUI 30m x 0.25 mm, 0.5 µm	Same
Flow rate	2 mL/min (constant flow)	Same
Oven program	40 °C (2 minutes), then 20 °C/min to 260 °C, then 6 °C/min to 330 °C (1.33 minutes)	Same
MS transfer line temperature	330 °C	Same
Ion source temperature	330 °C	Same
Quadrupole temperature	200 °C	Same

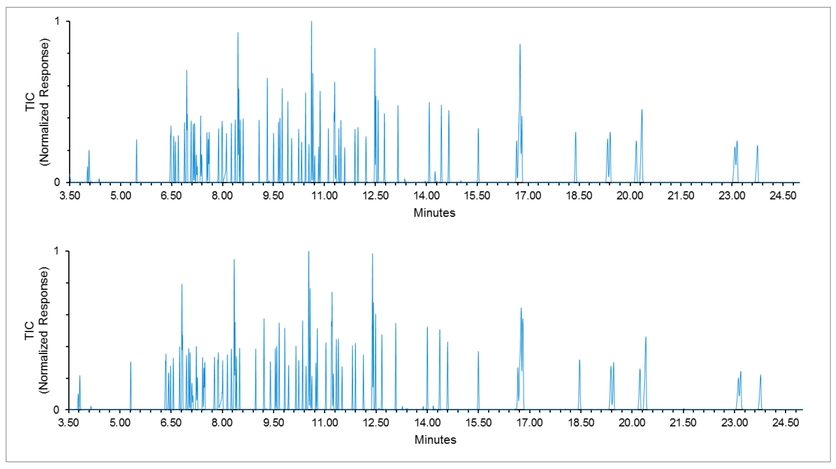

Figure 2. The Intuvo GC system (top) yields nearly identical performance as the 7890 GC system (bottom) with the same method parameters.

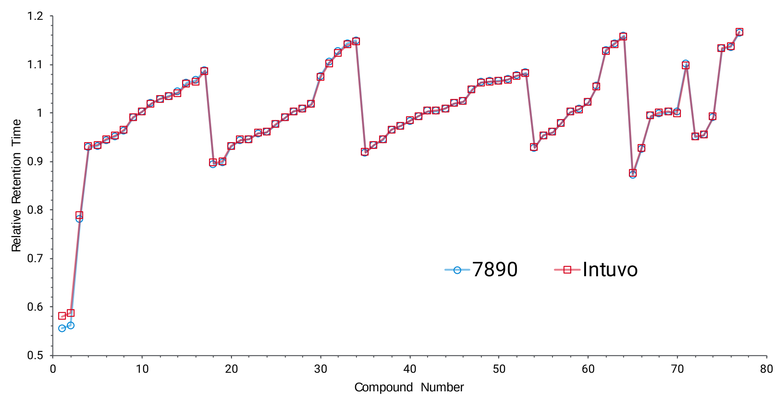

Figure 3. Relative retention times for the 7890 GC system and Intuvo GC system are virtually identical.

Pesticides in Food

Global food supplies require the ability to detect and quantitate multiple classes of compounds in a single analysis. Despite advances in sample preparation, the resulting matrix for pesticide analysis in food is complex, and can be detrimental to chromatographic systems and mass spectrometers. The addition of the Guard Chip in the Intuvo GC system flow path can improve the robustness of the system by protecting the column from matrix, and eliminating the need for column trimming[5]. The Guard Chip does not inhibit transferring complex pesticide methods to the Intuvo GC system because the same method setpoints can be used on both conventional and Intuvo GC systems (Table 3). Using the same method, similar results can be achieved (Figure 4).

Guard Chip Operation

Choose the Guard Chip for maximum protection of the column from sample matrix contamination, and the Jumper Chip where samples injected are of higher purity and the concern for flowpath contamination is low[6]. The Intuvo automatic default temperature setting, track oven, best replicates the outcome of conventional methodology. To help protect the column when analyzing samples with especially dirty matrices, tracking the oven ramp helps optimize the Guard Chip matrix-trapping potential.

Table 3. Instrument parameters for a simplified pesticide analysis. This can be extended to other configurations as well.

Parameter	Agilent 7890 Value	Agilent Intuvo Value
Injection volume	1 µL	Same
Split/Splitless inlet	280 °C	Same
Pulsed splitless	30 psi until 0.5 minutes	Same
Purge flow	15 mL/min at 0.50 minutes	Same
Guard Chip temperature		Track oven
Bus temperature		Default
Column	Agilent HP-5MSUI 15m x 0.25 mm, 0.5 µm	Same
Flow rate	1.3 mL/min (constant flow)	Same
Oven program	60 °C (1.5 minutes), then 50 °C/min to 160 °C, then 8 °C/min to 240 °C, then 50 °C/min to 280 °C	Same
MS transfer line temperature	310 °C	Same
Ion source temperature	280 °C	Same
Quadrupole temperature	150 °C	Same

Pesticides in Food

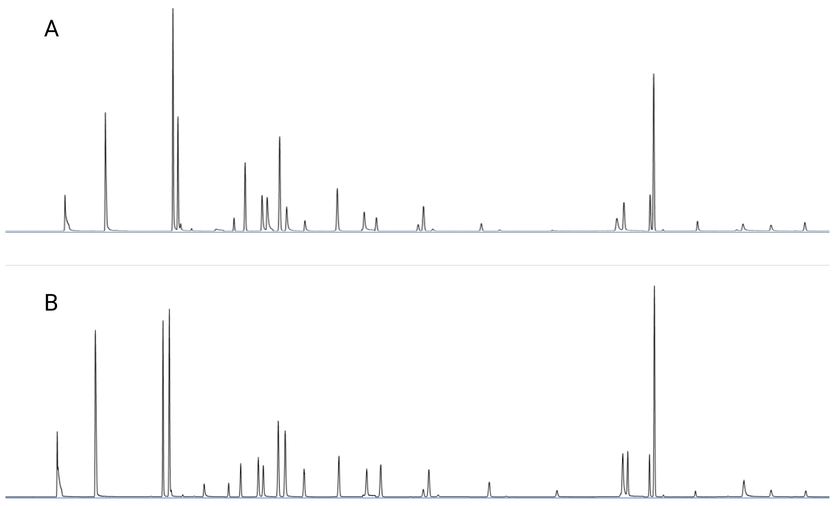

Figure 4. The Intuvo GC system (A) gave similar peak shape and responses as the Agilent 7890 GC system (B).

Analysis of Endrin and DDT

Analysis of endrin and DDT, two pesticides commonly found in soil and water samples is critical to the success of many contract laboratories. Being able to maintain endrin and DDT breakdown levels below method limits, typically <20%, directly impacts the number of samples able to be run in between maintenance and calibration events. Endrin is susceptible to isomerization to endrin aldehyde and endrin ketone at elevated temperatures. Therefore, in order to run samples containing endrin and DDT, it may be necessary to optimize the bus temperature for the Intuvo GC system since it behaves as the fluidic conduit throughout the instrument. When optimizing a method that contains thermally labile analytes, the default bus temperature may need to be reduced in order to achieve acceptable results. Evaluating endrin and DDT breakdown at two different bus temperatures demonstrates the importance of setting this parameter correctly. Starting at an elevated bus temperature of 320 °C resulted in unacceptable breakdown levels. The bus temperature was reduced and optimum breakdown was achieved with a bus temperature of 260 °C (Figure 5).

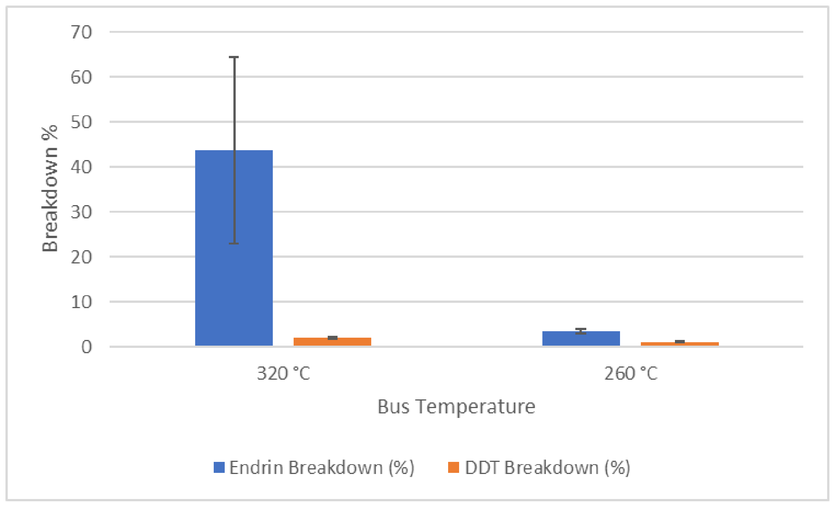

Figure 5. Endrin and DDT breakdown decreases to under 10% and becomes more repeatable when the bus temperature is lowered from 320 °C to 260 °C.

Conclusions

Fundamentally, the Agilent Intuvo 9000 GC system is a chromatographic system and it behaves as such. While the Intuvo GC system provides transformational GC technology, method transfer from a conventional GC system to Intvuo is straightforward. Split/splitless and multimode inlet parameters, capillary column stationary phases, oven temperature programs, and detector setpoints remain the same when moving methods between the two platforms. Beyond the few additional method setpoints, the software operation and detection operation are the same. Agilent MassHunter, CDS ChemStation, and Agilent OpenLab have the same look and feel regardless of hardware platform.

- A conventional method is a great way to start a method for Intuvo.
- Choose the Guard Chip for dirtier samples and the Jumper Chip for samples of higher purity.
- To achieve most equivalent outcomes with an existing method, allow default Guard and Jumper Chip setting to be set automatically (track oven).
- Consider manually setting slightly lower temperatures than default (20 °C lower, for example) for the bus heater assembly for highly thermally labile analytes.
- Consider a Jumper Chip set isothermally for measuring volatiles by headspace or purge-and-trap

References

[1] E. Denoyer, R. Veeneman. Simplifying Method Translation, *Agilent Technologies Technical Overview*, publication number 5991-9149EN, April **2018**.

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[3] R. Veeneman, Updating Pesticide Retention Time Libraries for the Agilent Intuvo 9000 GC, *Agilent Technologies Application Note*, publication number 5991-8446EN, November **2017**.

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