Application Note

Petrochemicals

Chromatographic Performance of Small ID Columns with the Intuvo 9000 GC System

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Abstract

Use of small inner diameter (id) columns in gas chromatography (GC) has become more popular because of the fast and efficient separation that can be obtained over a short period of time. Use of these columns within the Agilent Intuvo 9000 gas chromatography system provides a unique challenge to the user because of the built-in retention gap (Guard or Jumper chip) and the user-defined thermal zone within the flowpath of the system. Proper use of these features can provide a high-resolution fast separation similar to those found in an Agilent 7890 gas chromatography system. This Application Note demonstrates the use of small id columns in the Intuvo 9000 GC system.

Introduction

Small id columns are often used in GC to obtain efficient separation of analytes in a short period of time. Columns with an id of 0.1 mm typically provide efficiencies on the order of 10,000 theoretical plates per meter of column, allowing fast separations with high resolution. Compared to a traditional 7890, the Intuvo 9000 GC has a built-in retention gap (Guard Chip or Jumper Chip) that analytes must travel through before reaching the head of the analytical column. This must be considered before using small id columns in the Intuvo 9000 GC system.

The addition of the Guard Chip in the Intuvo 9000 has also introduced a user-controlled thermal zone. This heated zone can be set to track the column heater, run a user-defined temperature program, or operate as an isothermal zone. The amount of time an analyte spends in the Guard Chip depends on which mode is selected. Use of a small id column in conjunction with a Guard Chip in either track oven or isothermal mode has the potential to affect column efficiency and peak shape of the analytes before sample introduction onto the analytical column.

This document summarizes the use of small id columns with Guard Chips and Jumper Chips in the Intuvo 9000 GC system using track oven and isothermal temperature modes.

Experimental

A 7890 and an Intuvo 9000 GC system were evaluated to compare column efficiencies and peak shape with use of small id columns. The same method conditions and configurations were used on both systems. A split/splitless inlet was set to 300 °C with a split/splitless single taper liner (part number 5183-4647). A 1 µL injection of a 1,000 ppm C_{10}, C_{12}, C_{14}, and C_{16} alkane mixture in hexane was used as a sample. Helium carrier gas was used in constant flow mode. The FID was set to 300 °C with air, hydrogen, and nitrogen (make up) flow at 400, 30, and 25 mL/min, respectively. Software used for instrument control was Agilent OpenLab ChemStation edition version C.01.07.

Four different column ids were used for comparison of column efficiency on the two GC systems. Each column was first run in a 7890, then tested in the Intuvo form factor on an Intuvo 9000 GC system. The column flow rate was varied to maximize column efficiency with each type of column tested. Use of greater column flow rates required adjustment of the split ratio (from 2,500:1 to 1,000:1) depending on the type of column and flow rate used. Table 1 shows the four different types of columns with their respective split flow and column flow rates.

An isothermal oven method was used to obtain column efficiency data for the columns tested (oven held at 140 °C for nine minutes). A temperature program was also investigated (oven program: 40 °C, 0.5 minutes hold; 25 °C/min to 190°C, one minute hold) to demonstrate a typical GC method. Guard Chips (part number G4587-60565) and Jumper Chips (part number G4587-60675) were evaluated in isothermal (300 °C) and track-oven modes in the Intuvo 9000 GC.

Table 1. Columns and split ratios used.

Column id	Column	Split Ratio	Flow Rate
0.1 mm	DB-5 10 m × 100 µm, 0.1 µm (part number 127-5012)	2,500:1	0.4 mL/min
0.18 mm	DB-5 10 m × 180 µm, 0.18 µm (part number 125-5012)	2,000:1	0.6 mL/min
0.2 mm	DB-5 15 m × 200 µm, 0.2 µm (part number 128-50H7)	1,000:1	1 mL/min
0.25 mm	DB-5 15 m × 250 µm, 0.1 µm (part number 122-5011)	1,000:1	1.2 mL/min

Results and discussion

Oven isothermal results

The Intuvo 9000 GC provides many advantages not found in a conventional 7890 air bath oven. One of these is the built-in retention gap (Guard Chip), which allows the user to protect the GC column from sample contamination. The Intuvo GC also provides easy maintenance with replaceable flowpath chips, allowing the user to replace parts of the flowpath without cutting the analytical column. The built-in retention gap (Guard Chip or Jumper Chip) also provides users with a new temperature-controlled thermal zone that can be set to track the GC oven, run a user-defined temperature program, or set isothermally. Depending on the setting of this new temperature zone in conjunction with use of small id columns, the conditions may influence the peak profiles obtained from the GC system, leading to the possibility of analyte band broadening as analytes move through the system. Results presented below provide guidance on how to set this thermal zone to reduce analyte band broadening while using a small id column in the Intuvo GC system.

Initial experiments were performed on the 7890 and Intuvo 9000 GC with the oven and Guard Chip or Jumper Chip heated isothermally (oven set to 140 °C and Guard Chips and Jumper Chips set to the inlet temperature). Figure 1 demonstrates those conditions using the 0.1 mm id column. Results with a Jumper Chip, under these isothermal conditions, were similar to results obtained with a 7890, providing narrow peaks with little analyte band broadening.

Switching to a Guard Chip with the same isothermal oven conditions results in considerable analyte band broadening. Because of the extra column volume associated with the Guard Chip used in conjunction with a 0.1 mm id column, substantial analyte band broadening occurred as the analytes moved through the system. Use of isothermal conditions in the GC oven does not allow analytes to refocus on the head of the analytical column and recover from band broadening that may occur in the Guard Chip. Use of isothermal oven conditions with a Guard chip and a 0.1 mm id column provides the greatest challenge for analyte band broadening as analytes move through the system.

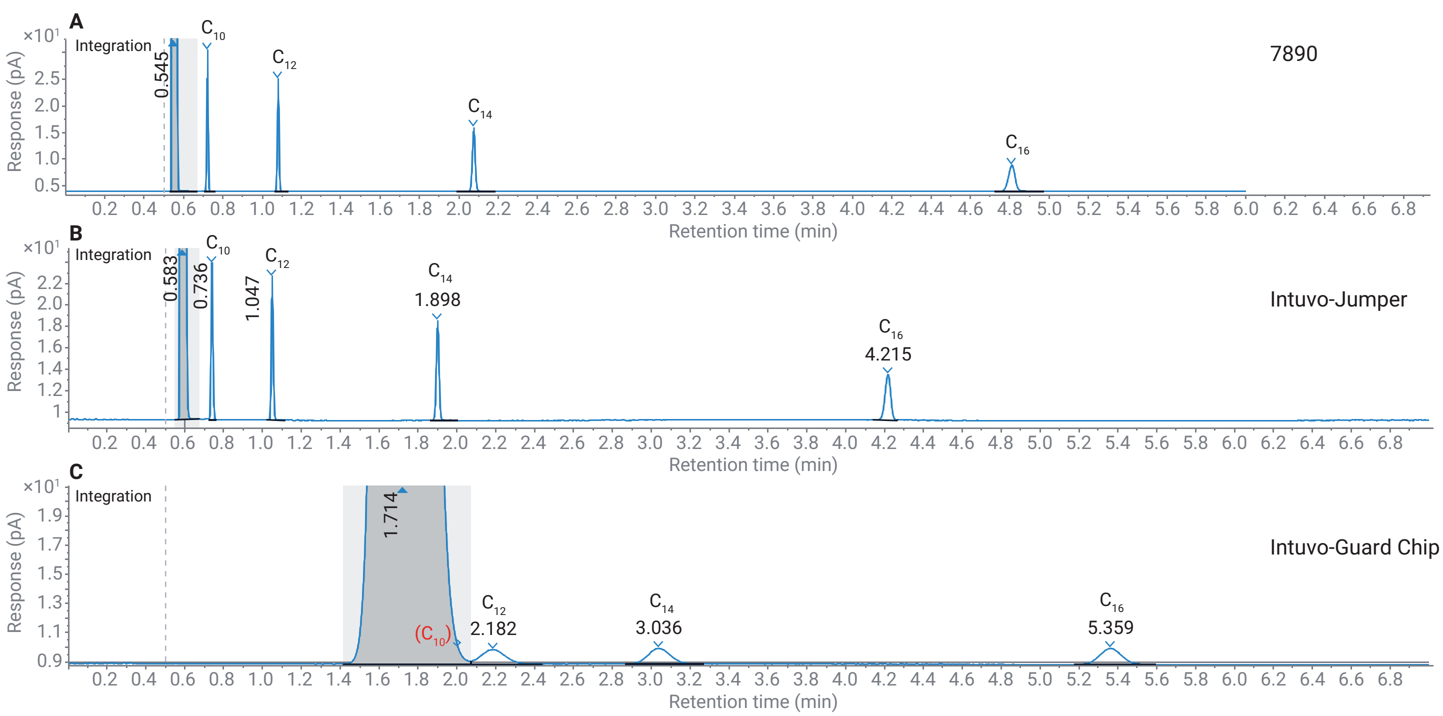

Figure 1. Use of an isothermal oven with a 0.1 mm id column (Jumper Chip/Guard Chip used in isothermal mode).

Increasing the column size to a 0.18 mm id was next investigated. Using a larger id column increased the column flow, while still obtaining a similar optimal linear velocity as with a 0.1 mm id column. Figure 2 shows the results from the 0.18 mm id column. As with the 0.1 mm id column, increasing the column id in conjunction with use of a Jumper Chip provided peak shape and column efficiencies similar to those obtained with a 7890. These experiments were also completed with a Guard Chip and 0.18 mm id column installed. As expected, using the 0.18 mm id column and increasing the carrier gas flow through the system reduced the amount of analyte band broadening that occurred in the Guard Chip, providing better peak shape compared to the 0.1 mm id column. Because of the increased flow through the system, analytes spent less time in the Guard Chip, reducing the amount of analyte band broadening.

Use of larger id columns continued to show improved results with isothermal oven conditions. Table 2 provides a summary of these results with respect to C_{16} for each of the columns tested with isothermal conditions.

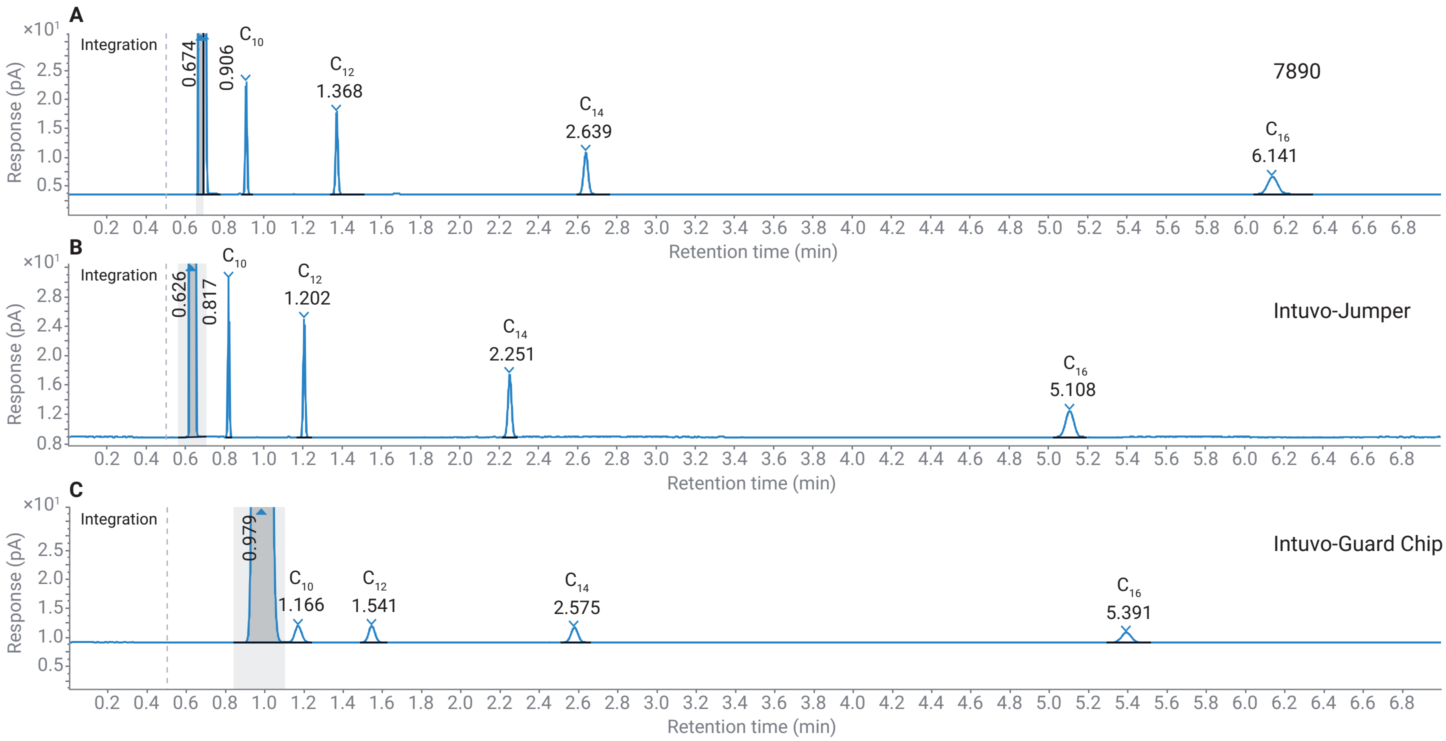

Figure 2. Use of an isothermal oven with a 0.18 mm id column (Jumper Chip/Guard Chip used in isothermal mode).

Table 2. Isothermal oven conditions data for C_{16}.

Column id	C_{16}	Retention Time (min)	Symmetry	Theoretical Plates Per Meter	Theoretical Plate Per Meter (Percent Normalized to an Agilent 7890)
0.100 mm	Agilent 7890	4.8	1.1	9733	
	Intuvo Jumper	4.2	1.2	9943	102
	Intuvo Guard Chip	5.4	0.9	1015	10
0.180 mm	7890	6.1	1.0	6222	
	Intuvo Jumper	5.1	1.0	6426	103
	Intuvo Guard Chip	5.4	1.0	5049	81
0.200 mm	7890	7.1	1.0	5502	
	Intuvo Jumper	5.9	1.0	5304	96
	Intuvo Guard Chip	6.1	1.0	5038	91
0.250 mm	7890	4.0	1.0	5032	
	Intuvo Jumper	3.3	1.0	5049	100
	Intuvo Guard Chip	3.4	1.0	4782	95

Column efficiencies were also calculated for comparison back to a 7890 using the same dimension columns. Table 2 shows that using the Jumper Chip provided similar column efficiencies as those found in the 7890. Use of a Jumper Chip with isothermal oven conditions will always provide better peak shape, especially with the use of smaller column ids (0.1 mm), compared to the same oven isothermal conditions with the use of a Guard Chip. As for the Guard Chip, column efficiencies continued to improve with increasing column diameter. Use of the 0.25 mm id column provided similar column efficiencies as those found in a 7890. If use of a Guard Chip and isothermal oven conditions are required, the user should consider use of a larger id column to help reduce the effects of band broadening that may occur in the Guard Chip.

Oven temperature program results

Typical GC methods use an oven temperature program for their analyses. Experiments were also run using an oven temperature program to determine the effects this would have on analyte peak shape. Using an oven temperature program allows analytes to refocus on the head of the GC column, thus reducing the effect of analyte band broadening that may occur in the Jumper Chip or Guard Chip (experiments with the Jumper Chips and Guard Chips in isothermal and track oven mode were also carried out). Figure 3 shows the results with use of a 0.1 mm id column installed with a Jumper Chip and an oven temperature program. As expected, peak widths and symmetries obtained are comparable to results with a 7890. As with previous experiments, larger id columns were also investigated. Table 3 presents data from these additional experiments with respect to C_{16}.

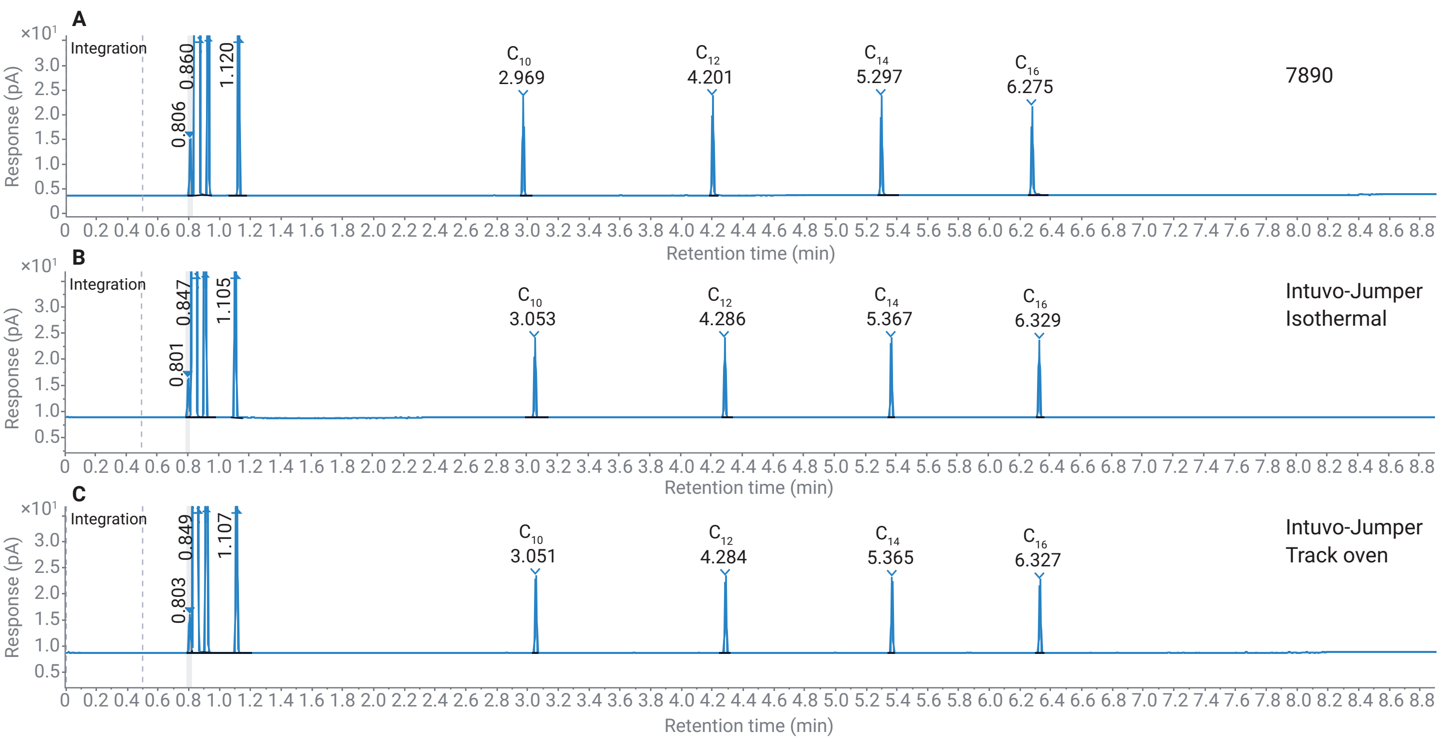

Figure 3. Use of an oven temperature program with a 0.1 mm id column (Jumper Chip used in both isothermal and track oven mode).

Using an oven temperature program with a Guard Chip reduces analyte band broadening. The first experiment completed consisted of a 0.1 mm id column and a Guard Chip in track oven and isothermal modes with an oven temperature program (Figure 4). Figure 4 shows that improved peak shape and reduced analyte band broadening was obtained with the Guard Chip heated isothermally and in track oven mode. Because analytes have a chance to refocus on the head of the GC column, the effects of band broadening are reduced. Analytes with lower boiling points, such as C_{10} in Figure 4, will not completely refocus on the analytical column, thus slightly more band broadening may occur. Other heavier analytes such as C_{16} easily recondense on the head of the GC column, recovering from any analyte band broadening that may have occurred in the Guard Chip.

Table 3. Oven temperature program data for C_{16} with Jumper Chip in isothermal and track oven mode.

Column id	C_{16}	Retention Time (min)	Width-1/2 Height (min)	Symmetry
0.100 mm	Agilent 7890	6.3	0.01	0.8
	Intuvo Jumper-Isothermal	6.3	0.01	1.0
	Intuvo Jumper-Track Oven	6.3	0.01	1.0
0.180 mm	7890	6.5	0.02	0.8
	Intuvo Jumper-Isothermal	6.5	0.01	1.0
	Intuvo Jumper-Track Oven	6.5	0.01	1.0
0.200 mm	7890	6.8	0.01	0.9
	Intuvo Jumper-Isothermal	6.7	0.01	1.0
	Intuvo Jumper-Track Oven	6.7	0.01	1.0
0.250 mm	7890	6.1	0.01	0.9
	Intuvo Jumper-Isothermal	6.1	0.01	1.0
	Intuvo Jumper-Track Oven	6.1	0.01	1.0

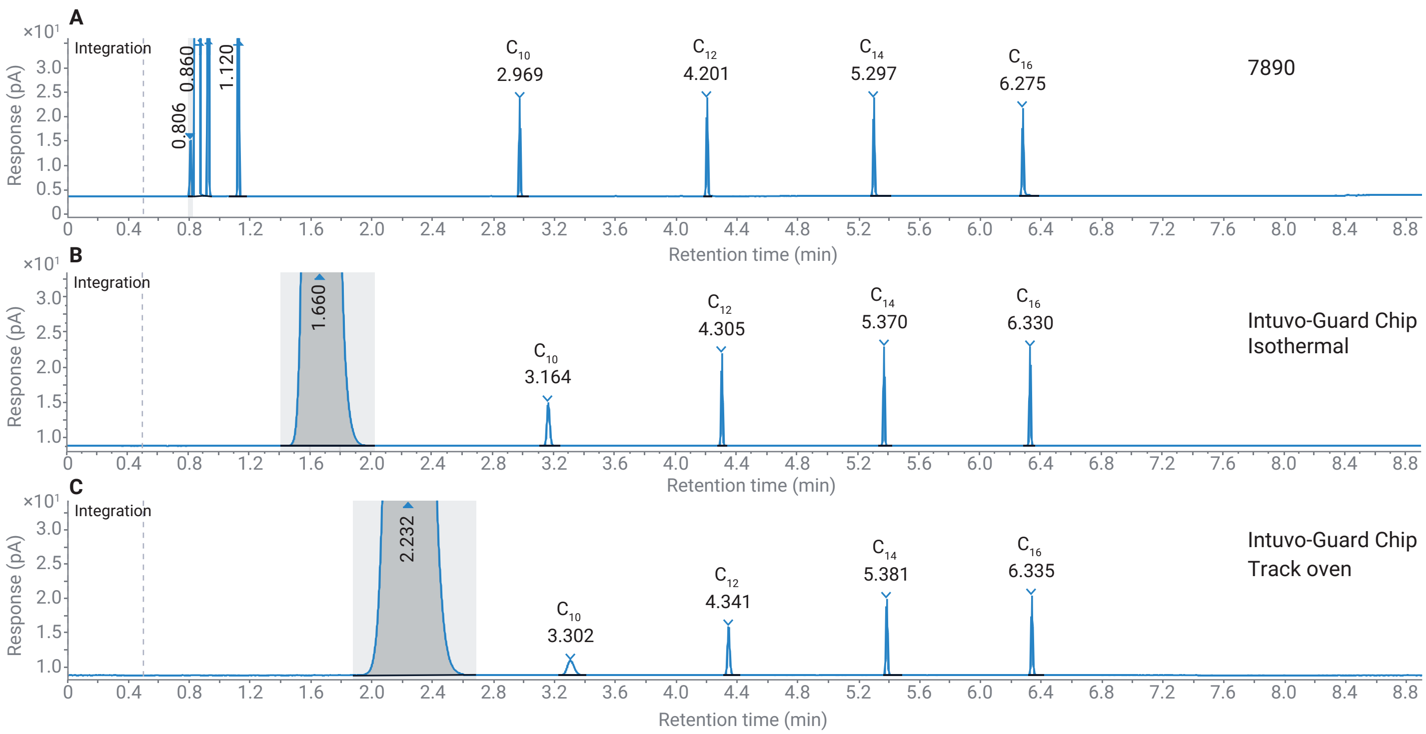

Figure 4. Use of an oven temperature program with a 0.1 mm id column (Guard Chip used in both isothermal and track oven mode).

As with the oven isothermal experiments, increasing the column id with an oven temperature program continued to improve peak shape. Figure 5 demonstrates the use of a 0.18 mm id column installed with a Guard Chip and an oven temperature program. The 0.18 mm id column with a Guard Chip in isothermal or track oven mode shows negligible amounts of band broadening. As with the oven isothermal experiments (Figures 1 and 2), greater column flow used with the 0.18 mm id column allowed for less band broadening to occur as the analytes moved through the Guard Chip. Table 4 summarizes the results with the various columns, and compares the retention time, peak width at half height, and peak symmetry for C_{16}. For this well retained analyte, the chromatographic results are nearly identical. Using an oven temperature program allows analytes to refocus on the head of the analytical column, thus reducing any analyte band broadening that might have occurred within the Guard Chip. Temperature programming the Guard Chip allows the user to best use the analyte matrix trapping ability of the Intuvo, thus preserving the analytical column.

Table 4. Oven temperature program data for C_{16} with Guard Chip in isothermal and track oven mode.

Column id	C_{16}	Retention Time (min)	Width-1/2 Height (min)	Symmetry
0.100 mm	Agilent 7890	6.3	0.01	0.8
	Intuvo Jumper-Isothermal	6.3	0.01	1.0
	Intuvo Jumper-Track Oven	6.3	0.01	1.0
0.180 mm	7890	6.5	0.02	0.8
	Intuvo Jumper-Isothermal	6.5	0.01	1.0
	Intuvo Jumper-Track Oven	6.5	0.01	1.0
0.200 mm	7890	6.8	0.01	0.9
	Intuvo Jumper-Isothermal	6.7	0.01	1.0
	Intuvo Jumper-Track Oven	6.7	0.01	1.0
0.250 mm	7890	6.1	0.01	0.9
	Intuvo Jumper-Isothermal	6.1	0.01	1.0
	Intuvo Jumper-Track Oven	6.1	0.01	1.0

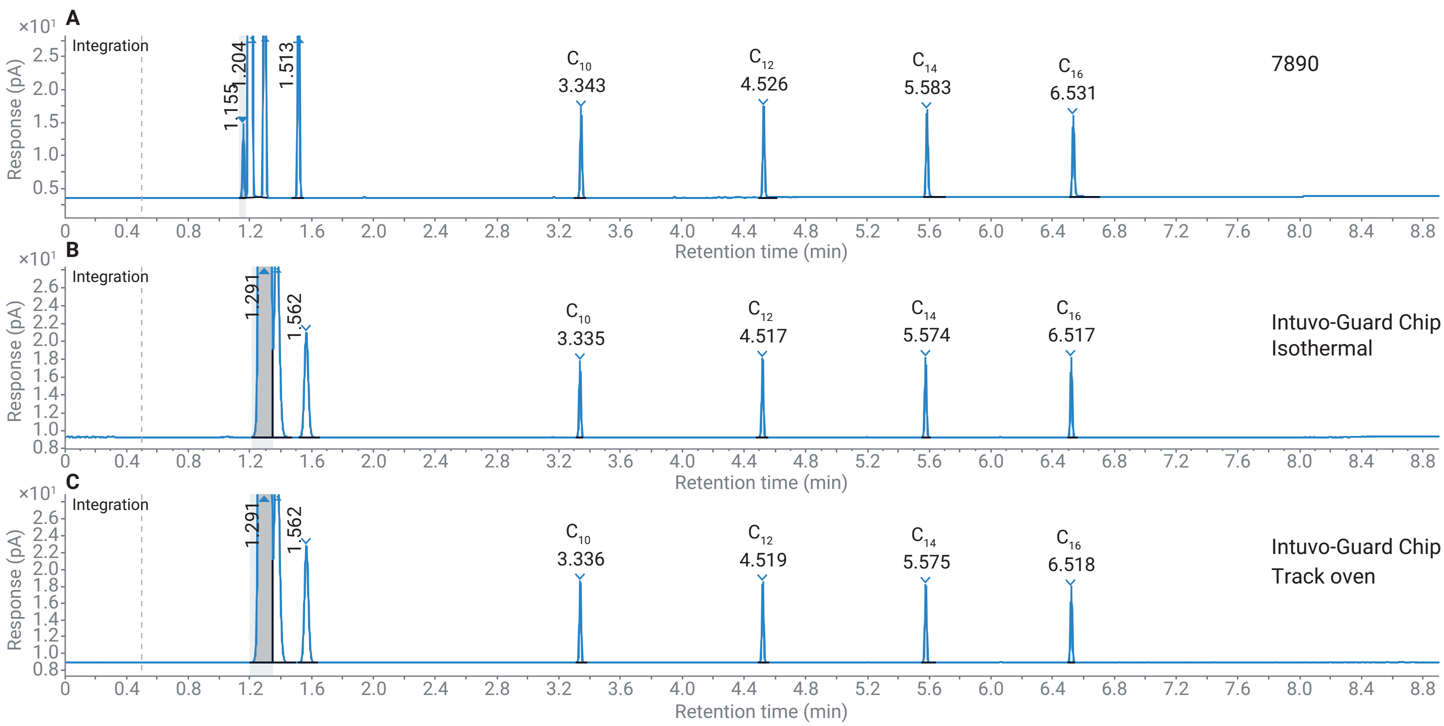

Figure 5. Use of an oven temperature program with a 0.18 mm id column (Guard Chip used in both isothermal and track oven mode).

Conclusion

The advantages of having a built-in retention gap in the Agilent Intuvo 9000 far outweigh the potential challenges faced when using a small id column in the GC system, and are easily overcome with proper selection of flow chip and method parameters. When using columns with an id greater than 0.2 mm, analyte band broadening is not an issue. If use of a column smaller than 0.2 mm id is required, the effects of extra column band broadening can be minimized. Using a Jumper Chip over a Guard Chip results in reduced extra-column volume, and thus reduced analyte band broadening. If a method requires the use of isothermal oven conditions, then a Jumper Chip should be used. This helps reduce the amount of analyte band broadening. If a Guard Chip is required (because of a dirty analyte matrix), then an oven temperature program or increasing the column id should be considered to aid in minimizing the effects of analyte band broadening that may occur with small id columns.

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