New High Temperature PEG GC Column with Increased Temperature Limit and Ultra-Low Bleed Level

Vanessa Abercrombie, Ngoc-A Dang, John Oostdijk, Frans Biermans, Laura Provoost, Daron Decker, Phil Stremple

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Introduction

Today's demanding GC and GC/MS applications mainly focus on sensitive and reproducible qualitative and quantitative analysis of more challenging analytes. Gas chromatography columns based on 100% Polyethylene Glycol (PEG) stationary phase, commonly referred to as WAX columns, can be used for a wide variety of applications, such as industrial chemical analysis. In comparison to polysiloxane stationary phases the maximum operating temperature of a traditional WAX GC column is much lower, mainly up to 250°C/260°C.

A new innovative WAX column is developed, with improved thermal stability, giving better column-to-column reproducibility over the life of the column. The Agilent J&W DB-HeavyWAX has an extended temperature limit, up to 280°C/290°C, and provides several advantages in the analysis of industrial chemicals and pyrolysis gasoline.

Its good inertness and ultra-low bleed level are suitable for the most sensitive GC and GC/MS applications and can be used as 2nd dimension polar column with extended upper temperature limits in comprehensive GC×GC applications.

Experimental

For the comparison study, a 100 ppm standard of BTEX, a commonly analyzed in industrial chemical applications, was injected onto a commercially available WAX column, as well as the Agilent J&W DB-HeavyWAX. In both cases, the columns were held isothermal until the final peak, o-xylene eluted, then ramped to a final temperature of 280 C, and held for one hour.

GC Conditions

Column	**Agilent J&W DB-HeavyWAX** 30m x 0.25 mm x 0.25 µm
Carrier	**Helium, constant flow, 1 mL/min**
Oven	**70°C (10.0 min), Ramp 5°C/min to 120°C (1.0 min), Ramp 20°C/min to 280°C (60 min)**
Inlet	**Split mode, 250°C, split ratio 200:1**
Inlet Liner	**Ultra Inert, split, low pressure drop, glass wool (p/n 5190-2295)**
GC/FID	**Agilent 7890B GC equipped with FID**
Sampler	**Agilent 7693 autosampler**

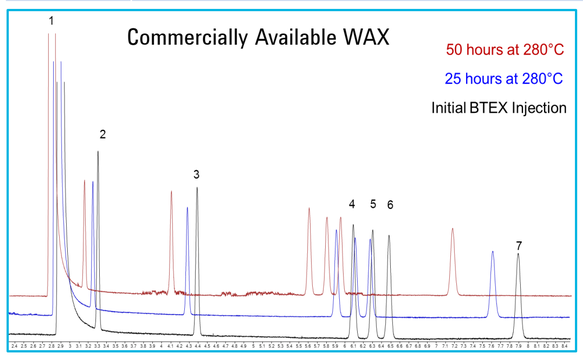

Figure1. Commercially available WAX column shows retention time shift after use at 280°C.

Peak identification: 1. Methanol, 2. Benzene, 3. Toluene, 4. Ethylbenzene, 5. p-Xylene, 6. m-Xylene, 7. o-Xylene

Retention Time Stability

Figure 2 demonstrates the improved thermal stability of the Agilent J&W DB-HeavyWAX column, when operating at high temperatures for extended periods of time. Even after fifty hours of use at 280°C, the retention times have not shifted.

Figure 3 compares the retention time shift of o-Xylene over 100 hours of use at 280 °C on two commercially available WAX columns in comparison to the DB-HeavyWAX. After 100 hours of use at 280°C, both Wax column "A" and Wax column "B" have had a retention time shift of around two minutes, while the Agilent J&W DB-HeavyWAX remains consistent.

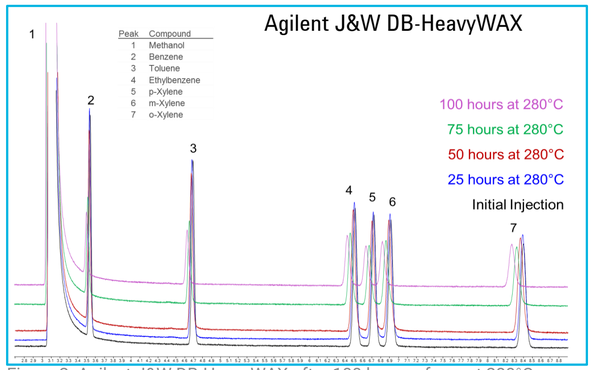

Figure 2. Agilent J&W DB-HeavyWAX after 100 hours of use at 280°C.

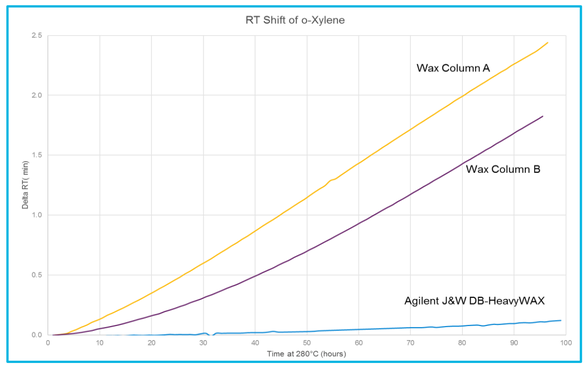

Figure 3. Retention Time shifting of o-Xylene on two commercially available WAX columns when used at 280°C for up to 100 hours and compared to the Agilent J&W DB-HeavyWAX.

Application: Pyrolysis Gasoline

For applications, such as the analysis of pyrolysis gasoline (PyGas) having a maximum temperature limits of 250°C/260°C results extended runtimes avoid carry over. Figure 4 demonstrates a sample of PyGas on the Agilent J&W DB-HeavyWAX. Having the ability to increase the temperature program to 280°C/290°C this provides an option to have these compounds elute at the end of the analysis.

In Figure 5 you can see that unlike traditional WAX columns, the Agilent J&W DB-HeavyWAX has less bleed at 280°C than a traditional WAX column does at 250 °C.

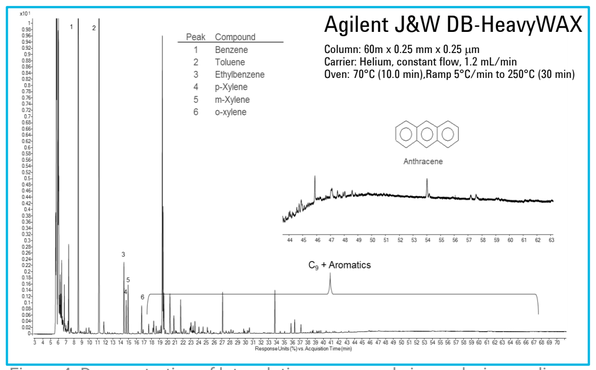

Figure 4. Demonstration of later eluting compounds in pyrolysis gasoline.

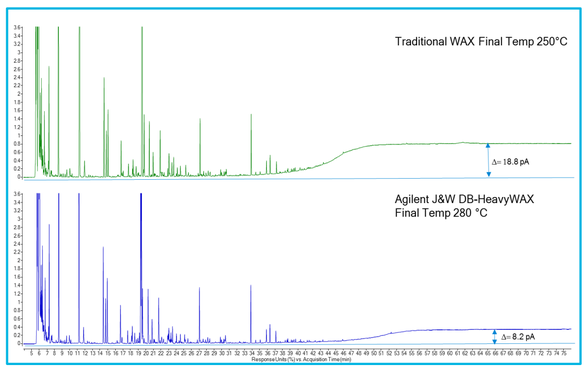

Figure 5. Comparison of a sample of pyrolysis gasoline run on a traditional Wax column with a final temperature of 250°C and DB-HeavyWAX with a final temperature of 280°C, and their associated column bleed levels.

Ultra-Low Column Bleed

It's a known phenomena, bleed levels of WAX type GC columns is much higher compared to polysiloxane type stationary phases. When WAX columns are used above it's maximum temperature, this is even more dramatic. Figure 6 shows bleed level comparisons of Agilent J&W DB-HeavyWAX to two commercially available WAX columns at 280°C.

After 100 hours of use at high temperatures, 280°C, the column bleed level of the Agilent J&W DB-HeavyWAX is still less than 10 pA, while the two commercially available WAX columns have column bleed levels between 50 and 60 pA.

Competitor WAX columns can't compete with the decreased bleed at high temperatures over extended periods of time of the Agilent J&W DB-HeavyWAX.

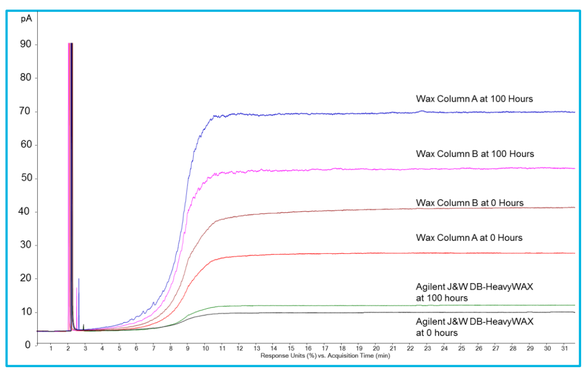

Figure 6. Bleed profiles for commercially available WAX columns and Agilent J&W DB-HeavyWAX over 100 hours of use at 280 °C

Similar Selectivity to DB-Wax

DB-Wax methods for industrial chemicals can easily be switched to the Agilent J&W DB-HeavyWAX, without modifying existing methods or updating retention times.

Similar selectivity, with the improved temperature range, bleed, and thermal stability allow you to increase your analyte range.

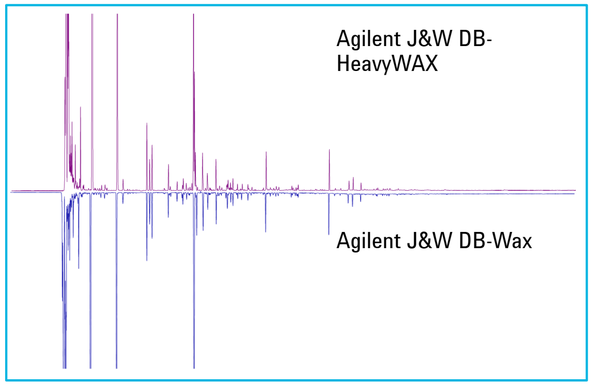

Figure 7. A sample of pyrolysis gasoline was run on an Agilent J&W DB-HeavyWAX (60m x 0.25 mm x 0.25 µm) and Agilent J&W DB-Wax (60m x 0.25 mm x 0.25 µm) by ASTM D6563 to compare selectivity.

Conclusions

The Agilent J&W DB-HeavyWAX column shows that it is a valuable addition to the WAX column family because it provides an increased maximum temperature range without having to sacrifice thermal stability and ultra-low bleed level.

The column's extended upper temperature limit, to approximately 280°C/290°C, provides significant benefits for conventional GC, GC/MS and comprehensive GC×GC applications. For application with late eluting compounds of heavy matrix, this will save time analyzing the samples.

Columns are available in various configurations
Spring of 2018

References

1. ASTM D2306 "Standard Test Method for C8 Aromatic Hydrocarbon Analysis by Gas Chromatography"

2. ASTM D6563 "Standard Test Method for Benzene, Toluene, Xylene (BTX) Concentrates Analysis by Gas Chromatography"

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