

Transferring Methods to Intuvo: Six Practical Examples

Introduction

This Technical Overview demonstrates different examples of method transfer from an Agilent 7890 GC system to an Agilent Intuvo 9000 GC system. More information on concepts related to transferring methods to the Intuvo GC system can be found in *Simplyfying Method Translation*, Agilent Technologies Technical Overview, publication number 5991-9149EN¹.

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) to measure them. 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 Intuvo². Only the Guard Chip temperature (track oven) and bus temperature (default) were added (Table 1). 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 was achieved on the Intuvo GC system when compared to the results achieved with the established 7890 GC system (Figure 1). In fact, the average relative retention time difference was only 0.0006 (Figure 2).

Table 1. 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 0.5 minutes	Same
Guard Chip temperature		Default (track oven)
Bus temperature		Default
Column	Agilent DB-5MS UI 30 m × 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

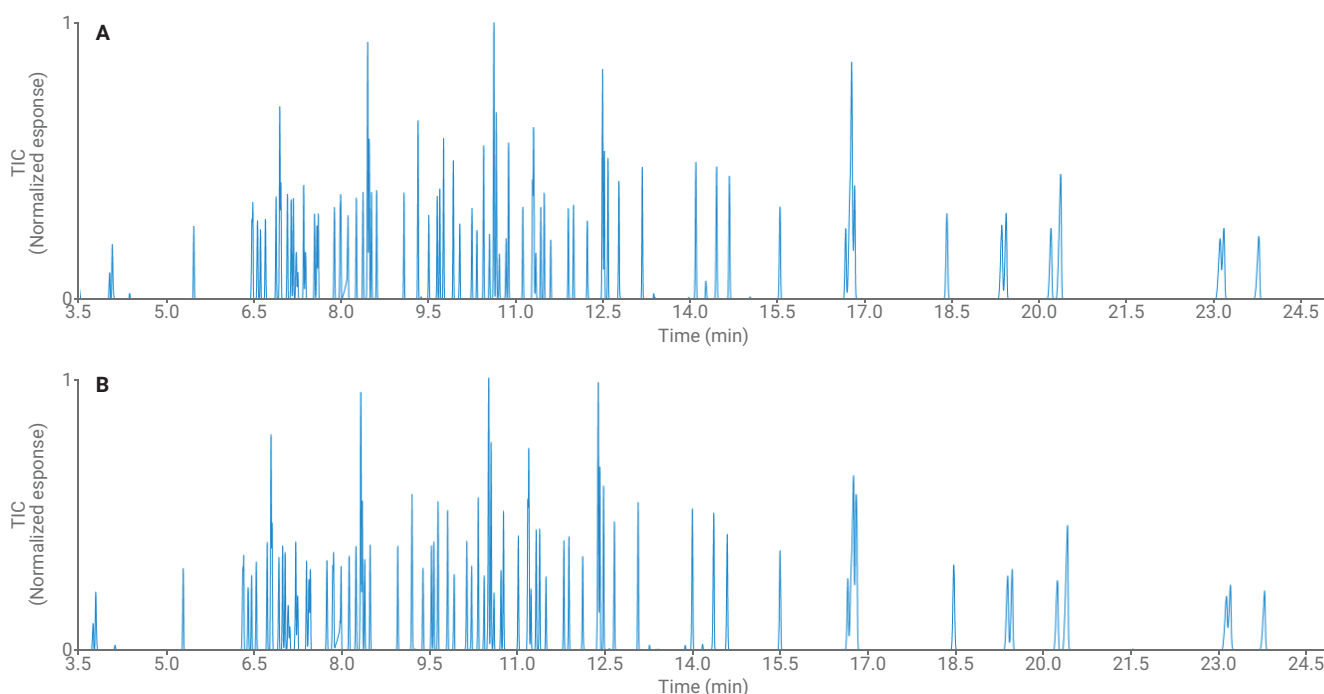


Figure 1. The Agilent Intuvo 9000 GC system (A) yields nearly identical performance as the Agilent 7890 GC system (B) with the same method parameters.

Light petroleum sulfur analysis

Sulfur-containing compounds present in petroleum feedstocks are odorous, corrosive, and monitored closely for process control³. Regulations requiring a reduction in sulfur emissions are increasing. As laboratory bench space becomes a premium at refineries, methods can be transferred from larger conventional GC platforms to a smaller Intuvo platform (Table 2). As with the 8270D method, nearly identical results for a light petroleum sulfur sample containing 23 components were achieved (Figure 3). A Jumper Chip was used for this application because these samples are typically light in matrix and do not require the same protection from the Guard Chip as a dirty sample. To allow the analytes to simply pass through to the column, the Jumper Chip was held at the same temperature as the inlet.

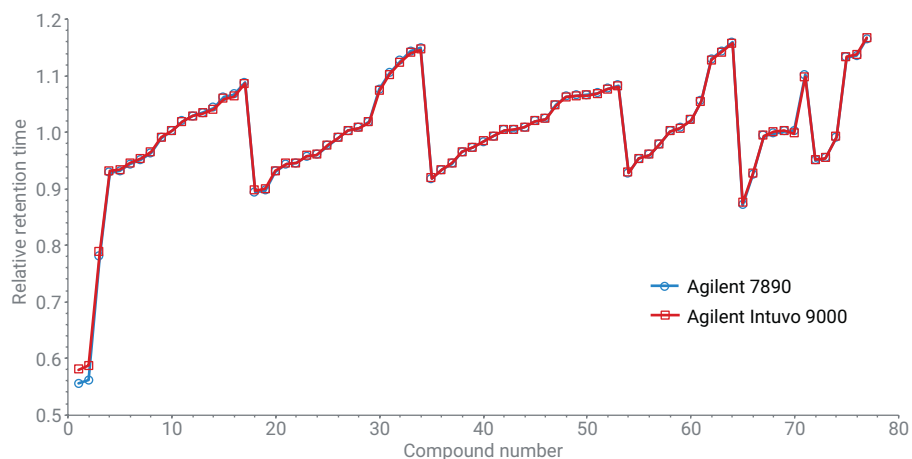


Figure 2. Relative retention times for the Agilent 7890 GC system and Agilent Intuvo 9000 GC system are virtually identical.

Table 2. Instrument parameters for an Agilent 7890 SCD system and an Agilent Intuvo SCD system. Because a standard was evaluated on Intuvo, the Guard Chip was held isothermal.

Parameter	Agilent 7890 value	Agilent Intuvo value
Injection volume	1 μ L	Same
Split/splitless inlet	300 $^{\circ}$ C	Same
Split	10:1	Same
Jumper Chip temperature		300 $^{\circ}$ C
Bus temperature		Default
Column	Agilent DB-1 UI 30 m \times 0.32 mm, 1 μ m	Same
Flow rate	2 mL/min (constant flow)	Same
Oven program	40 $^{\circ}$ C (0.71 minutes), then 14.1 $^{\circ}$ C/min to 250 $^{\circ}$ C (1 minute)	Same
SCD burner temperature	800 $^{\circ}$ C	Same
SCD base temperature	280 $^{\circ}$ C	Same
Oxidizer flow	50 mL/min	Same
Upper hydrogen flow	38 mL/min	Same
Lower hydrogen flow	8 mL/min	Same
Ozone flow	36 mL/min	Same
Range	6	Same

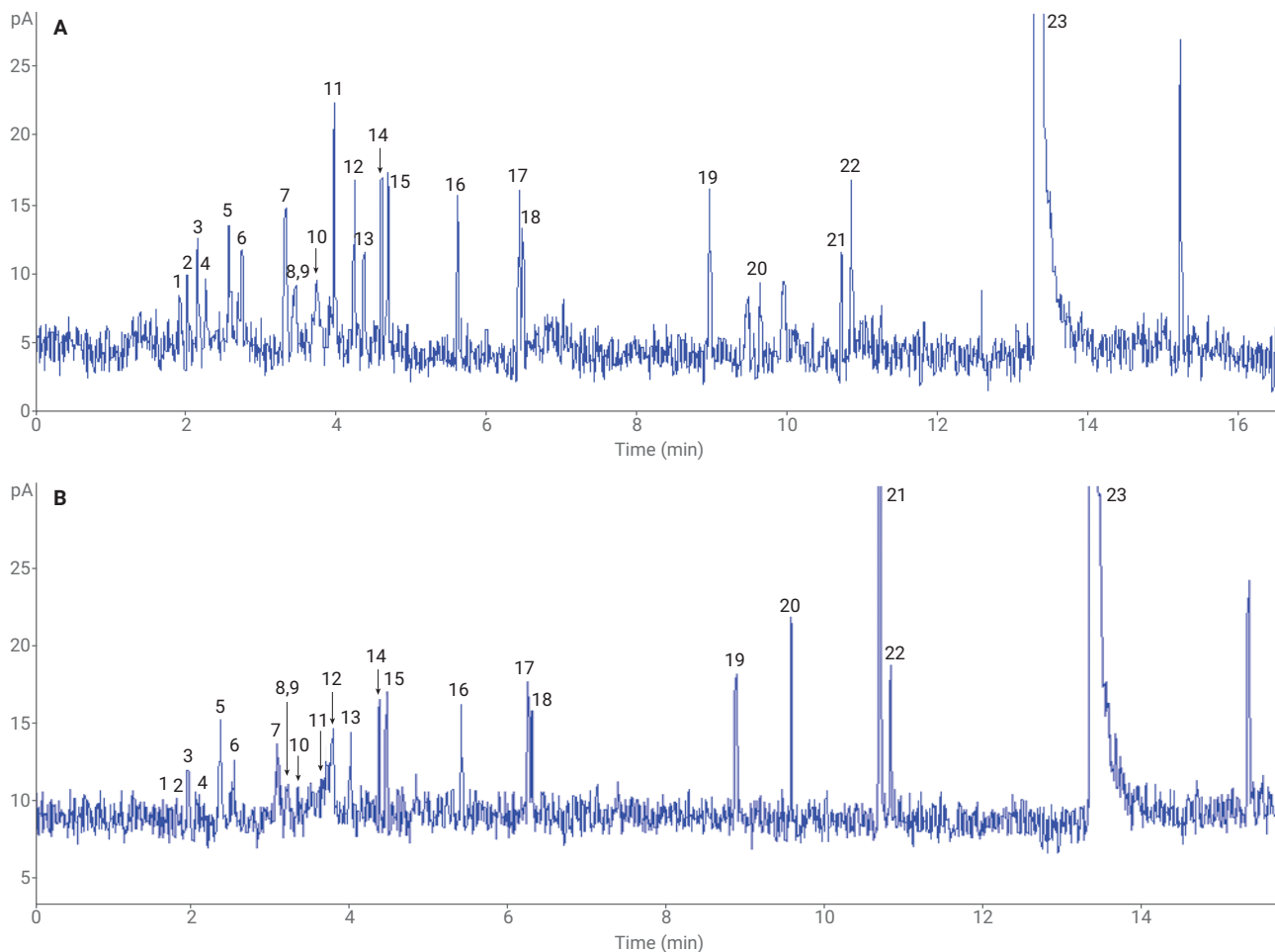


Figure 3. A 20 ppb standard with 23 sulfur-containing analytes was evaluated on an Agilent Intuvo 9000 GC system (A) and an Agilent 7890 GC system (B). Excellent signal-to-noise was achieved with both systems, indicating a practical limit of detection of 2 ppb.

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 flowpath can improve the robustness of the system by protecting the column from matrix, and eliminating the need for column trimming⁴. 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).

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 μ L	Same
Split/splitless inlet	280 °C	Same
Pulsed splitless	30 psi for 0.5 minutes	Same
Purge flow	15 mL/min at 0.5 minutes	Same
Guard Chip temperature		Default (track oven)
Bus temperature		Default
Column	Agilent HP5-MS UI 15 m \times 0.25 mm, 0.25 μ m	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

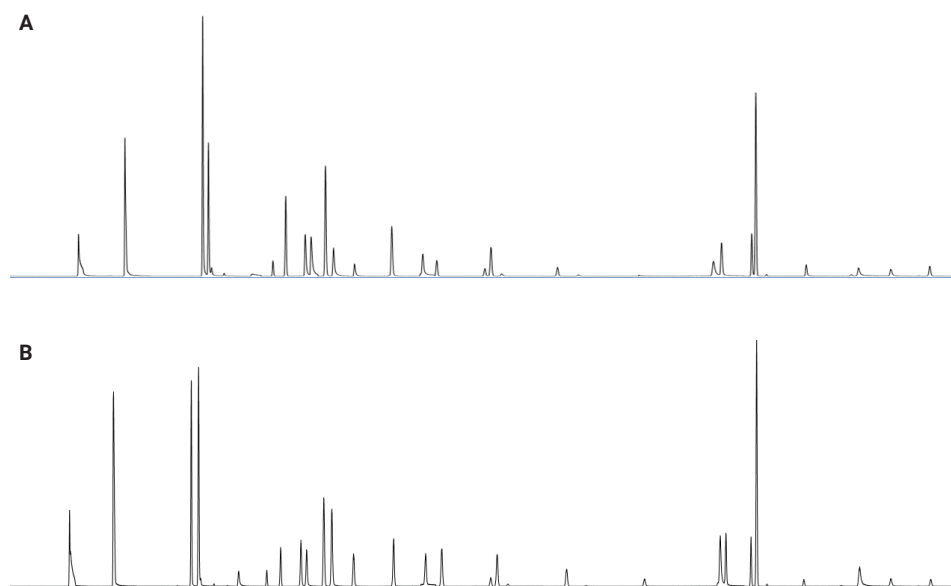


Figure 4. The Agilent Intuvo 9000 GC system (A) gave similar peak shape and responses as the Agilent 7890 GC system (B).

E-liquid analysis

Determination of nicotine, propylene glycol, and glycerol in liquids used in e-cigarettes is under development as an international standard: ISO/CD 20714⁵. The inertness of the GC flowpath contributes to the ability to achieve equivalency between a conventional GC system and the Intuvo GC system. Using the same method parameters on both a 7890B and an Intuvo resulted in excellent agreement in the results (Table 4 and Figure 5). Retention times differ by a maximum of 0.03 minutes (Table 5).

Table 4. A method based on ISO/CD 20714 was evaluated on an Agilent 7890 GC system and an Agilent Intuvo 9000 GC system. The Guard Chip and bus temperature are new features of the Intuvo GC system.

Parameter	Agilent 7890 value	Agilent Intuvo value
Injection volume	1 µL	Same
Split/splitless inlet	250 °C	Same
Split	50:1	Same
Guard Chip temperature		Default (track oven)
Bus temperature		Default
Column	DB-BAC1 UI 30 m × 0.32 mm, 1.8 µm	Same
Flow rate (hydrogen)	1.5 mL/min (constant flow)	Same
Oven program	100 °C (1 minute), then 15 °C/min to 130 °C, then 40 °C/min to 220 °C (10 minutes)	Same
Flame ionization detector temperature	275 °C	Same
Hydrogen	40 mL/min	Same
Air	400 mL/min	Same

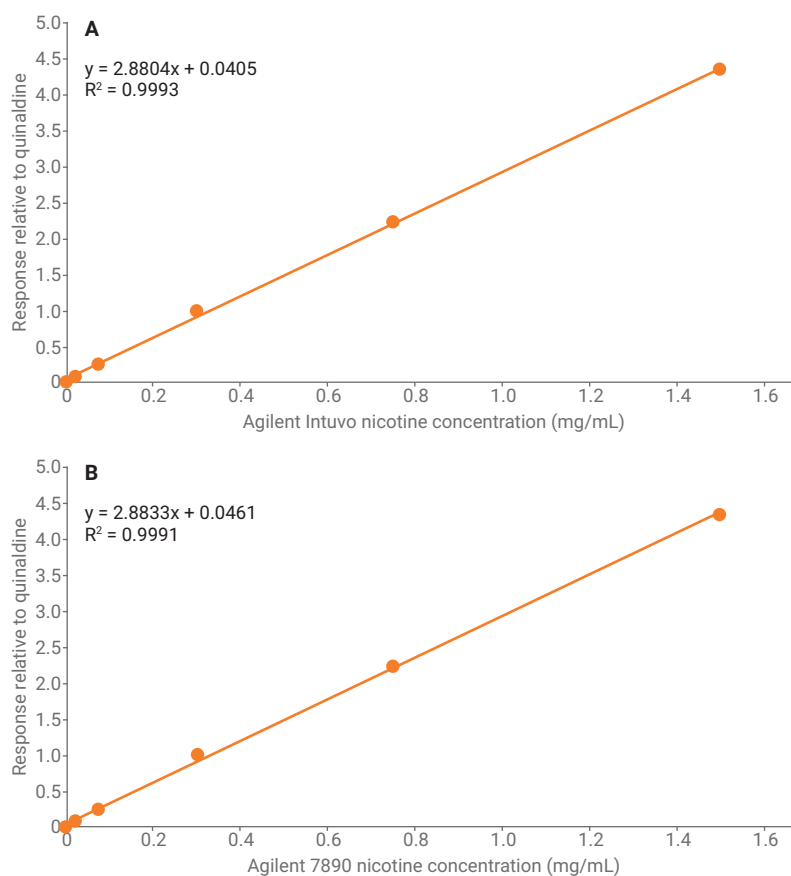


Figure 5. Calibration curves for nicotine on an Agilent Intuvo 9000 GC (A) and an Agilent 7890B GC (B) show nearly identical sensitivities.

Table 5. Retention times are repeatable for both Agilent Intuvo and Agilent 7890 GC systems.

Component	Agilent Intuvo RT (min)	Agilent 7890 RT (min)
Propylene glycol	3.34	3.37
Butanediol (ISTD 1)	4.31	4.32
Glycerol	5.00	4.98
Quinaldine (ISTD 2)	6.87	6.87
Nicotine	7.24	7.26

ASTM D7593 – Analysis of Diesel for In-Service Motor Oils

Diesel fuel contamination in lubricating oils is an important indicator of impending engine failure or required maintenance. To avoid catastrophic engine failure, technicians need a fast and robust analytical method for this measurement. ASTM method D7593 uses capillary gas chromatography (GC) to quickly quantify diesel fuel found in these oils⁶. Two recent Agilent

Application Notes describe operation and performance for the Agilent 7890 Series GC and Intuvo 9000 GC when measuring diesel contamination of in-service motor oil^{7,8}. Using the same method setpoints (Table 6), the average diesel contents and measurement precisions were calculated for five in-service motor oil samples and compared between the two platforms (Figure 6). A key to obtaining

quality results and system robustness for both systems was the use of the backflush technique for removing heavy sample matrix between runs. Intuvo smart flowpath components simplifies both the backflush hardware setup as well as routine operation while delivering the same outstanding performance as the 7890.

Table 6. Agilent 7890 and Intuvo GC system parameters motor oil analysis.

Parameter	7890 value	Intuvo value
Inlet liner	Ultra-Inert with glass wool (5190-2295)	Same
Injection volume	0.5 µL	Same
Split/Splitless Inlet	350 °C	Same
Split injection	100 mL/min	Same
Guard Chip temperature		Default (track oven)
Bus temperature		Default
Column	DB-1UI 15 m × 0.25 mm, 0.25 µm (122-0112UI)	122-0112UI-INT
Flow rate	10 mL/min (constant flow)	Same
Oven program	40 °C (0.5 minutes) then 150 °C/min to 350 °C (0.5 minutes)	Same
Flame ionization detector temperature	350 °C	Same
Post-column backflush	2-Way Splitter with Make-up (G3180B) Deactivated fused silica, 0.43 cm × 0.1 mm id (160-2635-5) Aux EPC: Helium at 27 psig for 1.45 minutes Aux EPC: Backflush at 1.45 minutes, 80 psig until end of run	D1 post column backflush flow chip (G4588-60302) PSD 1: Helium at 27 psig for 1.49 minutes PSD 1: Backflush: at 1.49 minutes, 80 psig until end of run PSD 1: Vent flow: 5 mL/min

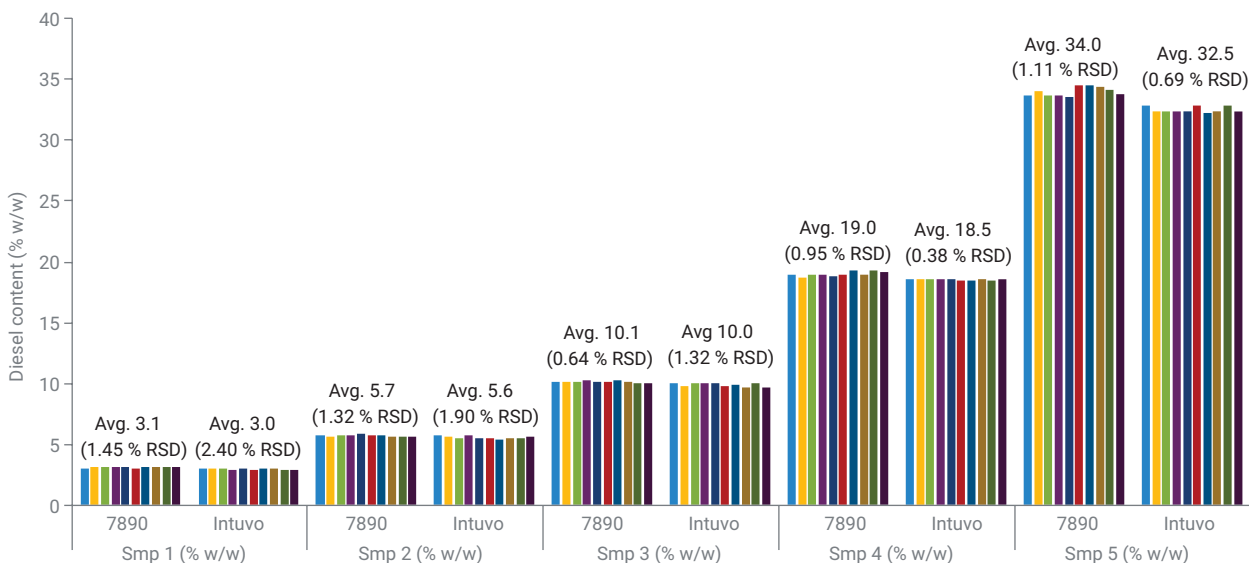


Figure 6. Comparison of 7890 and Intuvo for the analysis of diesel fuel contamination for in-service motor oil.

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 %, impacts the number of samples that can be run between maintenance and calibration events. Endrin is susceptible to isomerization to endrin aldehyde and endrin ketone at elevated temperatures. Therefore, 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 containing thermally labile analytes, the default bus temperature may need to be reduced 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 8).

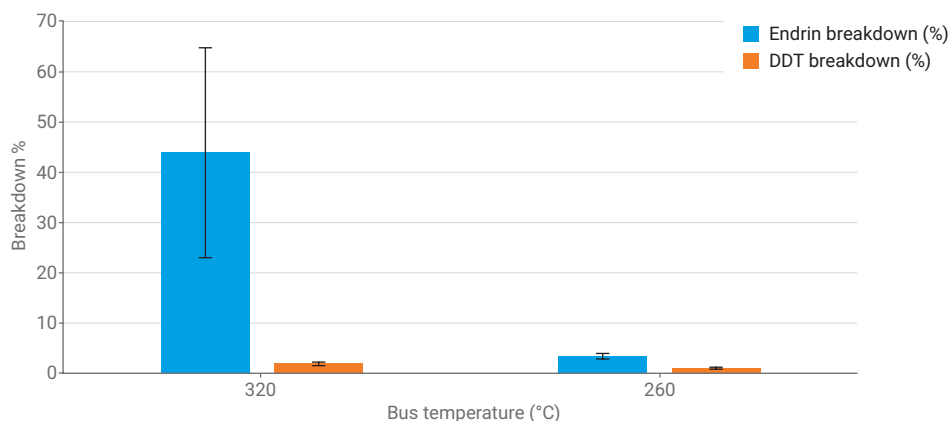


Figure 8. Endrin and DDT breakdown decreases to under 10 % when the bus temperature is lowered from 320 to 260 °C.

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 Intuvo 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 detector operation are the same. Agilent MassHunter, CDS ChemStation, and Agilent OpenLab have the same look and feel regardless of hardware platform.

Here is a high-level summary of things to consider when transferring conventional methods to Intuvo:

- A conventional method is a great way to start a method for Intuvo. You are probably more than 90 % done.
- 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 settings to be set automatically.
- Consider manually setting slightly lower temperatures than default (for example, 20 °C lower) 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**.
2. M. Giardina. Analysis of Semivolatiles Organic Compounds Using the Agilent Intuvo 9000 Gas Chromatograph, *Agilent Technologies Application Note*, publication number 5991-7256EN, September **2016**.
3. R. Veeneman. Detection of Sulfur Compounds in Light Petroleum Liquids According to ASTM D5623 with the Agilent Dual Plasma Sulfur Chemiluminescence Detector and the Agilent Intuvo 9000 GC, *Agilent Technologies Application Note*, publication number 5991-7215EN, September **2016**.
4. R. Veeneman, J. Stevens. Multiresidue Pesticide Analysis with the Agilent 9000 GC and Agilent 7000 Series Mass Spectrometer, *Agilent Technologies Application Note*, publication number 5991-7216EN, September **2016**.
5. F. David, *et al.* Determination of Nicotine, Propylene Glycol, and Glycerol in E-Liquids According to ISO/CD 20714 Using the Agilent 9000 Intuvo GC, *Agilent Technologies Application Note*, publication number 5991-8990EN, **2018**.
6. ASTM D7593-14, Standard Test Method for Determination of Fuel Dilution for In-Service Engine Oils by Gas Chromatography, *ASTM International*, West Conshohocken, PA, **2014**. www.astm.org.
7. McCurry, J.; Beard, K. ASTM D7593 – Analysis of Diesel for In-Service Motor Oils, *Agilent Technologies Application Brief*, publication number 5991-9279EN, April **2018**.
8. Beard, K.; McCurry, J. Gas Chromatographic Analysis of Diesel Fuel Dilution for In-Service Motor Oil Using ASTM Method D7593, *Agilent Technologies Application Note*, publication number 5991-9278EN, April **2018**.

www.agilent.com/chem

This information is subject to change without notice.

© Agilent Technologies, Inc. 2018
Printed in the USA, June 8, 2018
5991-9150EN

