Application Note

Industrial Chemicals

# Increased thermal stability and maximum temperature of the Agilent J&W DB-HeavyWAX column


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## Abstract

One-hundred percent polyethylene glycol columns, also known as WAX columns, are used for a wide variety of applications, such as industrial chemicals, flavors, and fragrances. Traditional WAX columns have a maximum temperature limit of 250/260 °C due to issues such as decreased thermal stability, which reduces the potential range of applications. The Agilent J&W DB-HeavyWAX has an extended temperature limit, up to 280/290 °C, and increased thermal stability, which increases injection-to-injection reproducibility.

## 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 a 100 % polyethylene glycol (PEG) stationary phase can be used for a wide variety of applications such as industrial chemical analysis. Compared to polysiloxane stationary phases, the maximum operating temperature of a traditional WAX GC column is much lower, up to 250/260 °C. When a traditional WAX GC column is taken above the stated maximum allowed operating temperature (MAOT) it exhibits high bleed levels and poor thermal stability, which are observed in retention time shifts.

The Agilent J&W DB-HeavyWAX has an extended temperature limit, up to 280/290 °C, which allows for an increased application range, and provides increased thermal stability. Benzene, toluene, ethylbenzene, *p*-xylene, *o*-xylene, and *m*-xylene, often referred to as BTEX, are some of the critical industrial chemicals frequently analyzed on columns with a PEG stationary phase. In the analysis of industrial chemicals (ASTM D2306[1] and D6563[2]), it is not uncommon for higher boiling compounds to be present. Previously, the only option to elute the heavier compounds was to hold the column at the maximum temperature for an extended time, or take the column above the MAOT and risk damaging the phase[3].

With the extended temperature limit of the DB-HeavyWAX, it is possible to sustain a higher final temperature of 280 °C, and maintain the thermal stability of the column phase. We compared the thermal stability of a traditional WAX column with the DB-HeavyWAX, using BTEX to show the improved thermal stability of the DB-HeavyWAX. This comparison demonstrated the improved stability of the DB-HeavyWAX when compared to traditional PEG phase columns, allowing increased temperature range without loss of stability.

## Materials and methods

- Agilent 7890 GC/FID equipped with a split/splitless inlet
- Agilent 7693 autosampler
- Agilent MassHunter control software

### Standards preparation

Pure benzene, toluene, ethylbenzene, *p*-xylene, *o*-xylene, and *m*-xylene (BTEX) were purchased from Sigma-Aldrich. A standard of 100 ppm each was prepared in methanol, purchased from Sigma-Aldrich.

### Instrument conditions

| GC Conditions | |
|---|---|
| Column | Agilent J&W DB-HeavyWAX, 30 m × 0.25 mm, 0.25 µm (p/n 122-7132)<br>Commercially available WAX A, 30 m × 0.25 mm, 0.25 µm<br>Commercially available WAX B, 30 m × 0.25 mm, 0.25 µm |
| Carrier | Helium, constant flow, 1 mL/min |
| Oven | 70 °C (10.0 minutes),<br>Ramp 5 °C/min to 120 °C (1.0 minute),<br>Ramp 20 °C/min to 280 °C (60 minutes) |
| 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 |
| **FID Conditions** | |
| Temperature | 280 °C |
| Hydrogen | 30 mL/min |
| Air | 400 mL/min |
| Column + make-up | 25 mL/min |

## Results and discussion

A BTEX standard was injected onto a commercially available WAX column. The DB-HeavyWAX was held isothermally until the final peak, *m*-xylene, eluted, then was ramped to a final temperature of 280 °C, and held for one hour.

Figure 1 demonstrates the shortcomings of traditional WAX columns that have maximum operating temperatures of 250/260 °C. When taken above their stated MAOT, the stationary phase will start to change; one of the results is retention time shifting. This commercially available WAX column has an MAOT of 250 °C isothermal, and 260 °C programmed. When used above its MAOT, up to a temperature of 280 °C, the column will immediately show signs of retention time shifting. The shift in retention time will change from slight to significant after 50 hours of use at 280 °C.

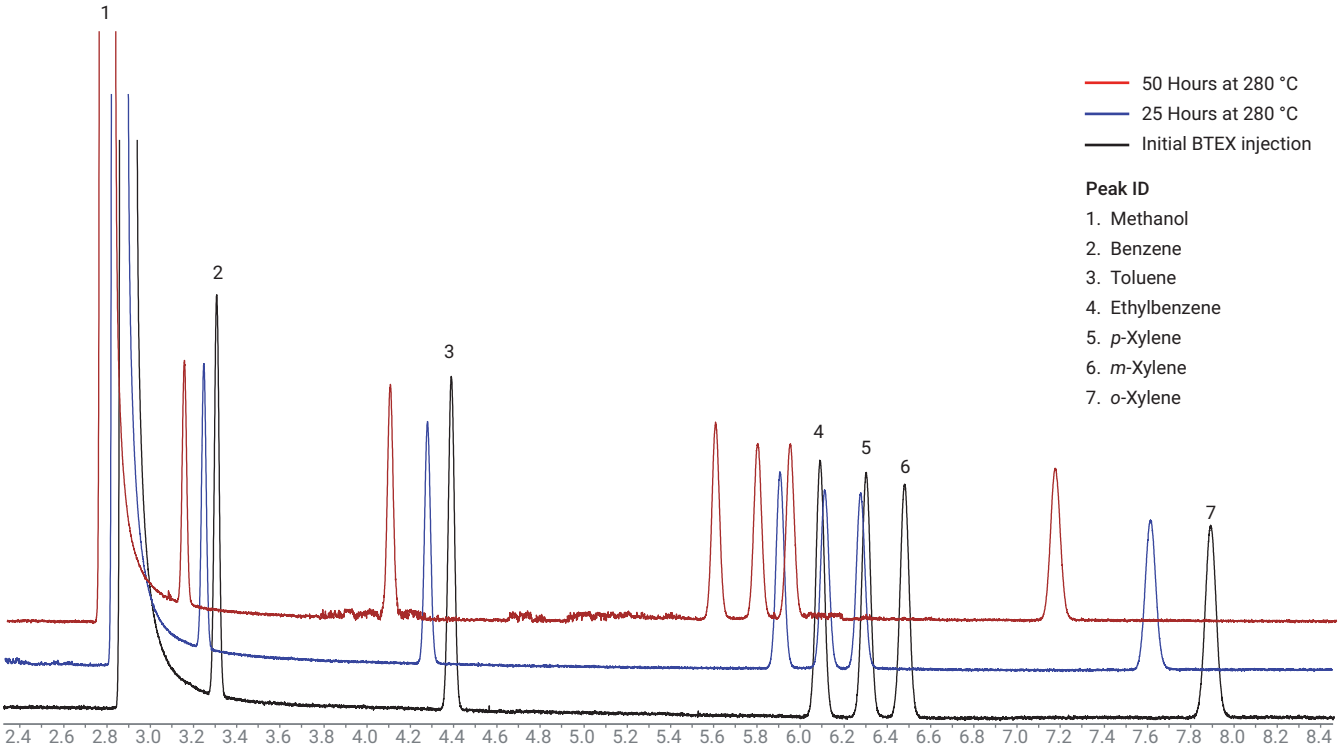


**Figure 1.** A commercially available WAX column shows retention time shift after use at 280 °C.

Figure 2 demonstrates the improved thermal stability of the DB-HeavyWAX column when operating at high temperatures for extended periods of time. Even after 50 hours of use at 280 °C, the retention times have not shifted. After 100 hours of use at 280 °C, there is a shift of a few seconds. This shift demonstrates the improved thermal stability of the DB-HeavyWAX column relative to the commercially available WAX column.

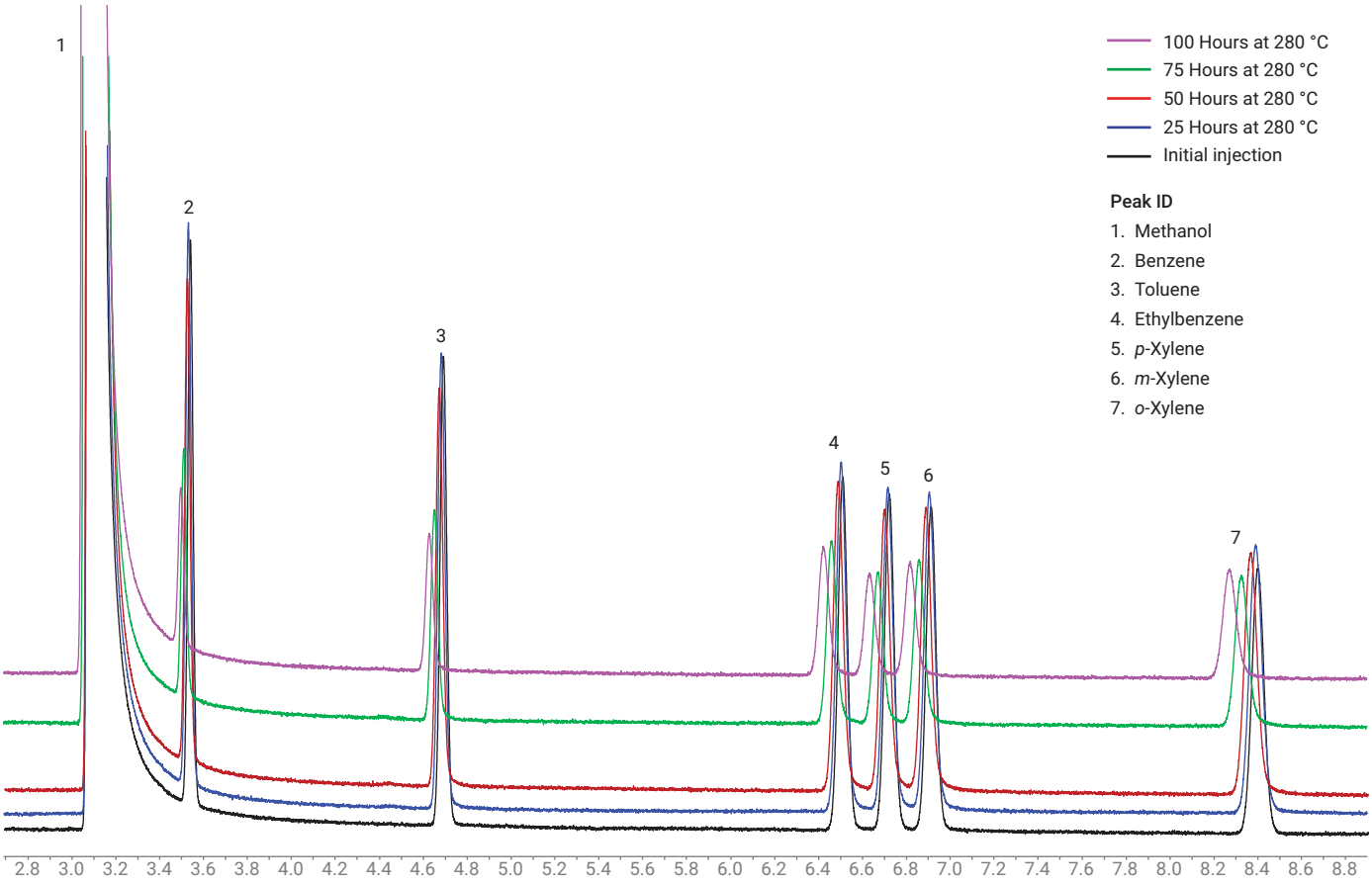


**Figure 2.** Agilent J&W DB-HeavyWAX column after 100 hours of use at 280 °C. The Y-axis is offset.

Figure 3 compares the retention time shift of the final eluting peak, *o*-xylene, on the DB-HeavyWAX with two commercially available WAX columns when operated at 280 °C for 100 hours. After 100 hours at 280 °C, WAX column A and WAX column B had a retention time shift of approximately two minutes, while the DB-HeavyWAX remains consistent. The retention time stability of the DB-HeavyWAX, compared to the two commercially available WAX columns, demonstrates the improved performance and stability of the column.

## Conclusion

The Agilent J&W DB-HeavyWAX column provides an increased maximum temperature range without having to sacrifice thermal stability. Traditional WAX columns often cannot maintain retention time stability at high temperatures, such as 280 °C, for extended periods of time. The higher temperature limit of the DB-HeavyWAX provides a more thermally stable column, even after operating at 280 °C for 100 hours.

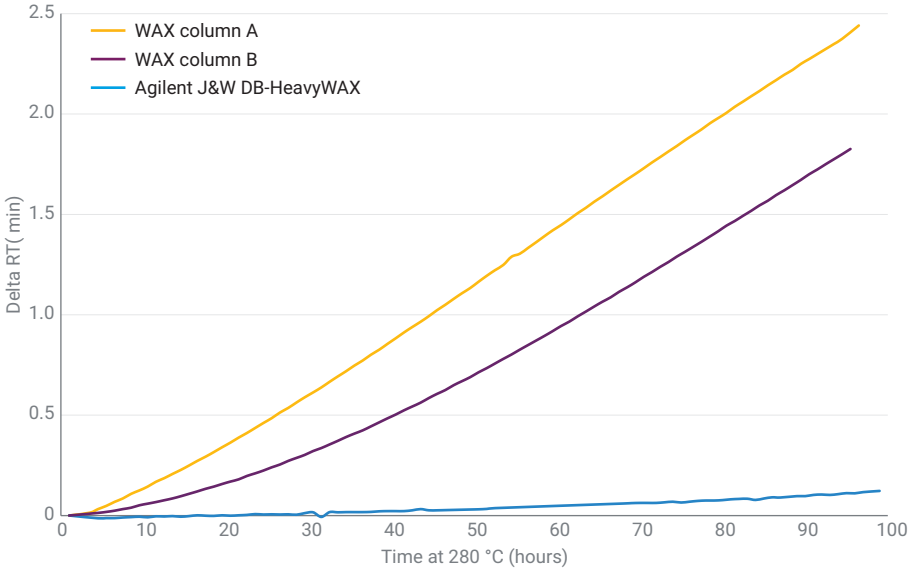


**Figure 3.** Retention time shifting of *o*-xylene on two commercially available WAX columns when used at 280 °C for up to 100 hours, compared to an Agilent J&W DB-HeavyWAX column.

## 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.
3. Reese, A.; Vickers, A.; George, C. GC Column Bleed: A MASS PerSPECtive. *Agilent Technologies*, publication number B-0442, **2001**.


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